ARCADIS
Infrastructure, environment, buildings

Imagine the result

Chevron Environmental Management Company

Remedial Investigation/ Feasibility Study Work Plan

Former Gulf Oil Terminal Oceanside
Town of Hempstead, New York
NYSDEC Site No. 130165

September 2010

ARCADIS

M. Nashashibi

Majda' Rabah, Ph.D., P.E.
Senior Engineer

Gregory R. Albright

Gregory Albright, P.G.
Principal Geologist

William T. McCune

William T McCune, P.G.
Project Manager

Remedial Investigation/ Feasibility Study Work Plan

Former Gulf Oil Terminal
Oceanside
Town of Hempstead, New York
NYSDEC Site No. 130165

Prepared for:
Chevron Environmental Management Company

Prepared by:
ARCADIS U.S., Inc.
1 International Boulevard
Suite 406
Mahwah
New Jersey 07495
Tel 201.684.1410
Fax 201.684.1420

Our Ref.:
B0047517.0003.00001

Date:
September 2010

1. Introduction **1**

1.1 Site Background 1

1.1.1 Site Description 1

1.1.2 Site History 1

1.2 Geology and Hydrogeology 2

1.2.1 Regional Geology/Hydrogeology 2

1.2.2 Site Geology/Hydrogeology 2

1.3 Previous Investigations 3

1.4 Site Redevelopment Plan 5

2. Remedial Investigation Work Plan **6**

2.1 Remedial Investigation Objectives 6

2.2 Remedial Investigation Scope of Work 6

2.3 Remedial Investigation Report 8

2.4 Supporting Documents 8

3. Feasibility Study Work Plan **9**

3.1 Summary of Historical Remedial Actions 9

3.1.1 Hotspot Soil Excavation – October 2002 9

3.1.2 In-Situ Chemical Oxidation Pilot Test – December 2002/January 2003 9

3.1.3 In-Situ Chemical Oxidation Pilot Test – November 2004 10

3.2 Soil Conditions 10

3.2.1 Shallow Sand Fill 10

3.2.2 Meadow Mat 11

3.2.3 Deep Sand Layer 11

3.3 Groundwater Conditions 11

3.3.1 Shallow Sand Fill 11

3.3.2 Deep Sand Layer 12

3.4 Soil and Groundwater Cleanup Objectives 12
3.4.1 Soil Cleanup Objectives 12
3.4.2 Groundwater Cleanup Objectives 13
3.5 Development and Screening of Remedial Alternatives for Soil and Groundwater 13
3.5.1 Phase I – Development of Alternatives 13
3.5.1.1 Development of Remedial Action Objectives 14
3.5.1.2 Development of General Response Actions 14
3.5.1.3 Soil Remedial Action Alternatives 15
3.5.1.4 Groundwater Remedial Action Alternatives 15
3.5.2 Phase II – Screening of Alternatives 18
3.5.3 Criteria for Preliminary Evaluation of Remedial Alternatives 19
3.5.3.1 Effectiveness 19
3.5.3.2 Implementability 19
3.5.3.3 Cost 19
3.6 FS Report 20
4. Schedule 21
5. Certification 22
6. References 23

Tables

Table 1 Summary of Soil Cleanup Objectives
Table 2 Summary of Groundwater Effluent Limitations – Class GA

Figures

Figure 1 Site Location Map
Figure 2 Site Plan
Figure 3 Soil Total VOC Contours for Fill Matrix
Figure 4 Soil Total SVOC Contour Map in Fill Matrix

Figure 5 Soil Total VOC Contours for Meadow Mat Matrix

Figure 6 Soil Total VOC Contours for Underlying Sand Matrix

Figure 7 Soil Total SVOC Map in Underlying Sand Matrix

Figure 8 Groundwater VOC Exceedance Contours for Fill Matrix

Figure 9 Groundwater VOC Contours for Underlying Sand Matrix

Figure 10 Proposed Monitoring Well Locations

Figure 11 Proposed Test Pit Locations

Figure 12 Proposed Soil Boring Locations

Appendices

A Quality Assurance Project Plan

B Health and Safety Plan

C Preliminary Implementation Schedule

D Historical Soil and Groundwater Data

E Community Air Monitoring Plan

1. Introduction

On behalf of Chevron Environmental Management Company (CEMC), ARCADIS U.S., Inc. (ARCADIS) is submitting this Remedial Investigation/Feasibility Study Work Plan (RI/FS WP) to the New York State Department of Environmental Conservation (NYSDEC) in accordance with the Order on Consent and Administrative Settlement for the former Gulf Oil Terminal in Oceanside, Township of Hempstead, New York (site).

Section 2 of this RI/FS WP presents the Remedial Investigation Work Plan (RIWP), which summarizes current site conditions and proposed remedial investigation (RI) activities to be conducted at the site. Section 3 of this RI/FS WP presents the Feasibility Study Work Plan (FSWP), which evaluates remedial alternatives for soil and groundwater at the site. The evaluation of alternatives is based on RI activities conducted at the site to date. The FS will be reevaluated once additional RI activities (as presented in the RIWP) are conducted and the results are evaluated.

This RI/FS WP has been prepared in accordance with the New York Codes, Rules, and Regulations, the draft version of the NYSDEC's Division of Environmental Remediation-10 – Technical Guidance for Site Investigation and Remediation (NYSDEC 2009), and the U.S. Environmental Protection Agency's (USEPA's) Guidance for Conducting Remedial Investigations and Feasibility Studies under CERCLA (USEPA 1988).

1.1 Site Background

1.1.1 Site Description

The site consists of 7.2 acres located at 1 Industrial Place in Oceanside, Township of Hempstead, Nassau County, New York. A Site Location Map is presented on Figure 1. The site is currently unoccupied and all former buildings have been demolished. The site is bound to the south by Barnum's Channel, to the east by the Long Island Rail Road, to the north by the former Exxon Petroleum Terminal, and to the west by Hampton Road. A junk yard and office buildings are located west of Hampton Road fronting Hog Island Channel. Daly Boulevard is located north of the former Exxon Petroleum Terminal and another petroleum terminal (Sprague) is located north of Daly Boulevard.

1.1.2 Site History

The site consists of three lots. A portion of the site (Lot 504) was first developed as a petroleum terminal by Gulf Oil Corporation in 1931. Lots 503 and 502 were purchased by Gulf Oil Corporation in 1950 and 1956, respectively. Chevron acquired Gulf Oil Corporation in the mid 1980s. As part of the purchase, Chevron was required to divest the northeast division. In 1986, Cumberland Farms, Inc. (CFI) purchased the northeast marketing assets of the Gulf Oil Corporation from Chevron. The purchase included the Oceanside Terminal, which was transferred to CFI in May 1986. In December 1993, CFI, in a joint venture with Catamount Petroleum LP (Catamount), formed Gulf Oil Limited Partnership (Gulf). CFI owned two-thirds of Gulf as a limited partner and Catamount owned one-third of Gulf as the general partner. In September 2005, CFI exercised its option to buy out Catamount and reorganized Gulf under a new general partner. However, ownership percentages of Gulf did not change during the reorganization. Gulf remains as the current owner/operator of the site.

Operation of the petroleum terminal was ceased in the early 1990s. The demolition of the aboveground storage tanks (ASTs) at the former petroleum terminal was completed in 2003. The remaining on-site buildings (maintenance building, office building and several small buildings associated with historical petroleum terminal operations) were demolished in 2005. The site is currently vacant.

The site has undergone extensive investigation and remediation since the 1990s under NYSDEC oversight (Spill No. 92-03883). In January 2007, the NYSDEC accepted Lowe’s Home Centers, Inc. (Lowe’s) as a volunteer under the Brownfield Cleanup Program (BCP) – Site No. C130165. Following the decision by Lowe’s to withdraw from the BCP in 2009, the NYSDEC transferred the site into the Hazardous Waste Program (State Superfund Site). An Order on Consent and Administrative Settlement was executed between the NYSDEC and CEMC on December 23, 2009 (Index # W3-1142-09-08, Site # 130165).

1.2 Geology and Hydrogeology

1.2.1 Regional Geology/Hydrogeology

The regional geology consists of upper glacial deposits (clay, sand, gravel and boulders) to a depth of 50 feet below ground surface (bgs), Gardiners Clay (clay, silt and lenses of sand) to a depth of 75 feet bgs, Magothy Aquifer (sand with lenses of clay) to a depth of 800 feet bgs, Raritan Confining Unit (clay with lenses of sand) to 900 feet bgs, and the Lloyd Aquifer (sand and gravel with lenses of clay) to 1,400 feet bgs. Bedrock is present beneath the Lloyd Aquifer and consists of crystalline metamorphic and igneous rock (muscovite-biotite schist, gneiss and granite).

1.2.2 Site Geology/Hydrogeology

Subsurface soil conditions encountered during previous environmental and geotechnical investigations have revealed the site lithology to consist of the following:

- sand fill from the ground surface to approximately 8 feet bgs
- meadow mat (silt with fibrous organics and trace clay) to approximately 17 feet bgs

- underlying sand (coarse to fine sand, trace to some fine gravel) to approximately 65 feet bgs
- Gardiners Clay

With the exception of the bulkhead area, the meadow mat was encountered throughout the site at a thickness ranging from 5 to 12 feet. Meadow mat was not observed during previous environmental investigations conducted in the bulkhead area.

Groundwater has historically been detected at elevations ranging between 1.5 to 3.5 feet above mean sea level (1 to 3 feet bgs). Groundwater flow direction is semi-radial with flow in the western portion of the site to the west (toward Hog Island Channel), in the southern portion of the site to the south (toward Barnum's Channel) and in the eastern portion of the site to the east (toward an unnamed channel). Tidal influence on the site has been historically documented.

1.3 Previous Investigations

Historical reports that were submitted to the NYSDEC for investigations conducted at the site are listed below:

- Hydrogeologic Site Assessment Report (ERM 1986)
- Sensitive Receptor and Potable Well Strategic Assessment (W.E.S., Inc. 1995)
- Comprehensive Site Assessment Report (Eder Associates [Eder] 1996a)
- Comprehensive Site Assessment Report - Additional Site Investigation (Eder 1996b)
- Quarterly Groundwater Monitoring Report (Eder 1996c)
- Remedial Activity Report – Surface Water Collection & Treatment System (Eder 1996d)
- Quarterly Groundwater Monitoring Report (Eder 1996e)
- Technical Specifications for On-Site Stormwater Treatment and Disposal System Upgrade Report (Eder 1996f)
- Quarterly Groundwater Monitoring Report (Eder 1997)
- Groundwater Sampling Results Report (Eder 1998)
- First Quarter 1999 Groundwater Sampling (Gannett Fleming 1999a)
- Second Quarter 1999 Groundwater Sampling (Gannett Fleming 1999b)
- Third Quarter 1999 Groundwater Sampling (Gannett Fleming 1999c)
- Fourth Quarter 1999 Groundwater Sampling (Gannett Fleming 2000a)
- First Quarter 2000 Groundwater Sampling (Gannett Fleming 2000b)
- Investigation Report (Leggette, Brashears and Graham Inc. [LBG] 2000)
- Second Quarter 2000 Groundwater Sampling (Gannett Fleming 2000c)
- Third Quarter 2000 Groundwater Sampling (Gannett Fleming 2000d)

- Fourth Quarter 2000 Groundwater Monitoring & Annual Summary (Gannett Fleming 2000e)
- Soil Investigation (LBG 2001)
- First Quarter 2001 Groundwater Monitoring (Gannett Fleming 2001a)
- Second Quarter 2001 Groundwater Monitoring (Gannett Fleming 2001b)
- Third Quarter 2001 Groundwater Monitoring (Gannett Fleming 2001c)
- Corrective Action Plan (Blasland, Bouck and Lee, Inc. [BBL] 2002a)
- Corrective Action Plan Addendum (BBL 2002b)
- Corrective Action Status Report (BBL 2003a)
- Soil Investigation Letter Report (LBG 2003)
- Remedial Action Report (BBL 2003b)
- Remedial Action Progress Report (BBL 2003c)
- Remedial Action Progress Report (BBL 2004a)
- Remedial Investigation Work Plan for the Vapor Recovery Unit Area (BBL 2004b)
- Remedial Action Progress Report (BBL 2004c)
- Environmental Investigation Report (Langan 2004)
- Revised Remedial Investigation Work Plan for the Vapor Recovery Unit Area (BBL 2004d)
- Remedial Action Progress Report (BBL 2004e)
- Corrective Action Plan (BBL 2004f)
- Remedial Action Progress Report (BBL 2004g)
- Chemical Oxidation Pilot Test (Cedar Creek Engineering 2005)
- Remedial Investigation Report and Corrective Action Plan (BBL 2005)
- Remedial Action Progress Report (BBL 2006)
- Brownfield Cleanup Program Application (Lowe’s 2006)
- Chlorinated Volatile Organic Compound Due Diligence Investigation Letter Report (Langan Engineering and Environmental Services [Langan] 2007a)
- Remedial Investigation Workplan and Remedial Action Workplan (Langan 2007b)

Each report listed above is summarized in the Remedial Investigation Workplan and Remedial Action Workplan prepared by Langan of Elmwood Park, New Jersey, and submitted to the NYSDEC in June 2007 (Langan 2007b).

The above reports document that the Gulf site operated from 1932 until the 1990s as a petroleum storage terminal. Nine large-quantity ASTs containing fuel oil, kerosene and gasoline; two small 550-gallon ASTs containing fuel oil for the on-site garage and office building; three underground storage tanks (USTs) containing fuel oil (one 550-gallon, one 1,000-gallon and one 5,000-gallon); one 1,000-gallon UST containing waste oil; a loading rack; a retention pond; a maintenance garage; and an office complex were previously located on the Gulf parcel. Four of the nine large ASTs were demolished prior to 2000, with the remaining five large ASTs reportedly demolished in 2003. The two 550-gallon ASTs containing fuel oil for the maintenance garage and the office building were demolished in 2005.

Historically, approximately 60 monitoring wells were located on the Gulf parcel. Currently, the following monitoring wells exist on site:

- four overburden (fill) wells (MW-8, MW-11, P-18 and P-26)
- seven shallow underlying sand well pairs (MW-23D, MW-24D, MW-25D, MW-27D, MW-28D, MW-29D and MW-30D) and five shallow underlying sand wells (MW-32D, MW-33D, MW-34D, MW-35D and MW-36D) installed with screened intervals below the meadow mat
- five deep underlying sand wells (MW-24VD, MW-25VD, MW-26VD, MW-29VD and MW-30VD)

The approximate location of the former building, structures, tanks and monitoring wells are depicted on the Site Plan (Figure 2).

Light nonaqueous phase liquid (LNAPL), consisting of a mixture of No. 2 and No. 4 fuel oil, kerosene, and gasoline, was previously detected in a monitoring well near the bulkhead at the southern portion of the site at a thickness of 2.28 feet. LNAPL was also encountered near the former vapor recovery unit (VRU) and the former garage building. Although measurable thicknesses of LNAPL have not been recently observed at the site, several of the monitoring points in areas where LNAPL was previously observed have been destroyed.

Soil impacted with volatile organic compounds (VOCs) at concentrations above NYSDEC Soil Cleanup Objectives is present primarily in the western and southern portions of the site, with VOC-impacted groundwater documented in the northeastern, northwestern, central and southern portions of the site. In addition to petroleum-impacted soil and groundwater, chlorinated VOCs (CVOCs), including trichloroethene (TCE), cis-1, 2-dichlorethene (cis-1,2-DCE), trans-1,2-dichloroethene and vinyl chloride (VC) were detected at the site near the former VRU. The highest concentration of CVOCs in groundwater was detected in the deep sand layer underlying the meadow mat. Tables and figures summarizing historical impacts in soil and groundwater are presented in the Remedial Investigation Workplan and Remedial Action Workplan (Langan 2007b).

Isopleth contour maps depicting the extents of VOC and semivolatile organic compound (SVOC) impacts in soil and groundwater are depicted on Figures 3 through 9.

1.4 Site Redevelopment Plan

Currently, tentative plans are being explored to redevelop the site for use as a Costco.

2. Remedial Investigation Work Plan

2.1 Remedial Investigation Objectives

The proposed RI objectives are:

- Complete supplemental investigations to address data gaps and to horizontally and vertically delineate soil and groundwater impacts. The results will be used to estimate the contaminants mass and distribution in the shallow sand fill, the underlying meadow mat and the deeper sand.
- Conduct additional pre-remedial design investigation (e.g., aquifer testing, bench-scale testing, specific-capacity testing, slug testing) to adequately understand the hydrogeologic and geochemical site characteristics to evaluate, select and design the appropriate remedial design for this site.
- Characterize the sources of contamination, migration pathways and actual or potential receptors of contaminants on or through air, soil, sediment, groundwater and surface water.
- Collect and evaluate additional data, as needed, to evaluate the remedial action alternatives.
- Collect data, as needed, to evaluate the actual and potential threats to public health and the environment.
- Collect and evaluate information for a Fish and Wildlife Resource Impact Analysis.

2.2 Remedial Investigation Scope of Work

The RI scope of work consists of the following:

1. Inspect existing monitoring wells on site to assess their conditions (e.g., number of existing wells, suitability for sampling). If any of the mapped existing wells are found to have been destroyed or are unsuitable for sampling and monitoring, CEMC will assess the need for well replacement.

2. Install additional monitoring wells in the shallow sand fill layer and the deep sand layer for vertical delineation of petroleum impacts as well as CVOCs. ARCADIS proposes to install seven monitoring wells in the shallow sand fill layer: four monitoring wells along the bulkhead and three monitoring wells in VRU area. ARCADIS also proposes to install three deep monitoring wells: two monitoring wells replacing known destroyed wells (MW-26D and MW-31D) and one new monitoring well for vertical delineation (monitoring well MW-25D3) at the former VRU. The proposed locations of the monitoring wells are depicted on Figure 10.

3. Perform test pit/trench excavations in the vicinity of the former barge dock and former garage building. A total of nine test pits/trenches will be excavated: six test pits near the former locations of the barge dock buildings and three test pits near the former garage building. The test pits will be excavated to a depth between 5 and 9 feet, depending upon the depth to water. The test pit excavations will be used to assess the extent of residual LNAPL and petroleum impacts in the shallow sand fill layer. The proposed locations of the test pits/trenches are depicted on Figure 11.

4. Conduct a soil boring investigation at the former VRU, including advancing three soil borings to complete further vertical delineation within the meadow mat. The soil borings will be drilled to an approximate maximum depth of 16 feet. The proposed locations of the soil borings are depicted on Figure 12.

5. Analyze select soil samples from soil borings and test pits for VOCs, SVOCs, Target Analyte List (TAL) metals, total organic carbon and ferrous iron.

6. Conduct aquifer testing of the deep sand layer. ARCADIS proposes to conduct a step pumping test and/or a 24-hour pumping test at monitoring well MW-30D. MW-30D was selected because this area and depth horizon may be the subject of future focus, to reduce the amount of and exposure to groundwater affected by higher concentrations of CVOCs.

7. Perform tracer testing at the deep sand layer. A nonreactive tracer (e.g., fluorescein) will be used to further assess hydraulic characteristics of the water-bearing units beneath the site.

8. Groundwater sampling of existing and newly installed monitoring wells. The groundwater samples will be analyzed for VOCs, SVOCs, methane, ethane, ethene, total dissolved solids, alkalinity, alkalinity-bicarbonate, sulfate, sulfide, nitrate, nitrite, nitrogen as ammonia, phosphorous as orthophosphate, calcium, total dissolved organic carbon, magnesium (total and dissolved), iron (total and dissolved), and TAL metals (total and dissolved).

9. Conduct specific-capacity testing during the proposed groundwater sampling event (Item 7 above).

10. Conduct bench-scale testing of soil and groundwater to estimate the soil oxidant demand and efficacy of persulfate under differing activation scenarios to evaluate the feasibility of an in-situ chemical oxidation (ISCO) remedial option for saturated soil and groundwater in both the shallow and deep sand layers. Soil samples for bench-scale testing will be collected during installation of the additional monitoring wells and during the soil boring investigation. Groundwater samples for bench-scale testing will be collected during the proposed groundwater sampling event (Item 7 above). Bench-scale tests will be conducted on samples collected from the meadow mat for thermal resistivity. Other bench-scale tests may be conducted, as necessary, as RI work progresses at the site.

11. Perform remediation hydraulics evaluations as needed to refine the understanding of contaminant fate and transport, aquifer architecture, and refinement of the conceptual site model.

After completing and evaluating the proposed RI work scope, additional remedial or pre-design investigation activities will be proposed if remaining data gaps are identified.

2.3 Remedial Investigation Report

Upon completion of the RI activities, ARCADIS, on behalf of CEMC, will prepare a Remedial Investigation Final Report for submittal to the NYSDEC for review and approval.

2.4 Supporting Documents

The following documents are included as appendices to this RI/FS Work Plan:

- Quality Assurance Project Plan (Appendix A)
- Health & Safety Plan (Appendix B)
- Tentative RI Implementation Schedule (Appendix C)
- Historical Soil and Groundwater Data (Appendix D)
- Community Air Monitoring Plan (Appendix E)

3. Feasibility Study Work Plan

This FSWP evaluates remedial alternatives for site soil and groundwater, with the goal of mitigating potential human health and environmental risks associated with the contaminants of concern (COCs) identified at the site. This FSWP provides a detailed analysis of preferred remedial technologies and remedial approaches, which are protective of human health and the environment, cost effective, implementable, and limit the migration of contaminants.

This FSWP is based on the site characterization results available to date. The results of additional RI activities described in Section 2 will be used to reevaluate the remedial alternatives presented herein.

3.1 Summary of Historical Remedial Actions

Remedial actions conducted at the site are discussed below.

3.1.1 Hotspot Soil Excavation – October 2002

In October 2002, BBL completed four hotspot excavations to remove previously observed LNAPL-impacted soil and areas of elevated petroleum-hydrocarbon-contaminated soil. The four hotspot excavation areas included the area surrounding the VRU/small AST pad, an area west of the former garage building, an area southeast of the former truck loading racks and an area southeast of the former garage building. The approximate locations of the excavated areas are depicted on Figure 2.

The excavations were completed to 4 feet bgs. Approximately 438 tons of soil was excavated for off-site disposal. Certified clean fill was used to backfill the excavations. The post-excavation soil sampling analytical results revealed that benzene, toluene, ethylbenzene and xylenes (BTEX) and naphthalene were detected above the NYSDEC Recommended Soil Cleanup Objectives in each of the four excavations.

LNAPL was observed seeping into the excavations and was recovered with a vacuum truck until only sheen remained. A total of 2,300 gallons of LNAPL/water were removed via vacuum truck for off-site disposal.

3.1.2 In-Situ Chemical Oxidation Pilot Test – December 2002/January 2003

An ISCO pilot test was completed in December 2002 and January 2003. The pilot test consisted of the injection of a 20 percent solution of sodium permanganate into the subsurface at Excavation A (Figure 2). The injection rates in the fill were found to be low, with each injection point accepting only ½ to ¾ of a drum over several hours.

One round of groundwater sampling was completed in March 2003 to assess the effectiveness of the ISCO pilot test. A significant decrease in groundwater concentrations was observed within wells reinstalled in the former excavation areas and was attributed to the October 2002 hotspot soil excavation. In addition, a decrease in CVOCs by 25 to 100 percent near the VRU was noted, suggesting that the ISCO pilot test was effective at treating CVOCs.

3.1.3 In-Situ Chemical Oxidation Pilot Test – November 2004

An ISCO pilot test study was completed in November 2004 within the Former Turbine Pump Area. A Modified Fenton’s Reagent consisting of ferrous sulfate, citric acid and hydrogen peroxide was used for the pilot test. The pilot test used 2 and 4 percent aqueous solutions.

Two types of injection methods were tested. The first method involved Geoprobe™ injection points and the second method used temporary well injection points. The reagent was injected at 20 pounds per square inch pressure in the Geoprobe points, whereas gravity feed was used in the temporary well injection points. Pre- and post-injection soil samples were collected and analyzed and results indicated no significant modification (less than 10 percent increase or decrease) to the concentration subsequent to injection. Fenton’s Reagent was determined to be an ineffective means of delivering the reagent to the soil due to poor reagent-soil mixing.

3.2 Soil Conditions

VOC soil impacts in the shallow sand fill are primarily located at the former VRU and the former garage building. VOC impacts in the underlying meadow mat and the deep sand layer are localized in the former VRU, with the exception of methylene chloride in the deep sand layer, which is detected in the former Truck Loading Area. The COCs present in each soil layer are presented in Sections 3.2.1, 3.2.2 and 3.2.3. Appendix D Figures 1, 2 and 3 present the historical soil concentrations for the COCs in each soil layer.

3.2.1 Shallow Sand Fill

The following COCs were detected in the shallow sand fill at concentrations exceeding the NYSDEC Unrestricted Use Soil Cleanup Objectives:

- BTEX at the former VRU and the former garage building
- TCE at the former VRU
- cis-1,2-DCE at the former VRU
- VC at the former VRU
- methylene chloride at the former VRU and the former garage building

3.2.2 Meadow Mat

The following COCs were detected in the meadow mat at concentrations exceeding the NYSDEC Unrestricted Use Soil Cleanup Objectives:

- BTEX at the former VRU
- TCE at the former VRU
- cis-1,2-DCE at the former VRU
- VC at the former VRU
- methylene chloride at the former VRU

3.2.3 Deep Sand Layer

The following COCs were detected in the deep sand layer at concentrations exceeding the NYSDEC Unrestricted Use Soil Cleanup Objectives:

- BTEX at the former VRU
- TCE at the former VRU
- cis-1,2-DCE at the former VRU
- VC at the former VRU
- methylene chloride at the former Truck Loading Area

3.3 Groundwater Conditions

VOC groundwater impacts in the shallow sand fill are located at the eastern, southern and western portions of the site (at the former VRU, former retention pond, former ASTs and the bulkhead area), with CVOCs being mainly detected at the former VRU. VOC impacts in the deep sand layer are localized in the former VRU. The COCs present in each groundwater zone are presented in Sections 3.3.1 and 3.3.2. Appendix D Figures 4 and 5 present the historical groundwater concentrations for the COCs in each groundwater zone.

LNAPL, consisting of a mixture of No. 2 and No. 4 fuel oil, kerosene, and gasoline, was detected in monitoring wells near the bulkhead at the southern portion of the site and near the former VRU and the former garage building.

3.3.1 Shallow Sand Fill

The following COCs were detected in the shallow groundwater at concentrations exceeding the NYSDEC Groundwater Quality Standards:

- BTEX at the former VRU, former garage building, bulkhead area, former detention pond and former AST area

- methyl tert-butyl ether (MTBE) at the former VRU, former garage building, bulkhead area, former retention pond and former AST area

- TCE at the former VRU

- cis-1,2-DCE at the former VRU

- VC at the former VRU

3.3.2 Deep Sand Layer

The following COCs were detected in the deep sand layer at concentrations exceeding the NYSDEC Groundwater Quality Standards:

- BTEX mainly at the former VRU
- MTBE at the former VRU and the former truck loading area
- TCE at the former VRU
- cis-1,2-DCE at the former VRU
- VC at the former VRU
- methylene chloride at the former VRU

3.4 Soil and Groundwater Cleanup Objectives

3.4.1 Soil Cleanup Objectives

Soil cleanup objectives are based on the NYSDEC Technical & Operational Guidance Series (TOGS) for Soil, Subpart 375-6: Remedial Program Soil Cleanup Objectives (effective December 14, 2006) (NYSDEC 2006). The Unrestricted Use Soil Cleanup Objectives for all compounds that have been detected on the site are included in Table 1. If unrestricted use soil cleanup objectives cannot be reasonably be obtained, restricted (commercial) soil clean up objectives will be requested based on the location of the property and its proposed use as a commercial facility.

3.4.2 Groundwater Cleanup Objectives

Groundwater cleanup objectives are based on the NYSDEC TOGS for Groundwater, Section 703.6 (effective August 4, 1999) (NYSDEC 1999). Groundwater cleanup standards for all compounds that have been detected on the site are included in Table 2.

3.5 Development and Screening of Remedial Alternatives for Soil and Groundwater

This section identifies and screens remedial technologies and process options that ensure protection of human health and the environment for the site. The identification and screening process outlined below was developed based on 6 NYCRR Part 375 (effective December 14, 2006). The technology/process identification, screening and selection process is divided into three phases:

1. Phase I – development of alternatives

2. Phase II – screening of alternatives

3. Phase III – detailed analysis of alternatives

Preliminary results of the development and screening of remedial alternatives completed to date discussed in Sections 3.5.1 and 3.5.2; A complete presentation of Phases I thru III will be presented in the Focused Feasibility Study Report to be submitted to the NYSDEC following the RI proposed in Section 2 of this RI/FS WP.

3.5.1 Phase I – Development of Alternatives

The alternative development process consists of a series of analytical steps that involve making successively more specific definitions and analysis of potential remedial activities.

The development of alternatives phase consists of the following general steps:

- develop remedial action objectives (RAOs) specifying contaminants and media of interest at the site

- develop general response actions for each media of interest to satisfy remedial objectives for the site

- identify technology types and process options applicable to the site and eliminate those that cannot be implemented technically

3.5.1.1 Development of Remedial Action Objectives

RAOs are site-specific cleanup objectives established to protect human health and the environment. RAOs specify contaminants and media of concern, potential exposure pathways, and receptors. RAOs indicate both a contaminant level and an exposure route, because protection of human and ecological receptors may be achieved by reducing or eliminating exposure pathways as well as by reducing contaminant concentrations.

RAOs have been established as part of the FS to evaluate remedial alternatives that will protect human health and the environment; consider the requirements of New York's Standards, Criteria and Guidance; provide practical, cost-effective remediation; and select permanent remedies that can be expedited as required and to the extent possible. Site-specific RAOs are developed based on the extent of identified impacts and on the site geologic and hydrogeologic conditions. COCs for impacted groundwater at the site are summarized in the RIWP (Section 2) and Sections 3.2 and 3.3 of this RI/FS Work Plan.

Groundwater is not used for potable or production purposes at the site. Thus, there is no direct exposure to contaminants in groundwater. However, because VOCs have been detected in soil and groundwater at the site above the applicable NYSDEC standards, the RAO for soil and groundwater at the site is to reduce the concentration of VOCs in soil and groundwater to the NYSDEC applicable standards, to the extent practicable (Tables 1 and 2, respectively).

3.5.1.2 Development of General Response Actions

General response actions are remediation alternatives and/or techniques that can be used individually or in combination to achieve the RAO established for soil and groundwater at the site.

The general response actions focus on:

- achieving the soil and groundwater RAO
- limiting concentrations in place
- preventing exposure to soil and groundwater with impacts above the applicable NYSDEC standards and unacceptable risk levels associated with soil and groundwater

The general response actions identified for the site include hotspot soil excavation and in-situ treatment. These response actions and specific technologies are discussed in Sections 3.5.1.3 and 3.5.1.4.

3.5.1.3 Soil Remedial Action Alternatives

Groundwater at the site is encountered at 1 to 3 feet bgs. Thus, impacts in the shallow sand fill, meadow mat and deep sand layer will be addressed along with the groundwater because the impacted soil is in the smear/saturated zone (except for hotspot excavation within the shallow sand fill).

In addition to hotspot excavation, in-situ soil mixing (a physical treatment technology for remediating soils contaminated with VOCs) combined with chemical oxidation and focused LNAPL recovery will be considered as a treatment alternative for this site.

3.5.1.4 Groundwater Remedial Action Alternatives

Groundwater impacts at the site include VOCs. This section summarizes the available remedial technology groups and specific remedial techniques for treatment of groundwater at the site. As indicated in Section 3.5.1.3, soil impacts will be addressed by addressing groundwater impacts because soil impacts are within the smear/ saturated zone (shallow water table at the site encountered at 1 to 3 feet bgs). In-situ remedial alternatives are evaluated below.

In-situ treatment technologies are those technologies used to treat impacted soil and groundwater “in place.” The in-situ treatment technologies applicable for remediation of soil and groundwater impacts at the site are discussed in Sections 3.5.1.4.1, 3.5.1.4.2, 3.5.1.4.3 and 3.5.1.4.4.

3.5.1.4.1 Biological Treatment

In-situ bioremediation is the enhancement of naturally occurring biological activity in groundwater to degrade chemical constituents present in the subsurface. In-situ bioremediation through biostimulation involves injecting certain compounds into the subsurface to stimulate the activity of indigenous microorganisms and increase rates of natural biodegradation. The effectiveness of in-situ bioremediation as a treatment technology is affected by the nature and distribution of contaminants at the site, biodegradability of the constituents, mineralization potential of constituents, existing microbial and substrate conditions, availability of nutrients, site geology and hydrogeology, and biogeochemical parameters and conditions in the subsurface. In-situ biological treatment alternatives including anaerobic bio-oxidation of petroleum hydrocarbons (i.e., sulfate amendments), anaerobic reductive dechlorination of chlorinated solvents (i.e., carbohydrate injections) and aerobic bioremediation of petroleum hydrocarbons (e.g., injection of oxygenated water, biosparging) will be further evaluated as potential remedial approaches for the shallow sand fill soil and groundwater at the site.

3.5.1.4.2 In-Situ Chemical Oxidation

ISCO normally consists of manipulation of groundwater chemistry to create oxidation reactions in groundwater for the in-situ treatment of impacts in groundwater.

In-situ oxidation is a potential chemical treatment technology that can be used to destroy on-site groundwater impacts in place. The addition of powerful oxidizing chemicals such as permanganate, persulfate, ozone or hydrogen peroxide into the groundwater can often result in destruction of VOCs.

ISCO using sodium permanganate was tested at the site in December 2002 and January 2003, with favorable results for oxidation of CVOCs. However, permanganate is least effective with petroleum hydrocarbons (e.g. BTEX). Permanganate works best with a double carbon bond. Second, some COCs react so rapidly with permanganate that the kinetics of contaminant disappearance will not be a limiting factor in almost any design scenario. Benzene reacts so slowly with permanganate that effective remediation with permanganate will be hard to achieve under most circumstances. Thus, ISCO using permanganate will not be considered for this site.

ISCO using a Modified Fenton's Reagent consisting of ferrous sulfate, citric acid and hydrogen peroxide was tested at the site in November 2004. Results of the pre- and post-injection soil samples revealed no significant modification (less than 10 percent increase or decrease) to the concentration subsequent to injection. Fenton's Reagent was determined to be an ineffective means of delivering the reagent to the soil due to poor reagent-soil mixing. Thus ISCO using Fenton's Reagent will not be further considered for this site.

ISCO using activated persulfate will probably oxidize both the petroleum hydrocarbons and the CVOCs in groundwater. Persulfate is an emerging oxidant that has a relatively long lifetime, lasting from weeks to months in most subsurface conditions. However, its stability comes along with slow reaction kinetics. Hence, persulfate is usually injected in conjunction with an activator (such as chelated iron, base or hydrogen peroxide) to increase reaction kinetics. The activator converts the slow persulfate kinetics into sulfate radicals, which are stronger oxidants with faster kinetics. This treatment alternative will be evaluated further as a remedial alternative for treatment of the meadow mat and the deep sand layer.

3.5.1.4.3 Physical Treatment

Physical treatment is a remedial technique whereby subsurface mass transfer conditions are enhanced to provide mass removal from groundwater. One of the most common physical treatment techniques is air sparging. Air sparging is an in-situ remedial technique in which air is injected under pressure below the water table. Air sparging is primarily used for the treatment of VOCs dissolved in groundwater and adsorbed to saturated soils. Air sparging is typically used in combination with soil vapor extraction (SVE) for the treatment of VOCs.

In-well sparging is a modification of the conventional method of air sparging and uses a double-cased well. The outer casing is screened in both the impacted zone and at the top of the water table. Compressed air is injected through a drop tube to the base of the well, which is screened in the impacted zone, inducing the water column within the inner casing to be lifted upward (similar to pumping) and to overflow the top of the casing. Due to the mixing of air and the impacted water, as the air/water mixture rises inside the inner tube, VOCs can be stripped and captured for treatment. Simultaneous with the injection of air into the bottom of the inner casing, a vacuum is applied to the wellhead and extracted air is directed through vapor phase treatment prior to discharge to the atmosphere. Treated, clean water, which spills over the top of the inner casing, is injected back into the formation via the top outer screen without actually leaving the subsurface.

Based on the nature of COCs identified at the site, either air sparging/SVE or in-well sparging may be suitable remedial alternatives. However, due to the shallow depth of the water table, an SVE system cannot be installed at the site (thus, stripped VOCs cannot be captured). In addition, the presence of LNAPL will limit the use of air sparging at the site. Therefore, these technologies will not be further evaluated for use at this site.

3.5.1.4.4 Thermal Treatment

In-situ thermal heating methods were first developed by the petroleum industry for enhanced oil recovery. These methods were adapted to the treatment of soil and groundwater. Initial variations included hot water injection, steam injection, hot air injection and electrical resistive heating (ERH). In the late 1980s and early 1990s, thermal conductive heating was developed. Thermal treatment will be primarily considered for low-permeability zones at the site, where the contaminant mass is adsorbed and entrapped.

Steam injection, ERH and thermal conductive heating are used for remediation of soil and groundwater in source zones contaminated with chlorinated solvents.

3.5.1.4.4.1 Steam Enhanced Extraction

Steam enhanced extraction typically involves the injection of steam into the subsurface to dissolve, vaporize and mobilize contaminants that are then recovered. Mobilized contaminants are extracted from the subsurface using vapor and liquid extraction equipment. Extracted vapors and liquids are treated using conventional aboveground treatment technologies, such as condensation, air stripping, carbon adsorption and thermal oxidation. In low-permeability soil, steam cannot penetrate the pore space as rapidly, resulting in higher heat losses and, in some cases, the inability to completely heat the area. In addition, smaller pore diameters create higher capillary pressures and, as a result, lower the rate of evaporation of contaminants.

3.5.1.4.4.2 Electrical Resistive Heating

ERH involves the application of electrical current through the subsurface, which generates heat. ERH uses the natural electrical resistance within the subsurface where energy is dissipated through ohmic (or resistive) losses. This manner of in-situ heating allows energy to be focused into a specific source zone. Steam is generated when the subsurface temperature is increased to the boiling point of the pore water or the saturated media in the treatment zone. The steam strips contaminants from the soils and enables them to be extracted from the subsurface. ERH is particularly suited to the treatment of lower permeability strata and to dense nonaqueous phase liquids that have become consolidated within lower permeability zones with higher organic content. ERH will be evaluated further as a remedial alternative for treatment of the meadow mat and the deep sand layer at the site.

3.5.1.4.4.3 Thermal Conductive Heating

Thermal conductive heating involves simultaneous application of heat and vacuum to subsurface soils with an array of vertical heater/vacuum wells or, much less commonly, with surface blanket heaters and a vacuum insulated shroud. In both of these configurations, heat originates from a heating element and is transferred to the subsurface largely via thermal conduction and radiant heat transport, which dominates near the heat sources. There is also a contribution through convective heat transfer that occurs during the formation of steam from pore water. Because this technology can achieve elevated soil temperatures (in excess of 500 degrees Celsius), a significant portion (reported up to 99 percent at some sites) of organic contaminants either oxidize (if sufficient air is present) or pyrolize once high soil temperatures are achieved. Therefore, this technology is also considered to be an in-situ destruction method.

Low boiling point compounds such as chlorinated solvents can be treated with thermal conductive heating through achievement of steam distillation temperatures in the bulk of the interwell regions. Locations close to heaters may achieve temperatures well above the boiling point of water. However, boiling off all of the soil water is not necessary. Removal rates in excess of 99 percent have been measured for thermal conductive heating of chlorinated solvents. Thermal conductive heating will be evaluated further as a remedial alternative for treatment of meadow mat and the deep sand layer at the site.

3.5.2 Phase II – Screening of Alternatives

In the alternative preliminary screening process phase, remedial alternatives are evaluated based on effectiveness, implementability and cost. At the completion of this evaluation, remedial alternative technologies are eliminated or retained for a detailed analysis.

Based on screening of identified technology types and process options, the following remedial alternatives have been selected for preliminary evaluation:

- Alternative 1 – Hot-spot soil excavation and off-site disposal of the shallow sand fill layer.

- Alternative 2 – Injection of sulfates for anaerobic bio-oxidation of petroleum hydrocarbon impacts in shallow sand fill. This will target impacts in both soil and groundwater.

- Alternative 3 – ISCO of groundwater impacts in the deep sand layer, and impacts in the meadow mat and the deeper sand layer.

- Alternative 4 – Thermal treatment of groundwater impacts in the deep sand layer, and impacts in the meadow mat and the deep sand layer.

- Alternative 5 – In-situ soil mixing with chemical oxidation and focused LNAPL recovery.

- Alternative 6 – Aerobic treatment for the shallow sand layer.

3.5.3 Criteria for Preliminary Evaluation of Remedial Alternatives

The above alternatives will be screened based upon the COCs at the site, subsurface hydrogeologic conditions, anticipated future industrial land use, ability of the technology to meet the RAO and the criteria presented in Sections 3.5.3.1, 3.5.3.2 and 3.5.3.3.

3.5.3.1 Effectiveness

The remedial technologies and processes are evaluated based on their effectiveness relative to other potential processes based on their ability to achieve the RAO and to reliably treat the identified COCs. Effectiveness may be determined based on historical knowledge of the remedial process and/or the results of laboratory-scale or pilot testing. Common factors in determining the effectiveness of a technology are the ability of the remedial process to reduce toxicity, mobility and/or mass of constituents.

3.5.3.2 Implementability

Implementability encompasses the technical and institutional feasibility of implementing a remedial technology or process option. Technical implementability will be evaluated in relation to existing site conditions, such as hydrogeology and the distribution of constituents. Remedial alternatives that are ineffective and inapplicable to site conditions will be eliminated based on the following factors:

- difficulty in constructing or operating
- potentially adverse health and environmental impacts
- potential difficulties associated with material handling
- adverse effects of the chemicals and other materials used by the remedial process
- adverse effects to other selected remedial technologies implemented at the site

3.5.3.3 Cost

Remedial alternatives, which are higher in cost without offering greater implementability and effectiveness in relation to the other alternatives, will be eliminated.

A detailed analysis of each alternative versus the above criteria will be presented in the Focused Feasibility Study to be submitted to the NYSDEC following the RI proposed in Section 2 of this RI/FS WP.

3.6 FS Report

A FS Report will summarize the information developed during the FS process as described above. In accordance with NYSDEC guidance, a comparative analysis of each remedial action alternative for addressing impacted media at the Site using the evaluation criteria discussed above will be conducted. The purpose of the comparative analysis will be to identify the relative advantages/disadvantages of each remedial action alternative and to highlight the differences between the alternatives. The results of the comparative analysis will be presented in the FS Report and will be used as the basis for recommending a remedial alternative for addressing impacted media at the Site. The FS Report will be submitted to the NYSDEC for review and approval.

4. Schedule

A preliminary schedule for implementation of the RI/FS tasks detailed herein is included in Appendix C.

5. Certification

This is to certify that the Remedial Investigation/Feasibility Study Work Plan for the Former Gulf Oil Terminal, Oceanside, New York, Site # 130165 was prepared in accordance with the Order on Consent, Index # W3-1142-09-08, as entered into by the NYSDEC.

6. References

Blasland, Bouck and Lee, Inc. 2002a. Corrective Action Plan.

Blasland, Bouck and Lee, Inc. 2002b. Corrective Action Plan Addendum.

Blasland, Bouck and Lee, Inc. 2003a. Corrective Action Status Report.

Blasland, Bouck and Lee, Inc. 2003b. Remedial Action Report.

Blasland, Bouck and Lee, Inc. 2003c. Remedial Action Progress Report.

Blasland, Bouck and Lee, Inc. 2004a. Remedial Action Progress Report.

Blasland, Bouck and Lee, Inc. 2004b. Remedial Investigation Work Plan for the Vapor Recovery Unit Area.

Blasland, Bouck and Lee, Inc. 2004c. Remedial Action Progress Report.

Blasland, Bouck and Lee, Inc. 2004d. Revised Remedial Investigation Work Plan for the Vapor Recovery Unit Area.

Blasland, Bouck and Lee, Inc. 2004e. Remedial Action Progress Report.

Blasland, Bouck and Lee, Inc. 2004f. Corrective Action Plan.

Blasland, Bouck and Lee, Inc. 2004g. Remedial Action Progress Report.

Blasland, Bouck and Lee, Inc. 2005. Remedial Investigation Report and Corrective Action Plan.

Blasland, Bouck and Lee, Inc. 2006. Remedial Action Progress Report.

Cedar Creek Engineering. 2005. Chemical Oxidation Pilot Test.

Eder Associates. 1996a. Comprehensive Site Assessment Report.

Eder Associates. 1996b. Comprehensive Site Assessment Report - Additional Site Investigation.

Eder Associates. 1996c. Quarterly Groundwater Monitoring Report.

Eder. 1996d. Remedial Activity Report – Surface Water Collection & Treatment System.

Eder. 1996e. Quarterly Groundwater Monitoring Report.

Eder. 1996f. Technical Specifications for On-Site Stormwater Treatment and Disposal System Upgrade Report.

Eder. 1997. Quarterly Groundwater Monitoring Report.

Eder. 1998. Groundwater Sampling Results Report.

ERM. 1986. Hydrogeologic Site Assessment Report.

Gannett Fleming. 1999a. First Quarter 1999 Groundwater Sampling.

Gannett Fleming. 1999b. Second Quarter 1999 Groundwater Sampling.

Gannett Fleming. 1999c. Third Quarter 1999 Groundwater Sampling.

Gannett Fleming. 2000a. Fourth Quarter 1999 Groundwater Sampling.

Gannett Fleming. 2000b. First Quarter 2000 Groundwater Sampling.

Gannett Fleming. 2000c. Second Quarter 2000 Groundwater Sampling.

Gannett Fleming. 2000d. Third Quarter 2000 Groundwater Sampling.

Gannett Fleming. 2000e. Fourth Quarter 2000 Groundwater Monitoring & Annual Summary.

Gannett Fleming. 2001a. First Quarter 2001 Groundwater Monitoring.

Gannett Fleming. 2001b. Second Quarter 2001 Groundwater Monitoring.

Gannett Fleming. 2001c. Third Quarter 2001 Groundwater Monitoring.

Langan Engineering and Environmental Services. 2004. Environmental Investigation Report.

Langan Engineering and Environmental Services. 2007a. Chlorinated Volatile Organic Compound Due Diligence Investigation Letter Report.

Langan Engineering and Environmental Services. 2007b. Remedial Investigation Workplan and Remedial Action Workplan.

Leggette, Brashears and Graham Inc. 2000. Investigation Report.

Leggette, Brashears and Graham Inc. 2001. Soil Investigation.

Leggette, Brashears and Graham Inc. 2003. Soil Investigation Letter Report.

Lowe's Home Centers, Inc. 2006. Brownfield Cleanup Program Application.

New York State Department of Environmental Conservation. 1999. Technical & Operational Guidance Series for Groundwater, Section 703.6.

New York State Department of Environmental Conservation. 2006. Technical & Operational Guidance Series for Soil, Subpart 375-6: Remedial Program Soil Cleanup Objectives.

New York State Department of Environmental Conservation. 2009. Technical Guidance for Site Investigation and Remediation.

U.S. Environmental Protection Agency. 1988. Guidance for Conducting Remedial Investigations and Feasibility Studies under CERCLA.

W.E.S., Inc. 1995. Sensitive Receptor and Potable Well Strategic Assessment.

TABLES

Table 1. Summary of Soil Cleanup Objectives, Former Gulf Oil Terminal, Oceanside, Town of Hempstead, New York

Compounds of Concern	Soil Cleanup Objectives Unrestricted Use
Detected Volatile Organics	
1,1-Dichloroethene	0.33
1,2,4-Trimethylbenzene	3.6
1,3,5-Trimethylbenzene	8.4
Acetone	0.05
Benzene	0.06
Carbon Disulfide	NS
cis-1,2-Dichloroethene	0.25
Ethylbenzene	1
Isopropylbenzene	NS
Methyl tert-butyl ether	0.93
Methylene Chloride	0.05
Naphthalene	12
n-Butylbenzene	12
n-Propylbenzene	3.9
p-Isopropyltoluene	NS
sec-Butylbenzene	11
Styrene	NS
tert-Butylbenzene	5.9
Tertiary butyl alcohol	NS
Tetrachloroethene	1.3
Toluene	0.7
trans-1,2-Dichloroethene	0.19
Trichloroethene	0.47
Vinyl Chloride	0.02
Xylenes (total)	0.26
Detected Semivolatile Organics	
2-Methylnaphthalene	NS
Acenaphthene	20
Acenaphthylene	100
Anthracene	100
Benzo(a)anthracene	1
Benzo(a)pyrene	1
Benzo(b)fluoranthene	1
Benzo(g,h,i)perylene	100
Benzo(k)fluoranthene	0.8
bis(2-Ethylhexyl)phthalate	NS
Chrysene	1
Dibenzo(a,h)anthracene	0.33
Fluoranthene	100
Fluorene	30
Indeno(1,2,3-cd)pyrene	0.5
Phenanthrene	100
Pyrene	100
Additional Detected Parameters	
Total Organic Carbon	NS
Total Petroleum Hydrocarbons	NS

Notes:
All Soil Cleanup Objectives are in parts per million (ppm) or milligrams per kilogram (mg/kg).
NS - Not Specified.

Reference:
Subpart 375-6: Remedial Program Soil Cleanup Objectives; Effective December 14, 2006.

Table 2. Summary of Groundwater Effluent Limitation - Class GA, Former Gulf Oil Terminal, Oceanside, Town of Hempstead, New York

Compounds of Concern	Limitations Class GA
Detected Volatile Organics	
1,1,2,2-Tetrachloroethane	5
1,1-Dichloroethane	5
1,1-Dichloroethene	5
1,2-Dichloroethane	0.6
1,3,5-Trimethylbenzene	5
2-Butanone	50
Acetone	50
Acrylonitrile	5
Benzene	1
Bromodichloromethane	50
Carbon Disulfide	60
Chloroethane	5
Chloroform	7
Chloromethane	5
cis-1,2-Dichloroethene	5
Ethylbenzene	5
Isopropylbenzene	5
m&p-Xylene	5
Methyl tert-butyl ether	10
Methylene Chloride	5
Naphthalene	10
n-Butylbenzene	5
n-Propylbenzene	5
o-Xylene	5
p-Isopropyltoluene	5
sec-Butylbenzene	5
tert-Butylbenzene	5
Tertiary butyl alcohol	NS
Tetrachloroethene	5
Toluene	5
trans-1,2-Dichloroethene	5
Trichloroethene	5
Trichlorofluoromethane	5
Vinyl Chloride	2
Xylenes (total)	5
Detected Semivolatile Organics	
2,4-Dimethylphenol	50
Acenaphthene	20
Acenaphthylene	NS
Anthracene	50
Benzo(a)anthracene	0.002
Benzo(a)pyrene	NS [1]
Benzo(b)fluoranthene	0.002
Benzo(g,h,i)perylene	NS
Benzo(k)fluoranthene	0.002
bis(2-Ethylhexyl)phthalate	5
Chrysene	0.002
Dibenzo(a,h)anthracene	NS
Di-n-Butylphthalate	50
Fluoranthene	50
Fluorene	50
Hexachlorocyclopentadiene	5
Hexachloroethane	5
Indeno(1,2,3-cd)pyrene	0.002
Naphthalene	10
Nitrobenzene	0.4
Pentachlorophenol	NS [2]
Phenanthrene	50
Phenol	1
Pyrene	50
Additional Detected Parameters	
Chloride	250,000
Total Dissolved Solids	1,000,000 [3]
Total petroleum hydrocarbor	NS

Notes:

Groundwater Effluent Limitations are in micrograms per liter (ug/l)

NS - Not Specified.

1. The maximum allowable concentration in listed as "ND".
2. The sum of all phenolic compounds (total phenols) must be less than 1 ug/L.
3. Only applies in Nassau and Suffolk County.

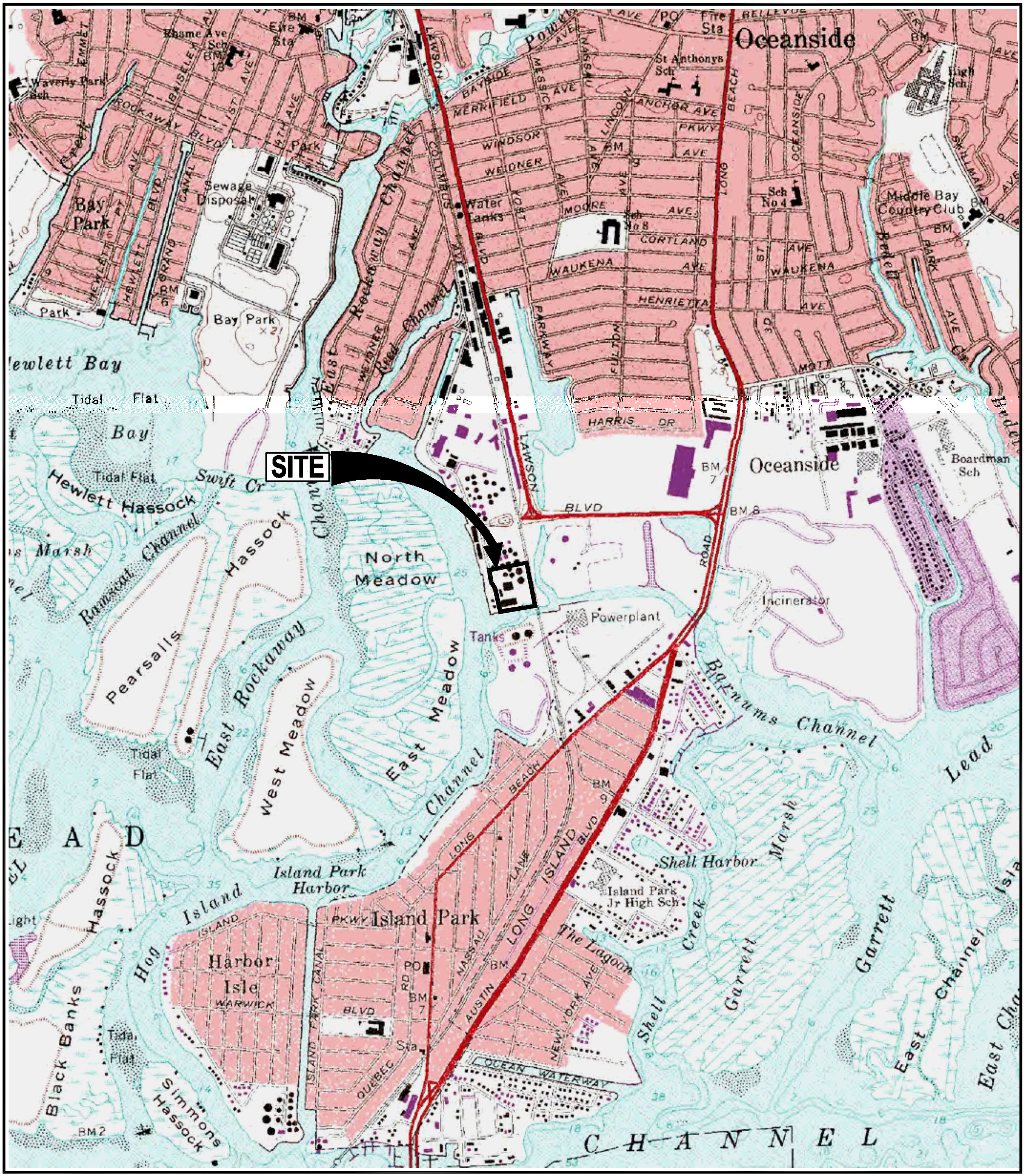
References:

- Part 703: Surface Water and Groundwater Quality Standards and Groundwater Effluent Limitations; Effective August 4, 1999.
- Technical & Operational Guidance Series 1.1.1: Ambient Water Quality Standards and Guidance Values and Groundwater Effluent Limitation; Published June 1998 and addended January 1999, April 2000, and June 2004.

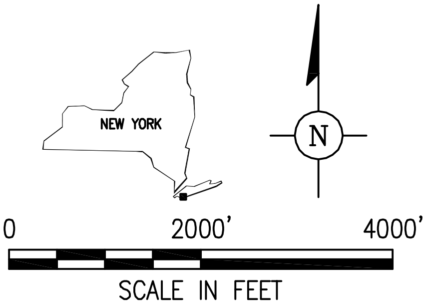
FIGURES

CITY:Melville - NY DIV/GROUP:ENR1 DB:A.Sanchez LD: PIC: PM: TM: LYR:ON=*;OFF=*REF*
G:\ENVCAD\Melville-NY\RETURN-TO\Mahwah-NJ\B0047517\0003\00001\Location Map.dwg LAYOUT: 1SAVED: 5/6/2010 4:15 PM ACADVER: 17.1S (LMS TECH) PAGESETUP: ----PLOTSTYLETABLE: ARCADIS_MELVILLE.CTB PLOTTED: 5/6/2010 4:15 PM BY: SANCHEZ, ADRIAN

XREFS: IMAGES: PROJECTNAME:
LAWRENCE.TIF
LYNBROOK.TIF
SEA CLIFF.TIF



MAP SOURCE: USGS 7.5 MINUTE QUADRANGLE 1979 LYNBROOK AND LAWRANCE, NEW YORK

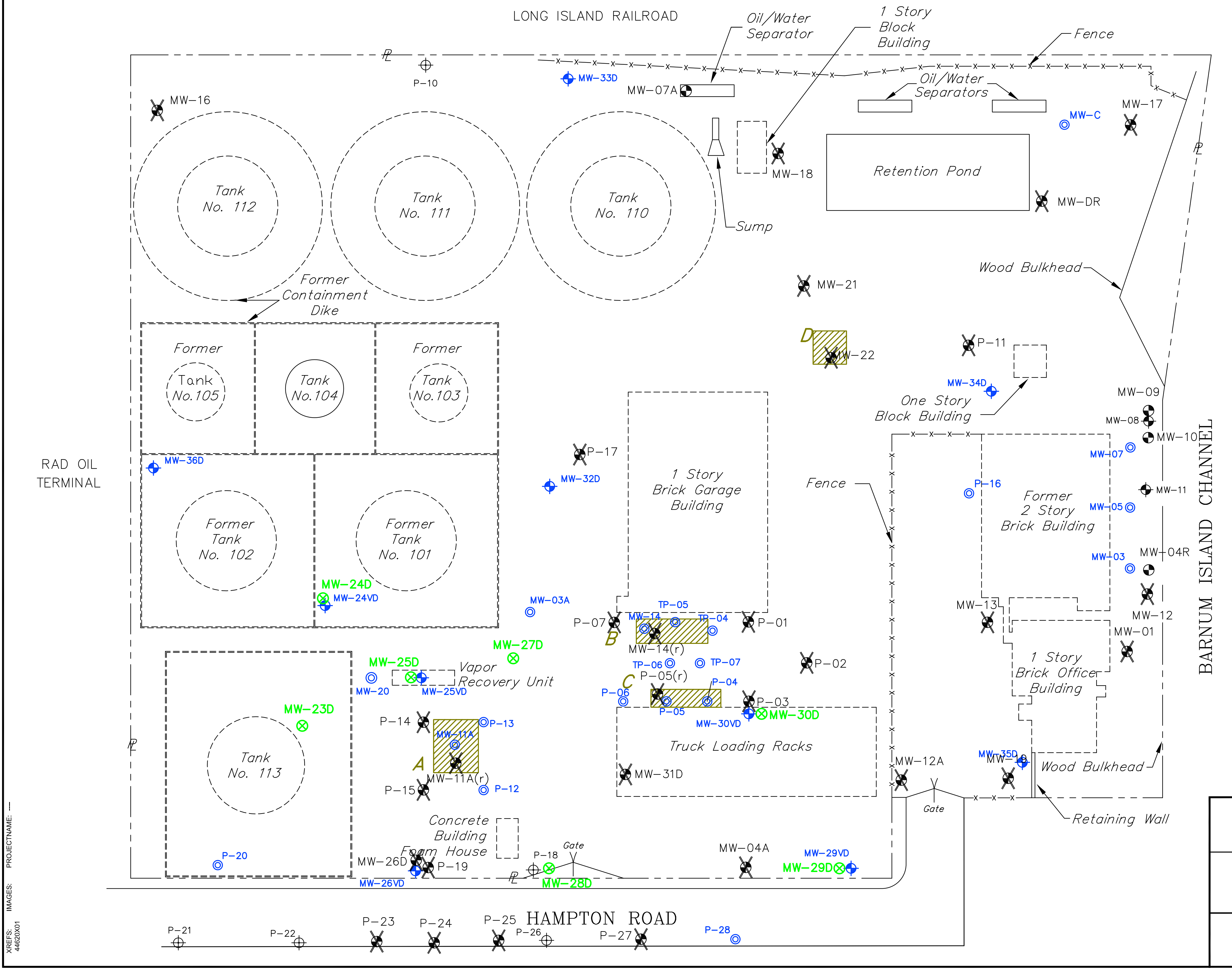


GULF OIL TERMINAL
(CHEVRON FACILITY #6518040)
OCEANSIDE, NEW YORK

SITE LOCATION MAP

ARCADIS

FIGURE
1



LEGEND

⌖	TEMPORARY PIEZOMETER (INSTALLED BY LBG)
⌖	DEEP UNDERLYING SAND WELLS
⌖	EXISTING MONITORING WELL
◎	ABANDONED MONITORING POINTS
⊗	PAIRED WELL LOCATIONS
⊗	DESTROYED MONITORING POINTS (MARCH – AUGUST 2005 GULF SITE DEMOLITION)
▨	EXCAVATION LOCATIONS (OCTOBER 2002)

SOURCE:

1. SITE PLAN DERIVED FROM STORB, INC. DRAWING ENTITLED "PAVING PLAN–GULF OIL COMPANY–U.S., OCEANSIDE TERMINAL" AUGUST 7, 1976.
2. MONITORING WELL LOCATIONS SURVEYED BY A. TAY.
3. DEEP UNDERLYING SAND WELLS ARE APPROXIMATE.

0 60' 120'
GRAPHIC SCALE

GULF OIL TERMINAL
(CHEVRON FACILITY #6518040)
OCEANSIDE, NEW YORK

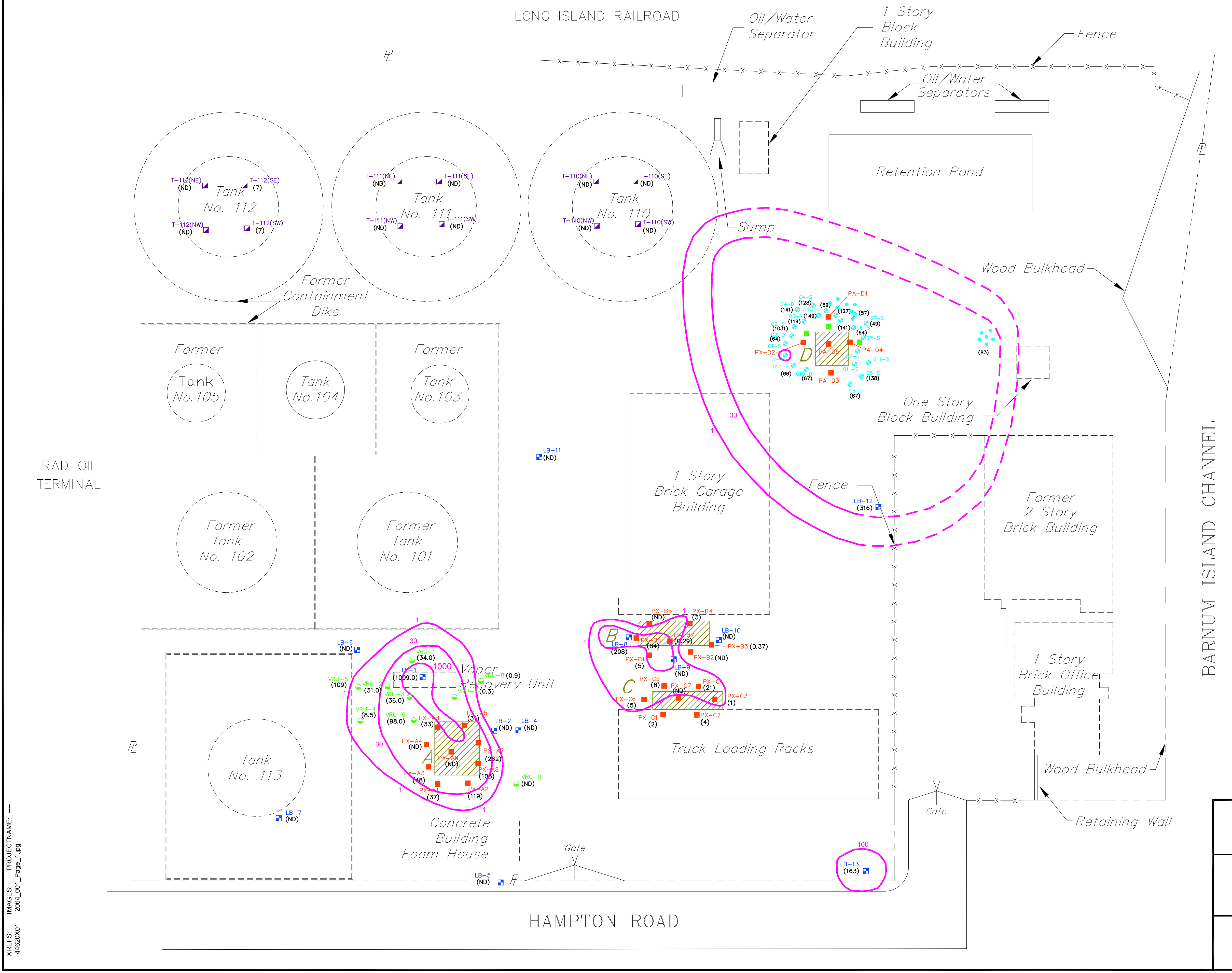
SITE PLAN

ARCADIS

FIGURE
2

CITY:(Reqd) DIV/GROUP:(Reqd) DB:(Reqd) LD:(Opt) PIC:(Opt) PM:(Reqd) TM:(Opt) LYR:(Opt)ON=*;OFF=*REF* G:\ENVCAD\Mahwah\ACT\B0047517\0003\00001\2010-05\4751731B3.dwg LAYOUT: 3 SAVED: 5/5/2010 3:48 PM ACADVER: 17.1S (LMS TECH) PAGESETUP: ---- PLOTSTYLETABLE: PLTHALF_MHWH.CTB PLOTTED: 5/5/2010 3:48 PM BY: GONZALEZ, JAMES

XREFS: 44620X01 IMAGES: 2064_001_Page_1.jpg PROJECTNAME: ----



LEGEND

●	2005 BBL SOIL BORING LOCATION
●	2005 BBL BORING LOCATION
🟩	2005 BBL POST EXCAVATION BORING LOCATION
▨	2004 LANGAN SOIL BORING LOCATION
●	2004 BBL TEMPORARY INJECTION WELLS
▨	2003 SOIL SAMPLING TANK LOCATIONS
🟥	2002 BBL POST EXCAVATION BORING LOCATION
—	FILL VOC SOIL CONTOUR (PPM)
(316)	TOTAL VOC CONCENTRATION (PPM)
▨	EXCAVATION LOCATIONS (OCTOBER 2002)

SOURCE:

1. SITE PLAN DERIVED FROM STORB, INC. DRAWING ENTITLED "PAVING PLAN–GULF OIL COMPANY–U.S., OCEANSIDE TERMINAL" AUGUST 7, 1976.
2. MONITORING WELL LOCATIONS SURVEYED BY A. TAY.
3. DEEP UNDERLYING SAND WELLS ARE APPROXIMATE.

0 60' 120'

GRAPHIC SCALE

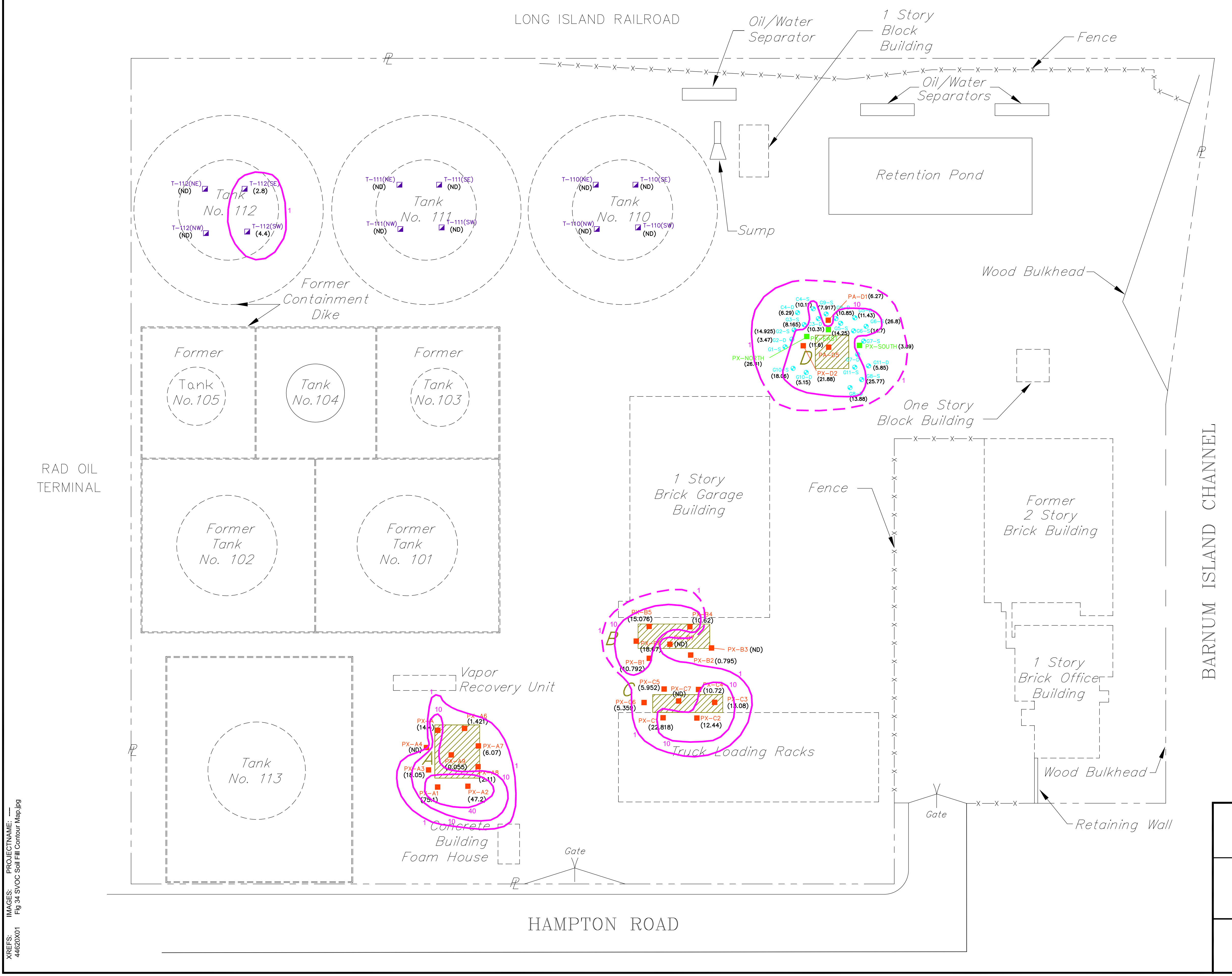
GULF OIL TERMINAL
(CHEVRON FACILITY #6518040)
OCEANSIDE, NEW YORK

SOIL TOTAL VOC CONTOURS FOR FILL MATRIX

ARCADIS

FIGURE
3

CITY:(Reqd) DIV/GROUP:(Reqd) DB:(Reqd) LD:(Opt) PIC:(Opt) PM:(Reqd) TM:(Opt) LYR:(Opt)ON=*;OFF=*REF*
G:\ENVCAD\Mahwah\ACT\B0047517\0003\00001\2010-05\4751731B4.dwg LAYOUT: 4 SAVED: 5/5/2010 5:04 PM ACADVER: 17.1S (LMS TECH) PAGESETUP: ---- PLOTSTYLETABLE: PLTHALF_MHWH.CTB PLOTTED: 5/5/2010 5:04 PM BY: GONZALEZ, JAMES
XREFS: IMAGES: PROJECTNAME: ----
44620X01 Fig 34 SVOC Soil Fill Contour Map.jpg



LEGEND

●	2005 BBL BORING LOCATION
🟩	2005 BBL POST EXCAVATION BORING LOCATION
◪	2003 SOIL SAMPLING TANK LOCATIONS
🟥	2002 BBL POST EXCAVATION BORING LOCATION
—	SVOC EXCEEDANCE CONTOUR IN FILL (PPM)
(6.27)	SVOC TOTAL CONCENTRATION (PPM)
▨	EXCAVATION LOCATIONS (OCTOBER 2002)

SOURCE:

1. SITE PLAN DERIVED FROM STORB, INC. DRAWING ENTITLED "PAVING PLAN-GULF OIL COMPANY-U.S., OCEANSIDE TERMINAL" AUGUST 7, 1976.
2. MONITORING WELL LOCATIONS SURVEYED BY A. TAY.
3. DEEP UNDERLYING SAND WELLS ARE APPROXIMATE.

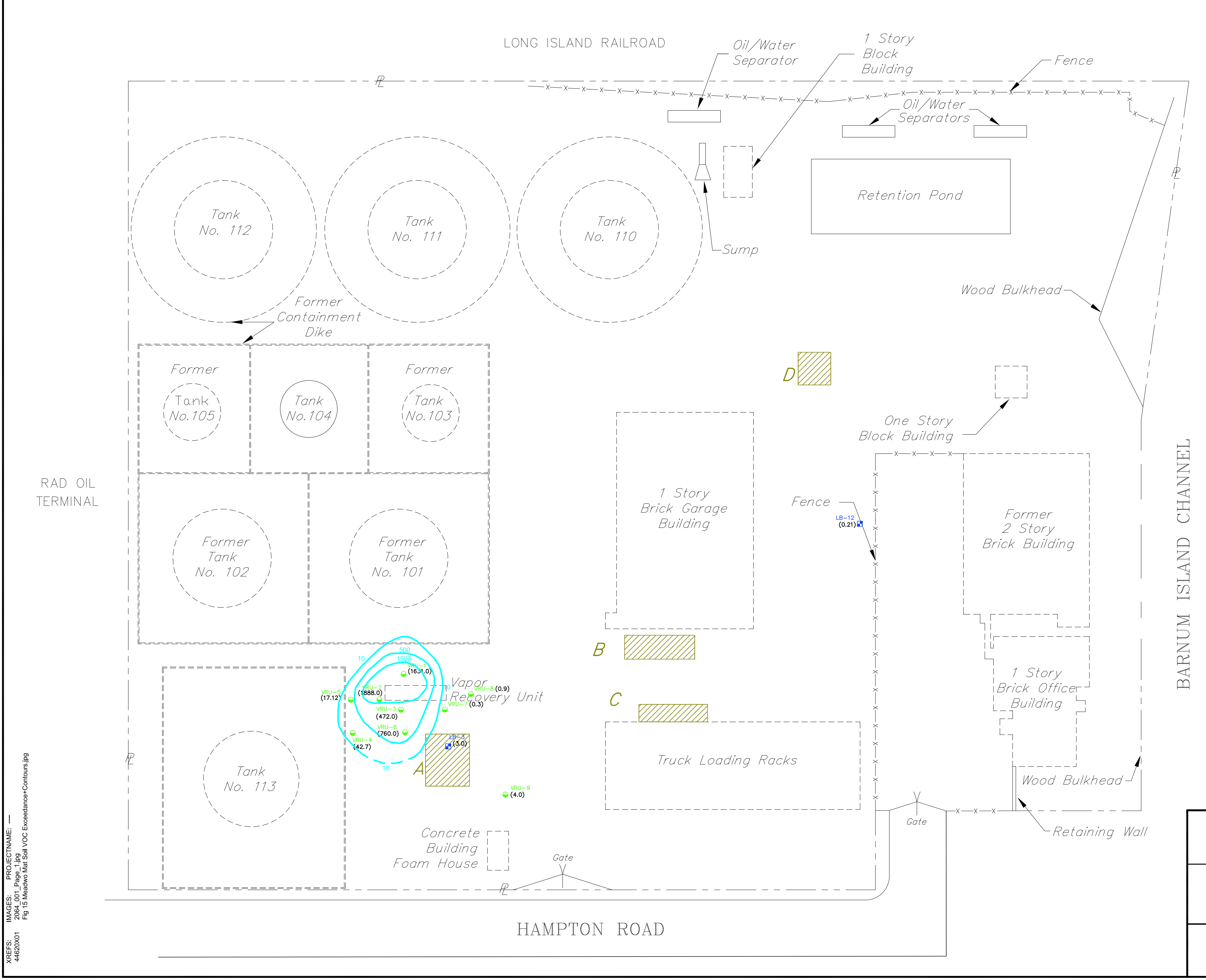
0 60' 120'
GRAPHIC SCALE

GULF OIL TERMINAL
(CHEVRON FACILITY #6518040)
OCEANSIDE, NEW YORK

SOIL TOTAL SVOC CONTOUR MAP IN FILL MATRIX

ARCADIS

FIGURE
4



LEGEND

●	2005 BBL SOIL BORING LOCATION
▨	2004 LANGAN SOIL BORING LOCATION
—	MEADOW MAT VOC SOIL CONTOUR (PPM)
(4.0)	TOTAL VOC CONCENTRATION
▨	EXCAVATION LOCATIONS (OCTOBER 2002)

SOURCE:

1. SITE PLAN DERIVED FROM STORB, INC. DRAWING ENTITLED "PAVING PLAN–GULF OIL COMPANY–U.S., OCEANSIDE TERMINAL" AUGUST 7, 1976.
2. MONITORING WELL LOCATIONS SURVEYED BY A. TAY.
3. DEEP UNDERLYING SAND WELLS ARE APPROXIMATE.

0 60' 120'
GRAPHIC SCALE

GULF OIL TERMINAL
(CHEVRON FACILITY #6518040)
OCEANSIDE, NEW YORK

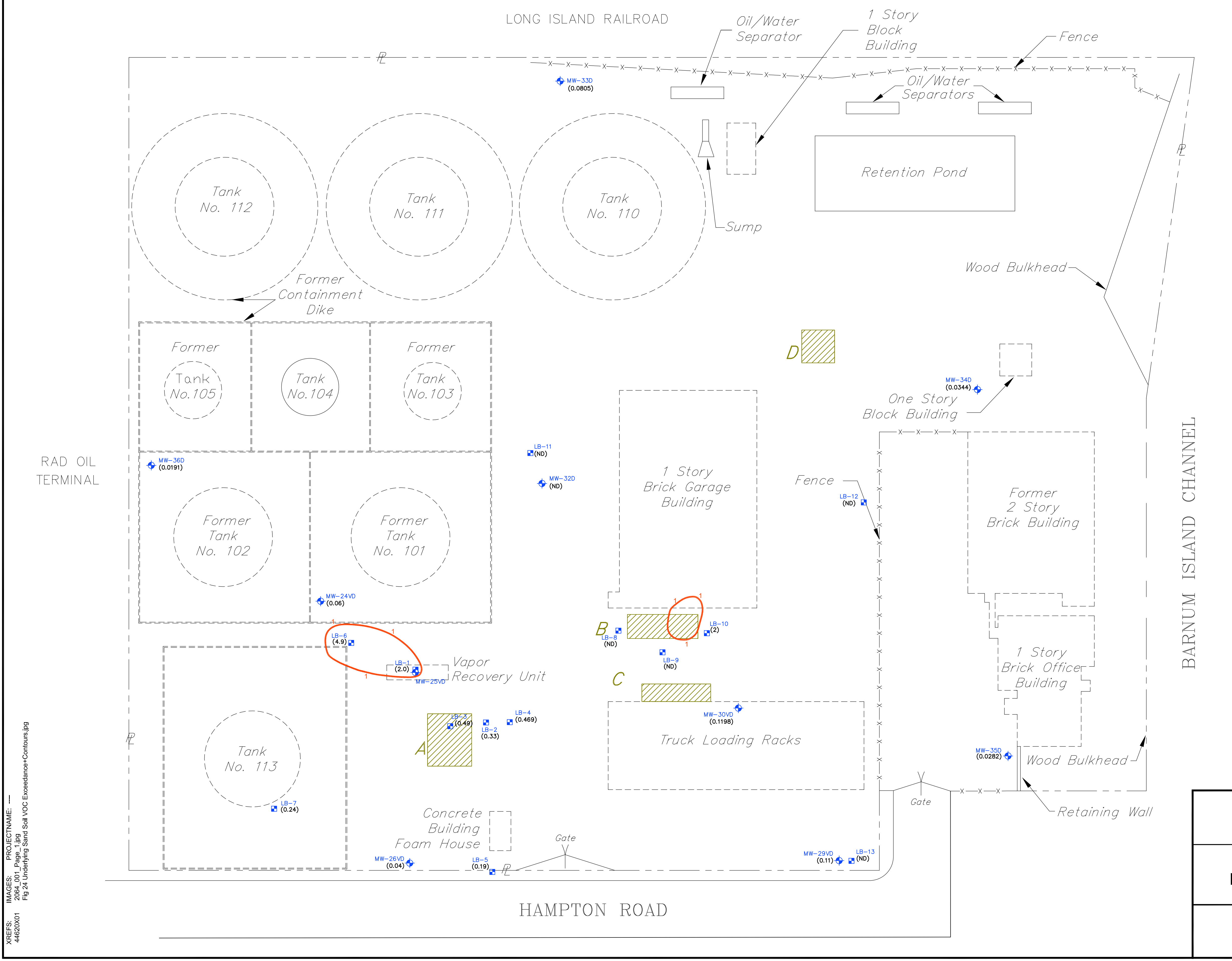
SOIL TOTAL VOC CONTOURS FOR MEADOW MAT MATRIX

ARCADIS

FIGURE
5

CITY:(Reqd) DIV/GROUP:(Reqd) DB:(Reqd) LD:(Opt) PIC:(Opt) PM:(Reqd) TM:(Opt) LYR:(Opt)ON=*;OFF=*REF*
G:\ENVCAD\Mahwah\ACT\B0047517\0003\00001\2010-05\4751731B5.dwg LAYOUT: 5 SAVED: 5/6/2010 11:45 AM ACADVER: 17.1S (LMS TECH) PAGESETUP: ---- PLOTSTYLETABLE: PLTHALF_MHWH.CTB PLOTTED: 5/6/2010 11:45 AM BY: GONZALEZ, JAMES

XREFS: IMAGES: PROJECTNAME: ----
44620X01 2064_001_Page_1.jpg
Fig 15 Meadwo Mat Soil VOC Exceedance+Contours.jpg



LEGEND

⌖	2007 LANGAN CHLORINATED VOC DUE DILIGENCE INVESTIGATION WELLS
▣	2004 LANGAN SOIL BORING LOCATION
(0.0805)	TOTAL VOCs (PPM) CONCENTRATION IN UNDERLYING SAND
(ND)	NOT DETECTED
🟥	UNDERLYING SAND VOC CONTOUR (PPM)
▨	EXCAVATION LOCATIONS (OCTOBER 2002)

SOURCE:

1. SITE PLAN DERIVED FROM STORB, INC. DRAWING ENTITLED "PAVING PLAN−GULF OIL COMPANY−U.S., OCEANSIDE TERMINAL" AUGUST 7, 1976.
2. MONITORING WELL LOCATIONS SURVEYED BY A. TAY.
3. DEEP UNDERLYING SAND WELLS ARE APPROXIMATE.

0 60' 120'
GRAPHIC SCALE

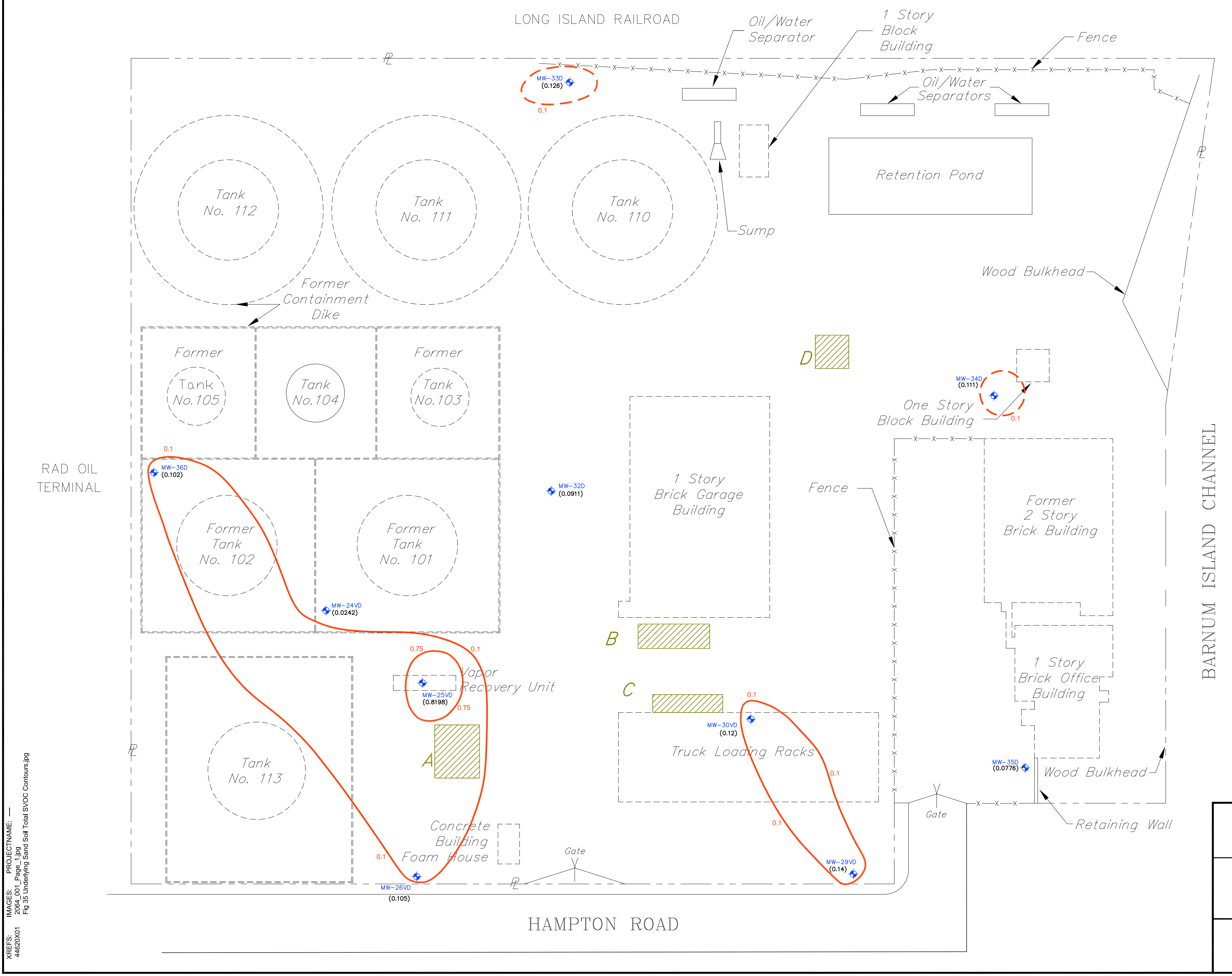
GULF OIL TERMINAL
(CHEVRON FACILITY #6518040)
OCEANSIDE, NEW YORK

**SOIL VOC CONTOURS
FOR UNDERLYING SAND MATRIX**

ARCADIS

FIGURE
6

CITY:(Reqd) DIV/GROUP:(Reqd) DB:(Reqd) LD:(Opt) PIC:(Opt) PM:(Reqd) TM:(Opt) LYR:(Opt)ON=*;OFF=*REF*
G:\ENVCAD\Mahwah\ACT\B0047517\0003\00001\2010-05\47517B6.dwg LAYOUT: 6 SAVED: 5/6/2010 11:54 AM ACADVER: 17.1S (LMS TECH) PAGESETUP: ---- PLOTSTYLETABLE: PLTHALF_MHWH.CTB PLOTTED: 5/6/2010 11:54 AM BY: GONZALEZ, JAMES
XREFS: IMAGES: PROJECTNAME: ----
4462OX01 2064_001_Page_1.jpg
Fig 24 Underlying Sand Soil VOC Exceedance+Contours.jpg



LEGEND

◆	2007 LANGAN CHLORINATED VOC DUE DILIGENCE INVESTIGATION WELLS
(0.0911)	TOTAL SVOCs (PPM) CONCENTRATIONS IN UNDERLYING SAND
—	UNDERLYING SAND VOC CONTOUR (PPM)
▨	EXCAVATION LOCATIONS (OCTOBER 2002)

SOURCE:

1. SITE PLAN DERIVED FROM STORB, INC. DRAWING ENTITLED "PAVING PLAN–GULF OIL COMPANY–U.S., OCEANSIDE TERMINAL" AUGUST 7, 1976.
2. MONITORING WELL LOCATIONS SURVEYED BY A. TAY.
3. DEEP UNDERLYING SAND WELLS ARE APPROXIMATE.

0 60' 120'
GRAPHIC SCALE

GULF OIL TERMINAL
(CHEVRON FACILITY #6518040)
OCEANSIDE, NEW YORK

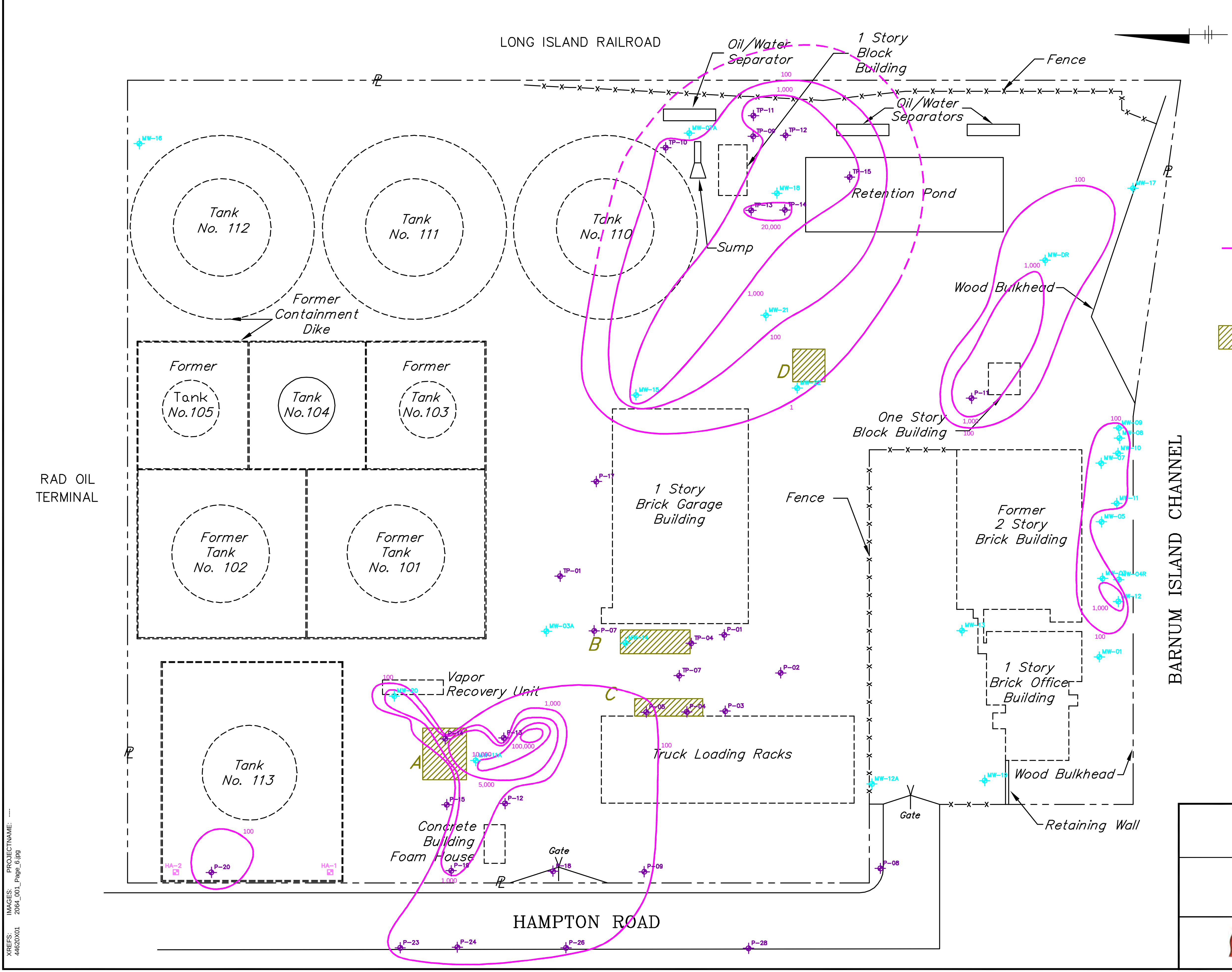
SOIL TOTAL SVOC MAP
IN UNDERLYING SAND MATRIX

ARCADIS

FIGURE
7

CITY:(Reqd) DIV/GROUP:(Reqd) DB:(Reqd) LD:(Opt) PIC:(Opt) PM:(Reqd) TM:(Opt) LYR:(Opt)ON=*;OFF=*REF*
G:\ENVCAD\Mahwah\ACT\B0047517\0003\00001\2010-05\47517317B7.dwg LAYOUT: 7 SAVED: 5/6/2010 11:59 AM ACADVER: 17.1S (LMS TECH) PAGESETUP: ---- PLOTSTYLETABLE: PLTHALF_MHWH.CTB PLOTTED: 5/6/2010 11:59 AM BY: GONZALEZ, JAMES

XREFS: IMAGES: PROJECTNAME: ---
44620X01 2064_001_Page_1.jpg
Fig 35 Underlying Sand Soil Total SVOC Contours.jpg



LEGEND

⊠	2000 LBG HAND AUGER MONITORING WELL
⌖	1996–2004 LBG/BBL MONITORING WELL LOCATION
⌖	1996 EDER TEMPORARY WELL LOCATION
⌖	1996 BBL TEMPORARY PIEZOMETER LOCATION
—	GROUND WATER VOC CONTOUR IN FILL (PPM)
(316)	TOTAL VOC CONCENTRATION (PPM)
▨	EXCAVATION LOCATIONS (OCTOBER 2002)

SOURCE:

1. SITE PLAN DERIVED FROM STORB, INC. DRAWING ENTITLED "PAVING PLAN–GULF OIL COMPANY–U.S., OCEANSIDE TERMINAL" AUGUST 7, 1976.
2. MONITORING WELL LOCATIONS SURVEYED BY A. TAY.
3. DEEP UNDERLYING SAND WELLS ARE APPROXIMATE.

0 60' 120'

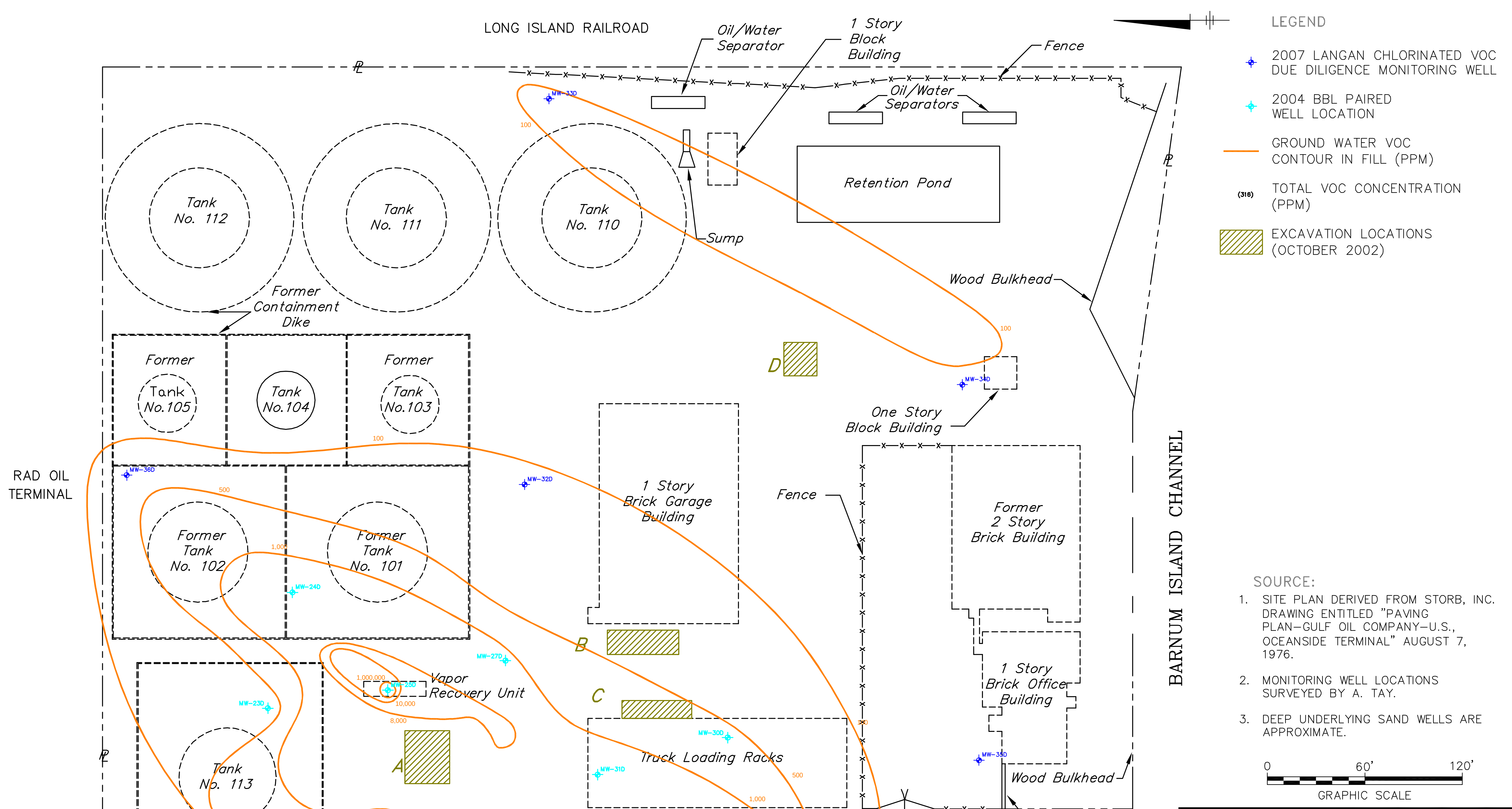
GRAPHIC SCALE

GULF OIL TERMINAL
(CHEVRON FACILITY #6518040)
OCEANSIDE, NEW YORK

GROUNDWATER VOC EXCEEDANCE CONTOURS FOR FILL MATRIX

ARCADIS

FIGURE
8



LEGEND

✦	2007 LANGAN CHLORINATED VOC DUE DILIGENCE MONITORING WELL
✦	2004 BBL PAIRED WELL LOCATION
—	GROUND WATER VOC CONTOUR IN FILL (PPM)
(316)	TOTAL VOC CONCENTRATION (PPM)
▨	EXCAVATION LOCATIONS (OCTOBER 2002)

SOURCE:

1. SITE PLAN DERIVED FROM STORB, INC. DRAWING ENTITLED "PAVING PLAN–GULF OIL COMPANY–U.S., OCEANSIDE TERMINAL" AUGUST 7, 1976.
2. MONITORING WELL LOCATIONS SURVEYED BY A. TAY.
3. DEEP UNDERLYING SAND WELLS ARE APPROXIMATE.

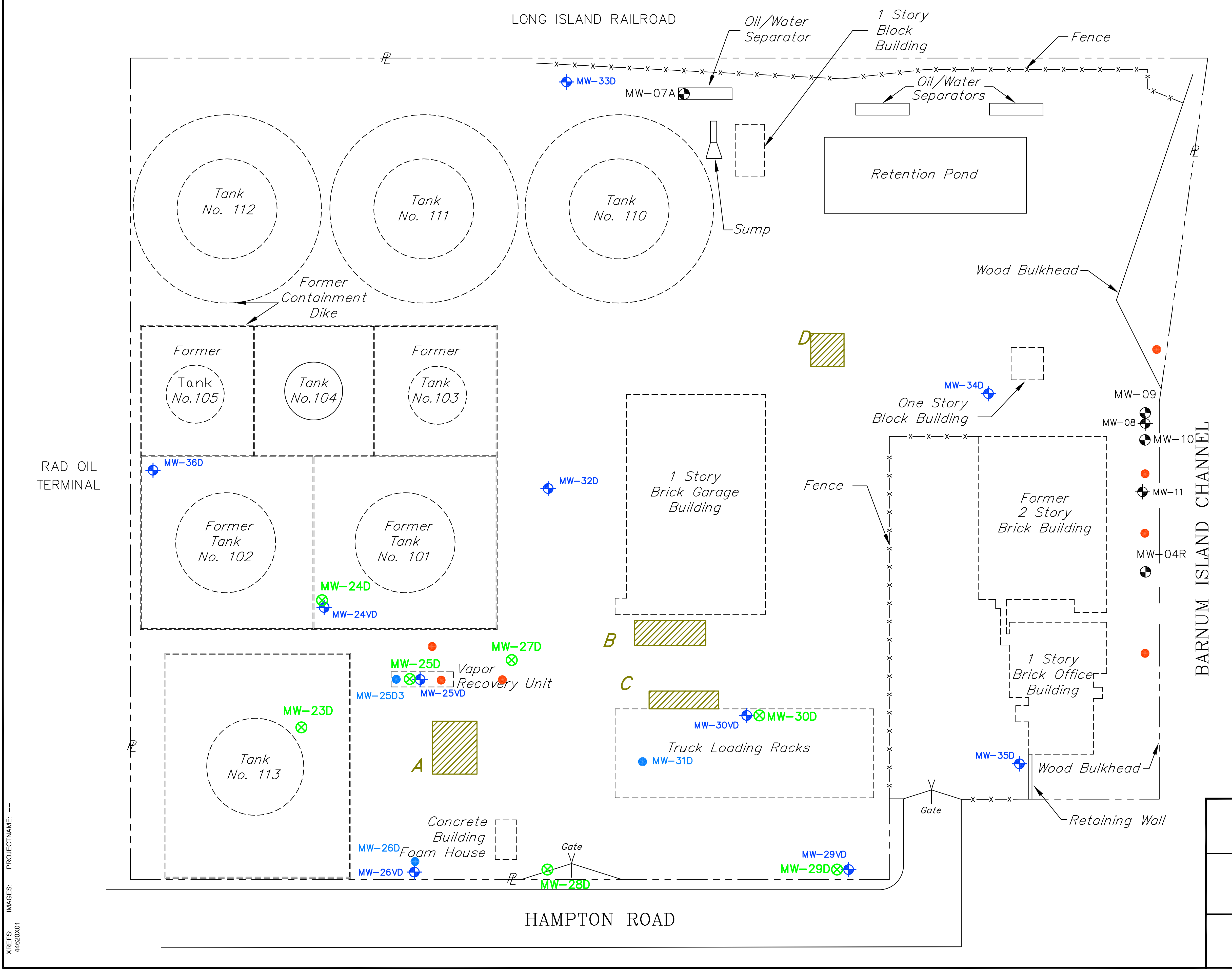
GULF OIL TERMINAL
(CHEVRON FACILITY #6518040)
OCEANSIDE, NEW YORK

GROUNDWATER VOC CONTOUR FOR UNDERLYING SAND

ARCADIS

FIGURE
9

CITY:(Reqd) DIV/GROUP:(Reqd) DB:(Reqd) LD:(Opt) PIC:(Opt) PM:(Reqd) TM:(Opt) LYR:(Opt)ON=*;OFF=*REF*
G:\ENVCAD\Melville-NY\RETURN-TO\Mahwah-NJ\B0047517\0003\00001\47517S189.dwg LAYOUT: 9 SAVED: 5/6/2010 4:31 PM ACADVER: 17.1S (LMS TECH) PAGESETUP: ---- PLOTSTYLETABLE: ARCADIS_MELVILLE.CTB PLOTTED: 5/6/2010 4:31 PM BY: SANCHEZ, ADRIAN
XREFS: 44620X01 IMAGES: 2064_001_Page_7.jpg PROJECTNAME: ----



LEGEND

⊕ DEEP UNDERLYING SAND WELLS

⊕ EXISTING MONITORING WELL

⊗ PAIRED WELL LOCATIONS

● PROPOSED SHALLOW SAND FILL MONITORING WELLS

● PROPOSED DEEP UNDERLYING SAND WELLS

▨ EXCAVATION LOCATIONS (OCTOBER 2002)

SOURCE:

1. SITE PLAN DERIVED FROM STORB, INC. DRAWING ENTITLED "PAVING PLAN-GULF OIL COMPANY-U.S., OCEANSIDE TERMINAL" AUGUST 7, 1976.
2. MONITORING WELL LOCATIONS SURVEYED BY A. TAY.
3. DEEP UNDERLYING SAND WELLS ARE APPROXIMATE.

0 60' 120'
GRAPHIC SCALE

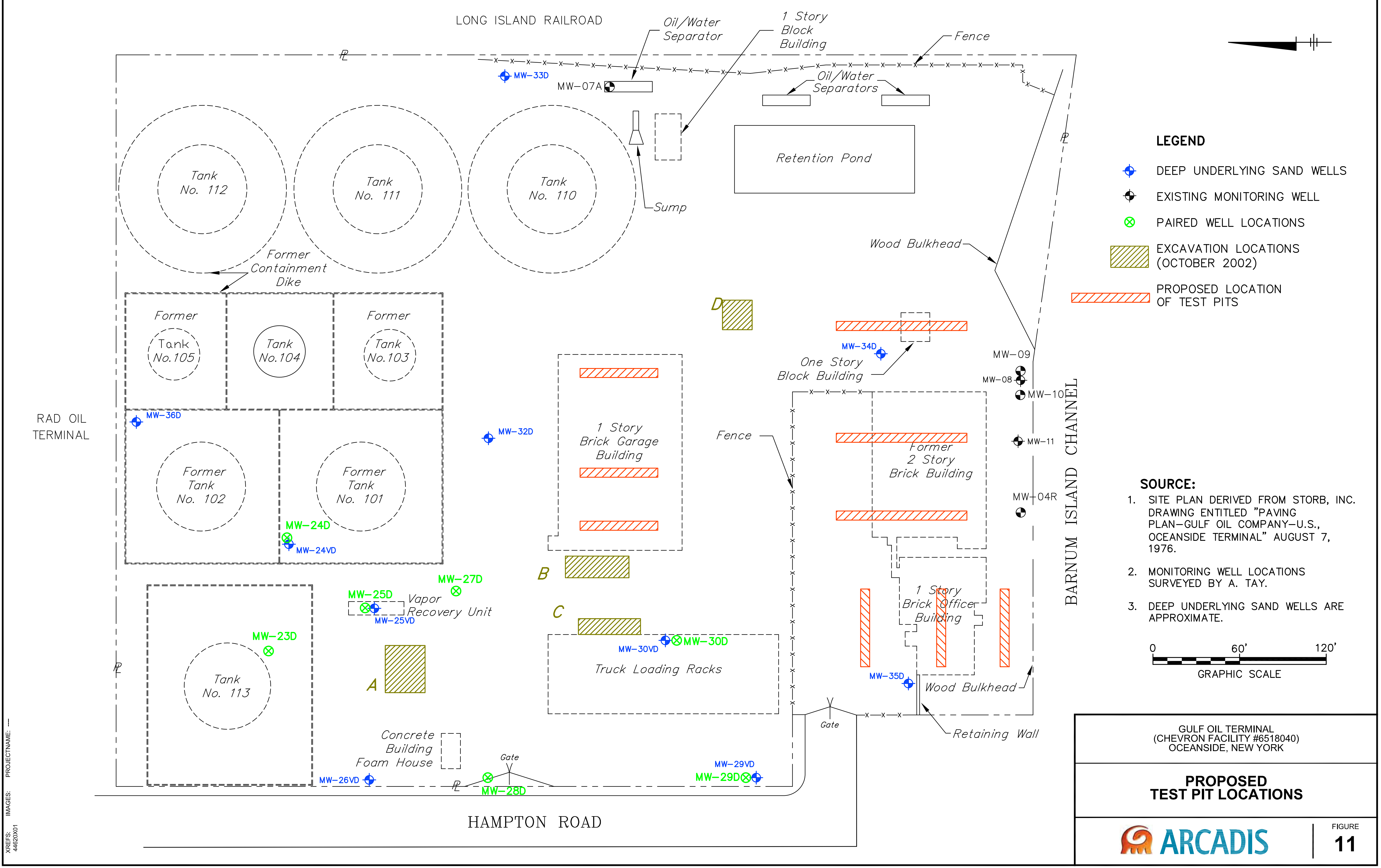
GULF OIL TERMINAL
(CHEVRON FACILITY #6518040)
OCEANSIDE, NEW YORK

PROPOSED MONITORING WELL LOCATIONS

ARCADIS

FIGURE
10

CITY:(Reqd) DIV/GROUP:(Reqd) DB:(Reqd) LD:(Opt) PIC:(Opt) PM:(Reqd) TM:(Opt) LYR:(Opt)ON=*;OFF=*REF*
G:\ENVCAD\Mahwah\ACT\B0047517\0003\00001\2010-05\4751731B10.dwg LAYOUT: 10 SAVED: 5/6/2010 2:28 PM ACADVER: 17.1S (LMS TECH) PAGESETUP: ---- PLOTSTYLETABLE: PLTFULL.CTB PLOTTED: 5/6/2010 2:29 PM BY: GONZALEZ, JAMES
XREFS: 44620X01 IMAGES: PROJECTNAME: ---



SOURCE:

1. SITE PLAN DERIVED FROM STORB, INC. DRAWING ENTITLED "PAVING PLAN-GULF OIL COMPANY-U.S., OCEANSIDE TERMINAL" AUGUST 7, 1976.
2. MONITORING WELL LOCATIONS SURVEYED BY A. TAY.
3. DEEP UNDERLYING SAND WELLS ARE APPROXIMATE.

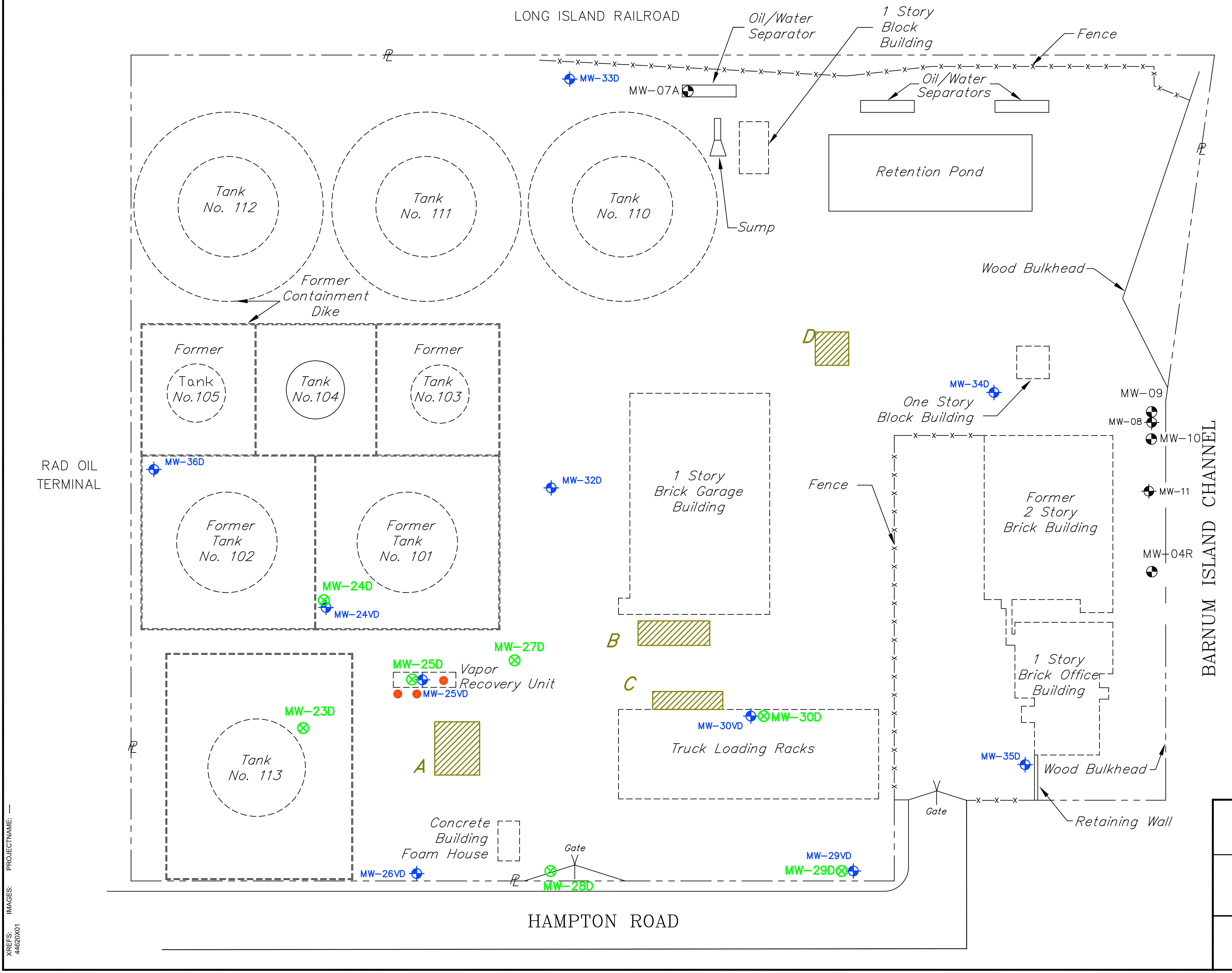
GULF OIL TERMINAL
(CHEVRON FACILITY #6518040)
OCEANSIDE, NEW YORK

PROPOSED TEST PIT LOCATIONS

ARCADIS

FIGURE
11

CITY:(Reqd) DIV/GROUP:(Reqd) DB:(Reqd) LD:(Opt) PIC:(Opt) PM:(Reqd) TM:(Opt) LYR:(Opt)ON=*;OFF=*REF*
G:\ENVCAD\Mahwah\ACT\B0047517\0003\00001\2010-05\4751731B11.dwg LAYOUT: 11 SAVED: 5/18/2010 10:20 AM ACADVER: 17.1S (LMS TECH) PAGESETUP: ---- PLOTSTYLETABLE: PLTFULL.CTB PLOTTED: 5/18/2010 10:20 AM BY: GONZALEZ, JAMES
XREFS: IMAGES: PROJECTNAME: ----
44620X01



LEGEND

⌖	DEEP UNDERLYING SAND WELLS
⊕	EXISTING MONITORING WELL
⊗	PAIRED WELL LOCATIONS
●	PROPOSED SOIL BORING LOCATIONS
▨	EXCAVATION LOCATIONS (OCTOBER 2002)

SOURCE:

1. SITE PLAN DERIVED FROM STORB, INC. DRAWING ENTITLED "PAVING PLAN-GULF OIL COMPANY-U.S., OCEANSIDE TERMINAL" AUGUST 7, 1976.
2. MONITORING WELL LOCATIONS SURVEYED BY A. TAY.
3. DEEP UNDERLYING SAND WELLS ARE APPROXIMATE.

0 60' 120'
GRAPHIC SCALE

GULF OIL TERMINAL
(CHEVRON FACILITY #6518040)
OCEANSIDE, NEW YORK

PROPOSED SOIL BORING LOCATIONS

ARCADIS

FIGURE
12

CITY:(Reqd) DIV/GROUP:(Reqd) DB:(Reqd) LD:(Opt) PIC:(Opt) PM:(Reqd) TM:(Opt) LYR:(Opt)ON=*;OFF=*REF*
G:\ENVCAD\Mahwah\ACT\B0047517\0003\00001\2010-05\4751731B12.dwg LAYOUT: 12 SAVED: 5/6/2010 2:21 PM ACADVER: 17.1S (LMS TECH) PAGESETUP: ---- PLOTSTYLETABLE: PLTFULL.CTB PLOTTED: 5/6/2010 2:21 PM BY: GONZALEZ, JAMES
XREFS: 4462OX01 IMAGES: PROJECTNAME: ----

APPENDICES

Appendix A

Quality Assurance Project Plan

ARCADIS
Infrastructure, environment, buildings

Imagine the result

Chevron Environmental Management Company

Quality Assurance Project Plan

Former Gulf Oil Terminal Oceanside
Town of Hempstead, New York
NYSDEC Site No. 130165

June 2010

Executive Summary **i**

Acronyms and Abbreviations **iii**

1. Project Organization **1**

1.1 Project Organization 1

1.1.1 Overall Project Management 1

1.1.2 Task Managers 1

1.2 Team Member Responsibilities 2

1.2.1 Chevron Environmental Management Company 2

1.2.2 ARCADIS 2

1.2.3 Analytical Laboratories 4

1.2.4 NYSDEC 5

2. Project Background **7**

2.1 Site Location and Description 7

2.1.1 Site History 7

3. Project Description **8**

3.1 Remedial Investigation Objectives 8

3.2 Remedial Investigation Scope of Work 8

4. Quality Objectives and Criteria for Measurement Data **11**

4.1 Step 1: State the Problem 11

4.2 Step 2: Identify the Goal of the Study 11

4.3 Step 3: Identify Information Inputs 11

4.4 Step 4: Define the Boundaries of the Study 11

4.5 Step 5: Developing the Analytical Approach 12

4.6 Step 6: Specify Performance or Acceptance Criteria 12

4.7 Data Categories 12

4.7.1 Screening Data 12

4.7.2 Screening Data with Definitive Confirmation 13

4.7.3 Definitive Data 13

4.7.3.1 Level 1 – Minimal Reporting 13

4.7.3.2 Level 2 – Modified Reporting 13

4.7.3.3 Level 3 – Full Reporting: 14

4.8 Field Investigations 14

5. Special Training Requirements/Certification 15

6. Documentation and Records 16

6.1 General 16

6.2 Sample Designation System 16

6.2.1 Sample Codes 16

6.2.2 Field Documentation 17

6.3 Laboratory Documentation Files 18

6.3.1 Laboratory Project Files 18

6.3.2 Laboratory Logbooks 18

6.3.3 Computer Tape and Hard Copy Storage 18

6.4 Data Reporting Requirements 18

6.4.1 Field Data Reporting 19

6.4.2 Laboratory Data Reporting 19

7. Sampling Process Design 21

8. Sampling Method Requirements 22

9. Sample Handling and Custody Requirements 23

9.1 Sample Containers and Preservation 23

9.2 Field Custody Procedures 23

9.2.1 Field Logbooks 23

9.2.2 Sample Labeling 24

9.2.3 Field COC Forms 25

9.3 Management of Investigation-Derived Materials and Wastes 25

9.4 Packing, Handling and Shipping Requirements 26

9.5 Laboratory Custody Procedures 27

9.5.1 General 27

9.5.2 Sample Receipt and Storage 27

9.5.3 Sample Analysis 28

9.5.4 Sample Storage Following Analysis 28

10. Analytical Method Requirements 29

10.1 Field Parameters and Methods 29

10.2 Laboratory Parameters and Methods 29

10.2.1 General 29

10.2.2 Remedial Investigation/Feasibility Study Sample Matrices 29

10.2.2.1 Groundwater 29

10.2.2.2 Soil 29

10.3 Analytical Requirements 30

11. Quality Control Requirements 31

11.1 Quality Assurance Indictors 31

11.1.1 Representativeness 31

11.1.2 Comparability 31

11.1.3 Completeness 32

11.1.4 Precision 32

11.1.5 Accuracy 32

11.2 Field Quality Control Checks 32

11.2.1 Field Measurements 32

11.2.2 Sample Containers 32

11.2.3 Field Duplicates 33

11.2.4 Rinse Blanks 33

11.2.5 Trip Blanks 33

11.3 Analytical Laboratory Quality Control Checks 33

11.3.1 General 33

11.3.2 Method Blanks 34

11.3.3 MS/MSDs 34

11.3.4 Surrogate Spikes 34

11.3.5 Laboratory Duplicates 35

11.3.6 Calibration Standards 35

11.3.7 Internal Standards 35

11.3.8 Reference Standards/Control Samples 35

11.4 Data Precision Assessment Procedures 36

11.5 Data Accuracy Assessment Procedures 36

11.6 Data Completeness Assessment Procedures 37

12. Instrument/Equipment Testing, Inspection and Maintenance Requirements 38

12.1 General 38

12.2 Field Instruments and Equipment 38

12.2.1 Equipment Maintenance 38

12.3 Laboratory Instruments and Equipment 39

12.3.1 General 39

12.3.2 Instrument Maintenance 39

12.3.3 Equipment Monitoring 39

13. Instrument Calibration and Frequency 40

13.1 Field Instruments and Equipment 40

13.2 Laboratory Instrument and Equipment 40

13.2.1 VOCs 41

13.2.2 SVOCs 41

13.2.3 Metals 41

13.2.4 TOC 41

14. Inspection/Acceptance Requirements for Supplies and Consumables 42

15. Data Acquisition Requirements for Non-Direct Measurements 43

16. Data Management 44

16.1 Sample Designation System 44

16.2 Field Activities 45

16.2.1 Field Documentation 45

16.2.1.1 Field Logs 45

16.2.1.2 COC Forms 45

16.2.1.3 Instrument Calibration Records 46

16.2.2 Data Security 46

16.3 Sample Management and Tracking 46

16.4 Data Management System 47

16.4.1 Computer Hardware 47

16.4.2 Computer Software 47

16.4.3 Survey Information 48

16.4.4 Field Observations 48

16.4.5 Analytical Results 49

16.4.6 Data Analysis and Reporting 50

16.5 Document Control and Inventory 50

17. Assessment and Response Actions 52

17.1 Field Audits 52

17.2 Laboratory Audits 52

17.3 Corrective Action 53

17.3.1 Field Procedures 53

17.3.2 Laboratory Procedures 53

18. Reports to Management 55

18.1 Internal Reporting 55

18.2 Remedial Investigation/Feasibility Study Reporting 55

19. Data Reduction and Review 56

19.1 Field Data Reduction and Review 56

19.1.1 Field Data Reduction 56

19.1.2 Field Data Review 56

19.2 Laboratory Data Reduction and Review 57

19.2.1 Laboratory Data Reduction 57

19.2.2 Laboratory Data Review 57

19.3 Data Validation and Verification 57

20. Data Validation and Verification 58

21. Reconciliation with User Requirements 60

22. References 61

Tables

1 Environmental and Quality Control Analyses

2 Analytical Quality Control Limits

3 Parameters, Methods and Target Reporting Limits

4 Sample Containers, Preservation Methods and Holding Times

5 Electronic Data Deliverable Format

Attachments

1 Laboratory Standard Operating Procedures

2 TestAmerica Quality Manual

3 Field Standard Operating Procedures

3A Chain-of-Custody, Handling, Packing, and Shipping

3B Field Equipment Decontamination

3C Soil Drilling and Sample Collection

3D Field Log Book Entries

3E Low-Flow Groundwater Purging and Sampling Procedures for Monitoring Wells

3F Investigation-Derived Waste Handling and Storage

3G Monitoring Well Installation

3H Monitoring Well Integrity Survey

3I Monitoring Well Development

3J Photoionization Detector Air Monitoring and Field Screening

3K Pump Test Standard Operating Procedures

3L Specific Capacity Testing and Data Reduction

3M Standard Groundwater Sampling for Monitoring Wells

3N Surface and Subsurface Soil Sampling Using Manual Methods

3O Test Pit Excavation (Non-Entry)

3P Water-Level and NAPL Thickness Measurements Procedures

Distribution List

Organization	Individual
ARCADIS of New York (ARCADIS)	William McCune, Dennis K. Capria
New York State Department of Environmental Conservation	Heidi Dudek
New York State Department of Health	Gary Litwin
TestAmerica	Tony Bogolin

FORMER GULF OIL TERMINAL OCEANSIDE
TOWN OF HEMPSTEAD, NEW YORK

QUALITY ASSURANCE PROJECT PLAN

Prepared By: ARCADIS

Approved: Peter Kosbohm
Project Manager – Peter Kosbohm
Chevron Environmental Management Company

Approved: ______
Project Manager – William McCune
ARCADIS

Approved: Dennis K. Capria
Quality Assurance Coordinator – Dennis Capria
ARCADIS

Approved: Tony Bogolin 6/10/10
Project Manager – Tony Bogolin
TestAmerica

Approved: Paula Benham 6/10/10
Quality Assurance Manager – Paula Benham
TestAmerica

Executive Summary

This Quality Assurance Project Plan (QAPP) was prepared for the Former Gulf Oil Terminal located in Oceanside, Town of Hempstead, New York (site). This QAPP will support future work plans for the site.

This QAPP was prepared in a manner consistent with the following reference and guidance documents:

- United States Environmental Protection Agency (USEPA) guidance document entitled EPA Requirements for Quality Assurance Project Plans, EPA-QA/R-5 (USEPA 2001), which replaces QAMS-005/80, Interim Guidance and Specifications for Preparing Quality Assurance Project Plans (USEPA 1980)

- USEPA Guidance for Quality Assurance Project Plans (USEPA 2002b)

Information contained in this QAPP has been organized into the following sections:

Section	Content
Project Management	
1	Project Organization
2	Project Background
3	Project Description
4	Quality Objectives and Criteria for Measurement Data
5	Special Training Requirements/Certification
6	Documentation and Records
Measurement/Data Acquisition	
7	Sampling Process Design
8	Sampling Method Requirements
9	Sample Handling and Custody Requirements
10	Analytical Method Requirements
11	Quality Control Requirements
12	Instrument/Equipment Testing, Inspection, and Maintenance Requirements
13	Instrument Calibration and Frequency
14	Inspection/Acceptance Requirements for Supplies and Consumables
15	Data Acquisition Requirements for Non-Direct Measurements
16	Data Management
Assessment/Oversight	
17	Assessment and Response Actions
18	Reports to Management

Section	Content
Data Validation and Usability	
19	Data Reduction and Review
20	Data Validation and Verification
21	Reconciliation with User Requirements

Each of the subjects listed above are discussed in the subsequent sections. This QAPP also contains pertinent information from the Remedial Investigation/Feasibility Study Work Plan (RI/FS WP) related to measuring and evaluating the analytical data.

Acronyms and Abbreviations

ASP	Analytical Services Protocol
BCP	Brownfield Cleanup Program
CEMC	Chevron Environmental Management Company
CFI	Cumberland Farms, Inc.
CLP	Contract Laboratory Program
COC	chain-of-custody
CVOC	chlorinated volatile organic compound
DQO	data quality objective
EDD	electronic data deliverable
GC/MS	gas chromatography/mass spectrometry
GIS	geographic information system
Gulf	Gulf Oil Limited Partnership
MS	matrix spike
MSD	matrix spike duplicate
NYSDEC	New York State Department of Environmental Conservation
OSHA	Occupational Safety and Health Administration
QA	quality assurance
QAC	Quality Assurance Coordinator
QAPP	Quality Assurance Project Plan

QA/QC	quality assurance/quality control
QC	quality control
RI	remedial investigation
RI/FS WP	Remedial Investigation /Feasibility Study Work Plan
RPD	relative percent difference
SDG	sample delivery group
site	Former Gulf Oil Terminal located in Oceanside, Town of Hempstead, New York
SOP	Standard Operating Procedure
SVOC	semivolatile organic compound
TAL	Target Analyte List
TOC	total organic carbon
USEPA	United States Environmental Protection Agency
VOC	volatile organic compound
VRU	vapor recovery unit

1. Project Organization

1.1 Project Organization

Investigations performed as part of the Remedial Investigation /Feasibility Study Work Plan (RI/FS WP) at the Former Gulf Oil Terminal located in Oceanside, Town of Hempstead, New York (site), will require integration of personnel from the organizations identified below, collectively referred to as the “project team.” The responsibilities for each member of the project team are described below.

1.1.1 Overall Project Management

On behalf of Chevron Environmental Management Company (CEMC), ARCADIS has overall responsibility for the remedial investigation (RI) activities at the site. ARCADIS personnel will perform related sampling activities, evaluate data and prepare the deliverables as specified in the RI/FS WP. Project direction will be provided by CEMC, with oversight by the New York State Department of Environmental Conservation (NYSDEC). A list of key project management personnel is provided below.

Company/Organization	Title	Name	Phone Number
NYSDEC	Project Manager	Heidi Dudek	518.402.9622
	Quality Assurance Manager	TBD	TBD
Chevron	Project Coordinators	Peter Kasbohm	770.984.3145
ARCADIS	Project Officer	John Vogeley	925.274.1100
	Project Manager	William McCune	315.671.9172
	Field Manager	Andy Korik	315.671.9353
	Quality Assurance Coordinator	Dennis K. Capria	315.671.9299
Lab – TestAmerica	Project Manager	Tony Bogolin	716.504.9822
	Quality Assurance Manager	Paula Benham	716.504.9833

1.1.2 Task Managers

The staff performing the investigations and site activities will be directed by representatives of the project team. The personnel responsible for each of the site activities are listed below.

Company/Organization	Title	Name	Phone Number
ARCADIS	Field Task Manager	Andy Korik	315.671.9353
	Health and Safety Officer	TBA	
	Database Administrator	TBA	
	Data Validator	TBA	

1.2 Team Member Responsibilities

The responsibilities of the various team members are summarized below by organization.

1.2.1 Chevron Environmental Management Company

Project Coordinator

Responsibilities and duties include:

- Provide overall direction of site actions.
- Direct ARCADIS project team members.
- Review ARCADIS work products, including data, memoranda, letters, reports and all other documents transmitted to the NYSDEC.

1.2.2 ARCADIS

Project Officer

Responsibilities and duties include:

- Oversee ARCADIS work products.
- Provide ARCADIS approval for major project deliverables.

Project Manager

Responsibilities and duties include:

- Manage and coordinate the project as defined in the RI/FS WP, focusing on the objectives of the site activities.
- Review documents prepared by ARCADIS.
- Ensure that corrective actions are taken for deficiencies cited during any audits of site activities.

Task Managers

The RI components will be managed by various Task Managers, as set forth in Section 1.1.2. Duties of each Task Manager include, as appropriate:

- Manage relevant daily activities.
- Develop, establish and maintain files on relevant site activities.
- Review data reductions from the relevant site activities.
- Perform final data review of field data reductions and reports on relevant site activities.
- Ensure that corrective actions are taken for deficiencies cited during audits of relevant site activities.
- Perform overall quality assurance/quality control (QA/QC) of the relevant portions of the site activities.
- Review relevant field records and logs.
- Instruct personnel working on relevant site activities.
- Coordinate field and laboratory schedules pertaining to relevant site activities.
- Request sample bottles from laboratory.
- Review field instrumentation, maintenance and calibration to meet quality objectives.
- Prepare reports pertaining to relevant site activities.
- Maintain field and laboratory files of notebooks/logs, data reductions and calculations; transmit originals to the Project Manager.

Field Personnel

Responsibilities and duties include:

- Perform field procedures associated with the investigations identified in the RI/FS WP.
- Perform field analyses and collect quality assurance (QA) samples.

- Calibrate, operate and maintain field equipment.
- Reduce field data.
- Maintain sample custody.
- Prepare field records and logs.

Quality Assurance Coordinator

Responsibilities and duties include:

- Review laboratory data packages.
- Oversee and interface with the analytical laboratory.
- Coordinate field QA/QC procedures with Task Managers (including audits of field activities), concentrating on field analytical measurements and practices to meet data quality objectives (DQOs).
- Review field reports.
- Perform and review audit reports.
- Prepare interim QA/QC compliance reports.
- Prepare a QA/QC report in accordance with United States Environmental Protection Agency (USEPA) Region II guidelines, which includes an evaluation of field and laboratory data and data usability reports.

1.2.3 Analytical Laboratories

General responsibilities and duties of the analytical laboratories include:

- Perform sample analyses and associated laboratory QA/QC procedures.
- Supply sampling containers and shipping cartons.
- Maintain laboratory custody of sample.
- Strictly adhere to all protocols in this Quality Assurance Project Plan (QAPP).

Project Manager

Responsibilities and duties include:

- Serve as primary communication link between ARCADIS and laboratory technical staff.
- Monitor workloads and ensure availability of resources.
- Oversee preparation of analytical reports.
- Supervise in-house chain-of-custody (COC).

Quality Assurance Manager

Responsibilities and duties include:

- Supervise personnel reviewing and inspecting all project-related laboratory activities.
- Conduct audits of all laboratory activities.

1.2.4 NYSDEC

Project Manager

Responsibilities and duties include:

- Provide NYSDEC review and approval of the RI/FS WP, supporting documents and future deliverables.
- Monitor the progress of site activities.

Quality Assurance Manager

Responsibilities and duties include:

- Review and approval of the QAPP.
- Review of the QA/QC portion of any submitted report.

- Monitor the progress of the RI/FS.

- Ensure that all activities are performed in compliance with applicable federal, state and regional requirements.

- Perform field and laboratory audits, if necessary.

2. Project Background

2.1 Site Location and Description

The site consists of 7.2 acres located at 1 Industrial Place in Oceanside, Township of Hempstead, Nassau County, New York. A Site Location Map is presented in the RI/FS WP on Figure 1. The site is currently unoccupied and all former buildings have been demolished. The site is bound to the south by Barnum’s Channel, to the east by the Long Island Rail Road, to the north by the former Exxon Petroleum Terminal and to the west by Hampton Road. A junk yard and office buildings are located west of Hampton Road, fronting Hog Island Channel. Daly Boulevard is located north of the former Exxon Petroleum Terminal and another petroleum terminal (Sprague) is located north of Daly Boulevard.

2.1.1 Site History

The site consists of three lots. A portion of the site (Lot 504) was first developed as a petroleum terminal by Gulf Oil Corporation in 1931. Lots 503 and 502 were purchased by Gulf Oil Corporation in 1950 and 1956, respectively. Chevron acquired Gulf Oil Corporation in the mid 1980s. As part of the purchase, Chevron was required to divest the northeast division. In 1986, Cumberland Farms, Inc. (CFI) purchased the northeast marketing assets of Gulf Oil Corporation from Chevron. The purchase included the Oceanside Terminal, which was transferred to CFI in May 1986. In December 1993, CFI, in a joint venture with Catamount Petroleum LP (Catamount), formed Gulf Oil Limited Partnership (Gulf). CFI owned two-thirds of Gulf as a limited partner and Catamount owned one-third of Gulf as the general partner. In September 2005, CFI exercised its option to buy out Catamount and reorganized Gulf under a new general partner. However, ownership percentages of Gulf did not change during the reorganization. Gulf remains as the current owner/operator of the site.

Operation of the petroleum terminal ceased in the early 1990s. Demolition of the former petroleum terminal aboveground storage tanks was completed in 2003. The remaining on-site buildings (maintenance building, office building and several small buildings associated with historical petroleum terminal operations) were demolished in 2005. The site is currently vacant.

The site has undergone extensive investigation and remediation since the 1990s under NYSDEC oversight (Spill No. 92-03883). In January 2007, the NYSDEC accepted Lowe’s Home Centers, Inc. (Lowe’s) as a volunteer under the Brownfield Cleanup Program (BCP) – Site No. C130165. Following the decision by Lowe’s to withdraw from the BCP in 2009, the NYSDEC transferred the site into the Hazardous Waste Program (State Superfund Site). An Order on Consent and Administrative Settlement was executed between the NYSDEC and CEMC on December 23, 2009 (Index # W3-1142-09-08, Site # 130165).

3. Project Description

3.1 Remedial Investigation Objectives

The proposed RI objectives for this site are:

- Complete supplemental investigations to address data gaps and to horizontally and vertically delineate soil and groundwater impacts. The results will be used to estimate the contaminant mass and distribution in the shallow sand fill, the underlying meadow mat, and the deep sand.

- Conduct additional pre-remedial design investigation (e.g., aquifer testing, bench-scale testing, specific-capacity testing, slug testing) to adequately understand the hydrogeologic and geochemical site characteristics to evaluate, select and design the appropriate remedial design for this site.

- Characterize the sources of contamination, migration pathways and actual or potential receptors of contaminants on or through air, soil, sediment, groundwater and surface water.

- Collect and evaluate additional data, as needed, to evaluate the remedial action alternatives.

- Collect data, as needed, to evaluate the actual and potential threats to public health and the environment.

- Collect and evaluate information for a Fish and Wildlife Resource Impact Analysis.

3.2 Remedial Investigation Scope of Work

The RI scope of work consists of the following:

1. Inspect existing monitoring wells on site to assess their conditions (e.g., number of existing wells, suitability for sampling). If any of the mapped existing wells are found to have been destroyed or are unsuitable for sampling and monitoring, CEMC will assess the need for well replacement.

2. Install additional monitoring wells in the shallow sand fill layer and the deep sand layer for vertical delineation of petroleum impacts as well as chlorinated volatile organic compounds (CVOCs). ARCADIS proposes to install seven monitoring wells in the shallow sand fill layer: four monitoring wells along the bulkhead and three monitoring wells in vapor recovery unit (VRU) area. ARCADIS also proposes to install three deep monitoring wells: two monitoring wells replacing known destroyed wells (MW-26D and MW-31D) and one new monitoring well for vertical delineation (monitoring well MW-

25D3) at the former VRU. The proposed locations of the monitoring wells are depicted in the RI/FS WP on Figure 10.

3. Perform test pit/trench excavations at the former barge dock and the former garage area. A total of nine test pits/trenches will be excavated: six test pits at the former locations of the barge dock buildings, and three test pits at the former garage area. The test pits will be excavated to a depth between 5 and 9 feet, depending upon the depth to water. The test pit excavations will be used to assess the extent of residual light nonaqueous phase liquid and petroleum impacts in the shallow sand fill layer. The proposed locations of the test pits/trenches are depicted in the RI/FS WP on Figure 11.

4. Conduct a soil boring investigation at the former VRU, including advancing three soil borings to complete further vertical delineation within the meadow mat. The soil borings will be drilled to an approximate maximum depth of 16 feet. The proposed locations of the soil borings are depicted in the RI/FS WP on Figure 12.

5. Analyze select soil samples from soil borings and test pits for volatile organic compounds (VOCs), semivolatile organic compounds (SVOCs), Target Analyte List (TAL) metals, total organic carbon and ferrous iron.

6. Conduct aquifer testing of the deep sand layer. ARCADIS proposes to conduct a step pumping test and/or a 24-hour pumping test at monitoring well MW-30D. MW-30D was selected because this area and depth horizon may be the subject of future focus, to reduce the amount of and exposure to groundwater affected by higher concentrations of CVOCs.

7. Perform tracer testing at the deep sand layer. A nonreactive tracer (e.g., fluorescein) will be used to further assess hydraulic characteristics of the water-bearing units beneath the site.

8. Complete groundwater sampling of existing and newly installed monitoring wells. The groundwater samples will be analyzed for VOCs, SVOCs, methane, ethane, ethene, total dissolved solids, alkalinity, alkalinity-bicarbonate, sulfate, sulfide, nitrate, nitrite, nitrogen as ammonia, phosphorous as orthophosphate, calcium, total dissolved organic carbon, magnesium (total and dissolved), iron (total and dissolved) and TAL metals (total and dissolved).

9. Conduct specific-capacity testing during the proposed groundwater sampling event (Item 7 above).

10. Conduct bench-scale testing of soil and groundwater to estimate the soil oxidant demand and efficacy of persulfate under differing activation scenarios to evaluate the feasibility of an in-situ chemical oxidation (ISCO) remedial option for saturated soil and groundwater in both the shallow and deep sand

layers. Soil samples for bench-scale testing will be collected during installation of the additional monitoring wells and during the soil boring investigation. Groundwater samples for bench-scale testing will be collected during the proposed groundwater sampling event (Item 7 above). Bench-scale tests will be conducted on samples collected from the meadow mat for thermal resistivity. Other bench-scale tests may be conducted as necessary as RI work progresses at the site.

11. Perform remediation hydraulics evaluations as needed to refine the understanding of contaminant fate and transport, aquifer architecture, and refinement of the conceptual site model.

After completing and evaluating the proposed RI work scope, additional remedial or pre-design investigation activities will be proposed if remaining data gaps are identified.

4. Quality Objectives and Criteria for Measurement Data

The DQO process, as described in the USEPA QA/G-4 QAPP (2006) instructions document, is intended to provide a “logical framework” for planning field investigations. This section addresses each of the seven sequential steps in the USEPA QA/G-4 QAPP DQO process.

4.1 Step 1: State the Problem

The RI/FS will be conducted at the site in two phases, including source investigation and characterization, and bench-scale testing. The sampling and analysis program is intended to generate data to evaluate remedial measures for treatment of the source areas.

4.2 Step 2: Identify the Goal of the Study

The initial use of the data is descriptive (distribution and concentration of the constitutes of concern) and there is no decision point for this descriptive application. Subsequent to review of the descriptive information, an exposure evaluation will be performed based on the findings of the site investigation.

4.3 Step 3: Identify Information Inputs

Decision inputs incorporate both concentrations and the distribution of constitutes of concern in site media. A fundamental basis for decision-making is that a sufficient number of data points of acceptable quality are available from the investigation to support the decision. Thus, the necessary inputs for the decision are: 1) the proportion of non-rejected (usable) data points and 2) the quantity of data needed to evaluate remedial measures.

The data will be evaluated for completeness, general conformance with requirements of this QAPP and consistency among data sets and with historical data, as appropriate.

4.4 Step 4: Define the Boundaries of the Study

The site consists of 7.2 acres located at 1 Industrial Place in Oceanside, Township of Hempstead, Nassau County, New York. The site is bound to the south by Barnum’s Channel, to the east by the Long Island Rail Road, to the north by the former Exxon Petroleum Terminal and to the west by Hampton Road. A junk yard and office buildings are located west of Hampton Road, fronting Hog Island Channel. Daly Boulevard is located north of the former Exxon Petroleum Terminal and another petroleum terminal (Sprague) is located north of Daly Boulevard.

4.5 Step 5: Developing the Analytical Approach

The decision on whether data can be used in the exposure evaluation will be based on the validation results. Following validation, the data will be flagged, as appropriate, and any use restrictions will be noted. The sampling plan has been devised so that the loss of any single data point will not hinder description of the distribution of constitutes of concern or the development of a risk assessment. Given this, a reasonable decision rule will be that 90 percent of the data points not be rejected and deemed unusable for exposure evaluation purposes. Applicable actions will be evaluated, if needed based on the results of the exposure evaluation.

4.6 Step 6: Specify Performance or Acceptance Criteria

Specifications for this step call for: 1) giving forethought to corrective actions to improve data usability and 2) understanding the representative nature of the sampling design. This QAPP meets both specifications for this step. The sampling and analysis program has been developed based on a review of previous site data and knowledge of present site conditions. Corrective actions are described elsewhere in the document and in the appended documents. The representative nature of the sampling design has been assured by discussions among professionals familiar with the site and the appropriate government agencies.

Step 7: Develop the Plan for Obtaining Data

The overall QA objective is to develop and implement procedures for field sampling, COC, laboratory analysis and reporting that will provide results to support the evaluation of the site data consistent with National Contingency Plan requirements. DQOs for the sampling investigation efforts are summarized in Section 4.7. The summary includes stated DQOs relative to data uses, data types, data quantity, sampling and analytical methods, and data measurement performance criteria.

4.7 Data Categories

Three data categories have been defined to address various analytical data uses, and the associated QA/QC effort and methods required to achieve the desired levels of quality. These categories are discussed in Sections 4.7.1, 4.7.2 and 4.7.3.

4.7.1 Screening Data

Screening data affords a quick assessment of site characteristics or conditions. This DQO is applicable to data collection activities that involve rapid, nonrigorous methods of analysis and QA. This objective is generally applied to physical and/or chemical properties of samples, degree of contamination relative to concentration differences, and preliminary health and safety assessment.

4.7.2 Screening Data with Definitive Confirmation

Screening data allows rapid identification and quantitation, although the quantitation can be relatively imprecise. This DQO is available for data collection activities that require qualitative and/or quantitative verification of a select portion of sample findings (10 percent or more). This objective can also be used to verify less rigorous laboratory-based methods.

4.7.3 Definitive Data

Definitive data are generated using analytical methods such as approved USEPA reference methods. Data are analyte-specific, with confirmation of analyte identity and concentration. Methods produce raw data (e.g., chromatograms, spectra, digital values) in the form of paper printouts or computer-generated electronic files.

ARCADIS anticipates that both screening and definitive data categories will be used during the investigation. Field parameters (e.g., turbidity, conductivity, temperature and pH), which will be obtained during water column sampling for use in qualitatively interpreting other site data will be determined using screening techniques. All remaining parameters will be determined using definitive techniques.

For this project, the following three levels of data reporting have been defined:

4.7.3.1 Level 1 – Minimal Reporting

Minimal or “results only” reporting is used for analyses that, either due to their nature (i.e., field monitoring) or the intended data use (i.e., preliminary screening), do not generate or require extensive supporting documentation.

4.7.3.2 Level 2 – Modified Reporting

Modified reporting is used for analyses that are performed following standard USEPA-approved methods and QA/QC protocols and that, based on the intended data use, require some supporting documentation, but not full Contract Laboratory Program- (CLP-) type reporting.

4.7.3.3 Level 3 – Full Reporting:

Full CLP-type reporting is used for those analyses that, based on intended data use, require full documentation. This reporting level would include Analytical Services Protocol (ASP) Superfund and Category B reporting.

During the RI/FS, the following analytical methods will be used: USEPA SW-846 methods with NYSDEC ASP Revision 2005, QA/QC requirement, and Category B reporting deliverables.

4.8 Field Investigations

As part of the RI/FS, field investigations will be conducted to support the DQOs. Details of the field sampling investigations are described in the RI/FS WP.

5. Special Training Requirements/Certification

In compliance with the Occupational Safety and Health Administration's (OSHA) final rule, Hazardous Waste Operations and Emergency Response, 29CFR§1910.120(e), all personnel performing RI/FS activities at the site will have completed the requirements for OSHA 40-Hour Hazardous Waste Operations and Emergency Response training. Persons in field supervisory positions will have also completed the additional OSHA 8-Hour Supervisory Training.

6. Documentation and Records

6.1 General

Samples of the various media will be collected as described in the RI/FS WP. This section describes the documentation and associated reporting requirements.

6.2 Sample Designation System

6.2.1 Sample Codes

Samples will be identified with a unique designation system that will facilitate sample tracking. The sample designation system to be employed during the sampling activities will be consistent, yet flexible enough to accommodate unforeseen sampling events and conditions. An alpha-numeric system is considered appropriate and will be used by field personnel to assign each sample with a unique sample identification number.

The samples types will be designated using the following codes:

- Surface Soil – “SS”
- Soil Boring – “SB”
- Groundwater – “MW”
- Trip Blank – “TB”
- Equipment Blank – “EB”

The two-digit sample number beginning with “01” will be assigned in the field and incremented by one as samples are collected. Where necessary, the code system will be supplemented to accommodate additional sample identification information. For example, the code for soil samples will include a qualifier to identify the section increment (e.g., 0 to 0.5 feet).

Additional sample volumes collected for matrix spike (MS) and matrix spike duplicate (MSD) analysis will be noted on the COC forms, and the associated additional sample containers will be labeled with the appropriate suffix (MS or MSD). Rinse blanks will use the same coding scheme noted above, substituting the location code with the prefix “RB” (e.g., the first rinse blank associated with soil collection will be named RBSD01). Field duplicates will be labeled as ordinary field samples with a unique identification number (e.g., the first field duplicate associated collection will be named DUPSB01). Duplicate samples will not be identified and the laboratory will analyze them as “blind” QC samples.

6.2.2 Field Documentation

Field personnel will provide comprehensive documentation covering all aspects of field sampling, field analysis and sample COC. This documentation constitutes of a record that allows reconstruction of all field events to aid in the data review and interpretation process. All documents, records and information relating to the performance of the field work will be retained in the project file.

The various forms of documentation to be maintained throughout the project include:

- *Daily Production Documentation*. A field notebook consisting of a waterproof, bound notebook that will contain a record of all activities performed at the site.

- *Sampling Information*. Detailed notes will be made as to the exact sampling location, physical observations and weather conditions (as appropriate).

- *Sample COC*. COC forms will provide the record of responsibility for sample collection, transport and submittal to the laboratory. COC forms will be filled out at each sampling site, at a group of sampling sites, or at the end of each day of sampling by ARCADIS’s field personnel designated to be responsible for sample custody. If the samples are relinquished by the designated sampling person to other sampling or field personnel, the COC form will be signed and dated by the appropriate personnel to document the sample transfer. The original COC form will accompany the samples to the laboratory and copies will be forwarded to the project files. A sample COC form is included in Attachment 3A.

  Persons will have custody of samples when the samples are in their physical possession, in their view after being in their possession or in their physical possession and secured so they cannot be tampered with. In addition, when samples are secured in a restricted area accessible only to authorized personnel, they will be deemed to be in the custody of such authorized personnel.

- *Field Equipment, Calibration and Maintenance Logs*. To document the calibration and maintenance of field instrumentation, calibration and maintenance logs will be maintained for each piece of field equipment that is not factory-calibrated.

6.3 Laboratory Documentation Files

6.3.1 Laboratory Project Files

The laboratory will establish a file for all pertinent data. The file will include all correspondence, faxed information, phone logs and COC forms. The laboratory will retain all project files and data packages for 5 years.

6.3.2 Laboratory Logbooks

Workbooks, bench sheets, instrument logbooks and instrument printouts will be used to trace the history of samples through the analytical process and document important aspects of the work, including the associated QCs. As such, logbooks, bench sheets, instrument logs and instrument printouts will be part of the permanent record of the laboratory.

Each page or entry will be dated and initialed by the analyst at the time of entry. Errors in entry will be crossed out in indelible ink with one stroke, corrected without the use of white-out or by obliterating or writing directly over the erroneous entry, and initialed and dated by the individual making the correction. Pages of logbooks that are not used will be completed by lining out unused portions.

Information regarding the sample, analytical procedures performed and results of the testing will be recorded on laboratory forms or personal notebook pages by the analyst. These notes will be dated and will also identify the analyst, instrument used and instrument conditions.

Laboratory notebooks will be periodically reviewed by the laboratory group leaders for accuracy, completeness and compliance with this QAPP. The laboratory group leader will verify all entries and calculations. If all entries on the pages are correct, the laboratory group leader will initial and date the pages. Corrective action will be taken for incorrect entries before the laboratory group leader signs.

6.3.3 Computer Tape and Hard Copy Storage

All electronic files and deliverables will be retained by the laboratory for not less than 5 years; hard copy data packages (or electronic copies) will also be retained for not less than 5 years.

6.4 Data Reporting Requirements

Data will be reported both in the field and by the analytical laboratory, as described in Sections 6.4.1 and 6.4.2.

6.4.1 Field Data Reporting

Information collected in the field through visual observation, manual measurement and/or field instrumentation will be recorded in field notebooks or data sheets and/or on forms. Such data will be reviewed by the appropriate Task Manager for adherence to the RI/FS WP and for consistency. Concerns identified as a result of this review will be discussed with the field personnel, corrected if possible, and, as necessary, incorporated into the data evaluation process.

If applicable, field data forms and calculations will be processed and included in appendices to the appropriate reports (when generated). The original field logs, documents and data reductions will be kept in the project file at the ARCADIS office in Syracuse, New York.

6.4.2 Laboratory Data Reporting

The laboratory is responsible for preparing ASP Category B data packages for all VOCs, SVOCs, inorganics, total cyanide and total organic carbon (TOC) data; reduced data packages; and case narratives for all other analyses.

Data reports for all parameters will include, at a minimum, the following items:

Narrative. Summary of activities that took place during sample analysis, including the following information:

- laboratory name and address
- date of sample receipt
- cross reference of laboratory identification number to contractor sample identification
- analytical methods used
- deviations from specified protocol
- corrective actions taken

Analytical Results. Reported according to analysis type and including the following information, as acceptable:

- sample ID
- laboratory ID
- date of collection
- date of receipt
- date of extraction
- date of analysis
- detection limits

Sample results on the report forms will be collected for dilutions. Soil samples will be reported on a dry weight basis. Unless otherwise specified, results will be reported uncorrected for blank contamination.

The data for VOCs, SVOCs, inorganics and TOC analyses will be expanded to include all supporting documentation necessary to provide a Category B package. This additional documentation will include, but is not limited to, all raw data required to recalculate any result, including printouts, chromatograms and quantitation reports. The report also will include standards used to calibrate and calculate analytical results; sample extraction, digestion and other preparation logs; standard preparation logs; instrument run logs; and moisture content calculations.

7. Sampling Process Design

Information regarding the sampling design and rationale and associated sampling locations are presented in the RI/FS WP.

8. Sampling Method Requirements

Source materials, groundwater and soil samples will be collected as described in the RI/FS WP and in the standard operating procedure (SOP) for field activities contained in Attachment 3. The RI/FS WP also contains procedures that will be followed to drill and sample soil borings; install and develop monitoring wells; measure water levels; collect groundwater samples; perform field measurements; and handle, package and ship collected samples.

9. Sample Handling and Custody Requirements

9.1 Sample Containers and Preservation

Appropriate sample containers, preservation methods and laboratory holding times for samples collected during the RI/FS are shown in Table 4.

The analytical laboratory will supply appropriate sample containers and preservatives, as necessary. Precleaned bottles will be purchased according to USEPA Office of Solid Waste and Emergency Response Directive 9240.05A requirements. The field personnel will be responsible for properly labeling containers and preserving samples (as appropriate). Sample labeling procedures are discussed in Section 9.2.2.

9.2 Field Custody Procedures

The objective of field sample custody is to assure that samples are not tampered with from the time of sample collection through the time of transport to the analytical laboratory. Persons will have custody of samples when the samples are in their physical possession, in their view after being in their possession or in their physical possession and secured so they cannot be tampered with. In addition, when samples are secured in a restricted area that is accessible only to authorized personnel, they will be deemed to be in the custody of such authorized personnel.

Field custody documentation consists of both field logbooks and field COC forms.

9.2.1 Field Logbooks

Field logbooks will provide the means of recording data for the collection activities performed during the RI/FS. Entries will be described in detail so that persons going to the site can reconstruct a particular situation without reliance on memory.

Field logbooks will be bound field survey books or notebooks. Logbooks will be assigned to field personnel, but will be stored in a secure location when not in use. Each logbook will be identified by the project-specific document number. The title page of each logbook will contain the following information:

- person to whom the logbook is assigned
- logbook number
- project name

- project start date
- end date

Entries into the logbook will contain a variety of information. At the beginning of each entry, the date, start time, weather, names of all sampling team members present, level of personal protection being used and signature of the person making the entry will be entered. The names of visitors to the site, field sampling or investigation team personnel, and the purpose of their visit will also be recorded in the field logbook.

Field personnel will record measurements made and samples collected. Entries will be made in ink and no erasures will be made. If information is entered incorrectly, the information will be crossed out with one strike mark. Whenever a sample is collected or a measurement is made, the location of the station will be described in detail. The number of photographs taken of the station, if any, will also be noted. All equipment used to make measurements will be identified, along with the date of calibration.

Samples will be collected following the sampling procedures documented in the RI/FS WP. The equipment used to collect samples will be noted, along with the time of sampling, sample description, depth at which the sample was collected, volume and number of containers. Sample identification numbers will be assigned prior to sample collection. Field duplicate samples, which will receive an entirely separate sample identification number, will be noted under sample description.

9.2.2 Sample Labeling

Preprinted sample labels will be affixed to sample bottles prior to delivery at the sampling site. The following information is required on each sample label:

- project
- date collected
- time collected
- location
- sampler
- analysis to be performed

- preservative
- sample number

9.2.3 Field COC Forms

Completed COC forms will be required for all samples that are submitted for analysis. COC forms will be initiated by the field sampling personnel. The COC forms will contain the unique sample identification number, sample date and time, sample description, sample type, preservation (if any), and analyses required. The original COC form will accompany the samples to the laboratory. Copies of the COC will be made prior to shipment (or multiple copy forms used) for field documentation. The COC forms will remain with the samples at all times. The samples and signed COC forms will remain in the possession of the field sampling personnel until the samples are delivered to the express carrier (e.g., Federal Express) or hand delivered to a mobile or permanent laboratory, or placed in secure storage.

Sample labels will be completed for each sample using waterproof ink. The labels will include sample information such as: sample number and location, type of sample, date and time of sampling, sampler's name or initials, preservation, and analyses to be performed. The completed sample labels will be affixed to each sample bottle and covered with clear tape.

Whenever samples are split with a government agency or other party, a separate COC will be prepared for those samples and marked to indicate with whom the samples are being split. The person relinquishing the samples to the facility or agency should request the signature of the facility or agency representative who acknowledges receipt of the sample. If the representative is unavailable or refuses, this must be noted in the “Received By” space.

9.3 Management of Investigation-Derived Materials and Wastes

Management of investigation-derived materials and wastes will be performed consistent with the Guide to Management of Investigation – Derived Wastes*,* 9345.3-03FS (USEPA 1992). Disposable equipment (including personal protective equipment) and debris will be containerized and appropriately labeled during the sampling events, and will be disposed of accordingly. All purged groundwater and water generated during equipment decontamination will be containerized and temporally staged on site in a 55-gallon drum, and will be disposed of appropriately based on analytical results. Equipment will be decontaminated, as appropriate, as discussed in the Attachment 3B. All soil cuttings associated with drilling soil borings will also be collected and temporally stored on site in a 55-gallon drum(s), and disposed of properly following receipt of analytical results.

9.4 Packing, Handling and Shipping Requirements

Sample packaging and shipment procedures are designed so that the samples will arrive intact at the laboratory, with the COC.

Samples will be packaged for shipment as described below:

- Securely affix sample labels to the sample containers with clear packing tape.
- Check the caps on the sample containers so that they are properly sealed.
- Wrap the sample container cap with clear packing tape to prevent it from becoming loose.
- Complete the COC form with the required sampling information and ensure that the recorded information matches the sample labels. NOTE: If the designated sampler relinquishes the samples to other sampling or field personnel for packing or other purposes, the sampler will complete the COC prior to this transfer. The appropriate personnel will sign and date the COC form to document the sample custody transfer.
- Using duct tape, secure the outside drain plug at the bottom of the cooler.
- Wrap sample containers in bubble wrap or other cushioning material.
- Place 1 to 2 inches of cushioning material at the bottom of the cooler.
- Place the sealed sample containers into the cooler.
- Place ice in plastic bags and seal. Place loosely in the cooler.
- Fill the remaining space in the cooler with cushioning material.
- Place COC forms in a plastic bag and seal. Tape the forms to the inside of the cooler lid.
- Close the lid of the cooler, lock and secure with duct tape.
- Wrap strapping tape around both ends of the cooler at least twice.

- Mark the cooler on the outside with the following information: shipping address, return address, “Fragile” labels and arrows indicating “this side up.” Cover the labels with clear plastic tape. Place a signed custody seal over the sample cooler lid.

Samples will be hand-delivered or delivered by an express carrier within 48 hours of the time of collection. Shipments will be accompanied by the COC form identifying the contents. The original form will accompany the shipment; copies will be retained by the sampler for the sampling office records. If the samples are sent by common carrier, complete a bill of lading form. Receipts or bills of lading will be retained as part of the permanent project documentation. Commercial carriers are not required to sign off on the COC form as long as the forms are sealed inside the sample cooler and the custody seals remain intact.

The analytical laboratory will provide sample custody seals and packing materials for filled sample containers. Place the filled, labeled and sealed containers in a cooler on ice and pack carefully to eliminate the possibility of container breakage.

Additional procedures for packing, handling and shipping environmental samples are presented in the RI/FS WP.

9.5 Laboratory Custody Procedures

9.5.1 General

Upon sample receipt, laboratory personnel will be responsible for sample custody. The original field COC form will accompany all samples requiring laboratory analysis. The laboratory will use COC guidelines described in the USEPA guidance documents. Samples will be kept secured in the laboratory until all stages of analysis are complete. All laboratory personnel having samples in their custody will be responsible for documenting and maintaining sample integrity.

9.5.2 Sample Receipt and Storage

Immediately upon sample receipt, the laboratory sample custodian will verify the cooler seal, open the cooler and compare the contents against the field COC. The laboratory sample custodian will notify ARCADIS if a sample container is missing or received broken, the sample is in an inappropriate container, or the samples has not been preserved by appropriate means. The laboratory sample custodian will be responsible for logging the samples in, assigning a unique laboratory identification number to each sample, labeling the sample bottle with the laboratory identification number and moving the sample to an appropriate storage location to await analysis. The laboratory sample custodian will record the following information in the laboratory tracking system: project name, field sample code, date sampled, date received, analysis required, storage location and date, and action for final disposition. Relevant custody documentation will be placed in the project file.

9.5.3 Sample Analysis

Analysis of an acceptable sample will be initiated by worksheets that contain all pertinent information for analysis.

Samples will be organized into sample delivery groups (SDGs) by the laboratory. An SDG may contain up to 20 field samples (field duplicates, trip blanks and rinse blanks are considered field samples for SDG assignment). All field samples assigned to one SDG will be received by the laboratory over a maximum of 7 calendar days and must be processed through the laboratory (preparation, analysis and reporting) as a group. Every SDG must include a minimum of one site-specific MS/MSD pair, which will be received by the laboratory at the start of the SDG assignment.

9.5.4 Sample Storage Following Analysis

Samples will be maintained by the laboratory for at least 1 month after the final report is delivered to ARCADIS. The laboratory will be responsible for the eventual and appropriate disposal of the samples. The analytical laboratory will inform ARCADIS before disposing of any samples. Unused portions of the samples, sample extracts and associated wastes will be disposed of by the laboratory in accordance with applicable rules and regulations as specified in their SOP for waste disposal.

10. Analytical Method Requirements

10.1 Field Parameters and Methods

Field analytical procedures will include the measurement of pH, turbidity, temperature, conductivity and groundwater levels. Specific field measurement protocols are provided in the RI/FS WP and in the SOPS contained in Attachment 3.

10.2 Laboratory Parameters and Methods

The methods listed below include the range of analyses expected to be performed. The associated laboratory SOPs are included in Attachment 1.

Laboratory analytical requirements presented in Sections 10.2.1, 10.2.2 and 10.2.3 include a general summary of requirements, specifics related to each sample medium to be analyzed and details of the methods to be used for this project. SW-846 methods with NYSDEC ASP 2005 Revision, QA/QC and reporting deliverables requirements will be used for all analytes.

10.2.1 General

The following tables summarize general analytical requirements:

Table	Title
Table 1	Environmental and Quality Control Analyses
Table 3	Parameters, Methods and Target Reporting Limits
Table 4	Sample Containers, Preservation Methods and Holding Times

10.2.2 Remedial Investigation/Feasibility Study Sample Matrices

10.2.2.1 Groundwater

Analyses will be performed following the methods listed in Table 1. Analytical results for all analyses will be reported in units identified in Table 3.

10.2.2.2 Soil

Analyses of soil samples will be performed following the methods listed in Table 1. Results will be reported as dry weight, in units presented in Table 3. Moisture content will be reported separately.

10.3 Analytical Requirements

The primary sources to describe the analytical methods to be used during the investigation are provided in USEPA SW-846 Test Methods for Evaluating Solid Waste, Third Edition; and USEPA Methods for Chemical Analysis of Water and Waste with NYSDEC ASP 2005 Revision, QA/QC and reporting deliverables requirements. Detailed information regarding QA/QC is provided in NYSDEC ASP 2005 and methods and laboratory SOPs.

11. Quality Control Requirements

11.1 Quality Assurance Indictors

The overall QA objective for this QAPP is to develop and implement procedures for sampling, COC, laboratory analysis, instrument calibration, data reduction and reporting, internal QC, audits, preventive maintenance, and corrective action, to generate valid data. These procedures are presented or referenced in the following sections of the QAPP. Specific QC checks are discussed in Section 11.2.

QA indicators are generally defined in terms of five parameters:

1. Representativeness
2. Comparability
3. Completeness
4. Precision
5. Accuracy

Each parameter is defined below. Specific objectives for the site actions are set forth in other sections of this QAPP as referenced below.

11.1.1 Representativeness

Representativeness is the degree to which sampling data accurately and precisely represent site conditions, and is dependent on sampling and analytical variability and the variability of environmental media at the site. The actions have been designed to assess the presence of the chemical constituents at the time of sampling. The RI/FS WP presents the rationale for sample quantities and location. This QAPP presents field sampling and laboratory analytical methodologies. The use of the prescribed field and laboratory analytical methods with associated holding times and preservation requirements are intended to provide representative data.

11.1.2 Comparability

Comparability is the degree of confidence with which one data set can be compared to another. Comparability between this investigation, and to the extent possible, with existing data will be maintained through consistent sampling and analytical methodology set forth in the RI/FS WP and this QAPP, SW-

846 analytical methods with NYSDEC ASP Revision 2005, QA/QC requirements, and Category B reporting deliverables, and through use of QA/QC procedures and appropriately trained personnel.

11.1.3 Completeness

Completeness is defined as a measure of the amount of valid data obtained from an event and/or investigation compared to the total amount that was obtained. This will be determined upon final assessment of the analytical results, as discussed in Section 11.6.

11.1.4 Precision

Precision is the measure of reproducibility of sample results. The goal is to maintain a level of analytical precision consistent with the project objectives. To maximize precision, ARCADIS will follow the sampling and analytical procedures established in this QAPP. All work for this investigation will adhere to established protocols presented in the RI/FS WP. Checks for analytical precision will include the analysis of MSDs, laboratory duplicates and field duplicates. Checks for field measurement precision will include obtaining duplicate field measurements. Precision QC checks are discussed in Section 11.4.

11.1.5 Accuracy

Accuracy is the deviation of a measurement from the true value of a known standard. ARCADIS will monitor both field and analytical accuracy through initial and continuing calibration of instruments. In addition, ARCADIS will use internal standards, MSs, blank spikes and surrogates (system monitoring compounds) to assess the accuracy of the laboratory analytical data. These QC samples are discussed in Section 11.5.

11.2 Field Quality Control Checks

11.2.1 Field Measurements

To verify the quality of data using field instrumentation, ARCADIS will obtain and report all duplicate field measurements. A duplicate measurement involves obtaining a second measurement at the same sampling location.

11.2.2 Sample Containers

The laboratory will supply certified-clean sample containers in accordance with Exhibit I of the NYSDEC ASP Revision 2005 (Eagle Picher precleaned containers or equivalent).

11.2.3 Field Duplicates

ARCADIS will collect field duplicates from the different site materials to verify the reproducibility of the sampling methods. Field duplicates will be prepared by placing well-homogenized aliquots (except samples for VOC analysis) from the same sample location into individual sample containers, which will be submitted blind to the laboratory. Field duplicate water samples and soil samples for VOC analysis will constitute co-located samples rather than homogenized aliquots. In general, field duplicates will be analyzed at a 5 percent frequency (every 20 samples) for the chemical constituents. Table 1 provides an estimated number of field duplicates to be prepared for each applicable parameter and matrix.

11.2.4 Rinse Blanks

Rinse blanks are used to monitor the cleanliness of the sampling equipment and the effectiveness of the cleaning procedures. Rinse blanks will be prepared and submitted for analysis once per day per matrix. Rinse blanks will be prepared by filling sample containers with analyte-free water (supplied by the laboratory) that has been routed through a cleaned sampling device. When dedicated sampling devices or sample containers are used to collect the samples, rinse blanks will not be necessary. Table 1 provides an estimated number of rinse blanks for environmental media samples to be collected during the RI/FS.

11.2.5 Trip Blanks

Trip blanks will be used to assess whether site samples have been exposed to non-site-related volatile constituents during storage and transport. Trip blanks will be analyzed at a frequency of once per day, per cooler containing samples to be analyzed for volatile organic constituents. A trip blank will consist of a container filled with analyte-free water (supplied by the laboratory) that remains unopened with field samples throughout the sampling event. Trip blanks will only be analyzed for VOCs. Table 1 provides an estimated number of trip blanks collected for each matrix and parameter during the RI/FS.

11.3 Analytical Laboratory Quality Control Checks

11.3.1 General

Internal laboratory QC checks will be used to monitor data integrity. The TestAmerica Quality Manual is provided in Attachment 2. These checks will include method blanks, MS/MSDs, spike blanks, internal standards, surrogate samples, calibration standards and reference standards. Project QC limits for duplicates and MSs are identified in Table 2. Laboratory control charts will be used to determine long-term instrument trends.

11.3.2 Method Blanks

Sources of contamination in the analytical process, whether specific analyses or interferences, must be identified, isolated and corrected. The method blank is useful in identifying possible sources of contamination within the analytical process. For this reason, the method blank must be initiated at the beginning of the analytical process and encompass all aspects of the analytical work. As such, the method blank will assist in accounting for any potential contamination attributable to glassware, reagents, instrumentation or other sources that could affect sample analysis. One method blank will be analyzed with each analytical series associated with no more than 20 samples.

11.3.3 MS/MSDs

MS/MSDs will be used to measure the accuracy of analyte recovery from the sample matrices and will be site-specific. MS/MSD pairs will be analyzed at a 5 percent frequency (every 20 samples or once every week, whichever comes first).

When MS recoveries are outside QC limits, associated control sample and surrogate spike recoveries will be evaluated, as applicable, to attempt to verify the reason for the deviation and determine the effect on the reported sample results. Table 1 presents an estimated number of MS and MSD analyses for each applicable parameter.

11.3.4 Surrogate Spikes

Surrogates are compounds that are unlikely to occur under natural conditions and have properties similar to the analytes of interest. This type of control is primarily used for organic samples analyzed by gas chromatography/mass spectrometry (GC/MS) and GC methods and is added to the samples prior to purging or extraction. The surrogate spike is used to provide broader insight into the proficiency and efficiency of an analytical method on a sample-specific basis. This control reflects analytical conditions that may not be attributable to sample matrix.

If surrogate spike recoveries exceed specified QC limits, the analytical results must be evaluated thoroughly in conjunction with other control measures. In the absence of other control measures, the integrity of the data may not be verifiable and reanalysis of the samples with additional control may be necessary.

Surrogate spike compounds will be selected using the guidance provided in the analytical methods.

11.3.5 Laboratory Duplicates

For inorganics, laboratory duplicates will be analyzed to assess laboratory precision. Laboratory duplicates are defined as a separate aliquot of an individual sample that is analyzed as a separate sample. Table 1 presents an estimated number of laboratory duplicates for each applicable parameter.

11.3.6 Calibration Standards

Calibration check standards analyzed within a particular analytical series provide insight regarding the instruments' stability. A calibration check standard will be analyzed at the beginning and end of an analytical series, or periodically throughout a series containing a large number of samples.

In general, calibration check standards will be analyzed after every 12 hours, or more frequently, as specified in the applicable analytical method. In analyses where internal standards are used, a calibration check standard will only be analyzed in the beginning of an analytical series. If results of the calibration check standard exceed specified tolerances, then all samples analyzed since the last acceptable calibration check standard will be reanalyzed.

Laboratory instrument calibration standards will be selected using the guidance provided in the analytical methods, as summarized in Section 13.

11.3.7 Internal Standards

Internal standard areas and retention times will be monitored for organic analyses performed by GC/MS methods. Method-specified internal standard compounds will be spiked into all field samples, calibration standards and QC samples after preparation and prior to analysis. If internal standard areas in one or more samples exceed the specified tolerances, the cause will be investigated, the instrument will be recalibrated if necessary, and all affected samples will be reanalyzed.

The acceptability of internal standard performance will be determined using the guidance provided within the analytical methods.

11.3.8 Reference Standards/Control Samples

Reference standards are standards of known concentration and independent in origin from the calibration standards. The intent of reference standard analysis is to provide insight into the analytical proficiency within an analytical series. This includes preparation of calibration standards, validity of calibration, sample preparation, instrument set-up and the premises inherent in quantitation. Reference standards will be analyzed at the frequencies specified within the analytical methods.

11.4 Data Precision Assessment Procedures

Field precision is difficult to measure because of temporal variations in field parameters. However, precision will be controlled through the use of experienced field personnel, properly calibrated meters and duplicate field measurements. Field duplicates will be used to assess precision for the entire measurement system, including sampling, handling, shipping, storage, preparation and analysis.

Laboratory data precision for organic analyses will be monitored through the use of MS/MSD and laboratory duplicates as identified in Table 1.

The precision of data will be measured by calculating the relative percent difference (RPD) by the following equation:

$$RPD = \frac{(A-B)}{(A+B)/2} \times 100$$

Where:

A = Analytical result from one of two duplicate measurements
B = Analytical result from the second measurement

Precision objectives for MSD and laboratory duplicate analyses are identified in the NYSDEC ASP Revision 2005 and contained in Table 2.

11.5 Data Accuracy Assessment Procedures

The accuracy of field measurements will be controlled by experienced field personnel, properly calibrated field meters, and adherence to established protocols. The accuracy of field meters will be assessed by review of calibration and maintenance logs.

Laboratory accuracy will be assessed via the use of MSs, surrogate spikes, internal standards and reference standards. Where available and appropriate, QA performance standards will be analyzed periodically to assess laboratory accuracy. Accuracy will be calculated in terms of percent recovery as follows:

$$\%\ Recovery = \frac{A-X}{B} \times 100$$

Where:

A = Value measured in spiked sample or standard
X = Value measured in original sample
B = True value of amount added to sample or true value of standard

This formula is derived under the assumption of constant accuracy over the original and spiked measurements. If any accuracy calculated by this formula is outside of the acceptable levels, data will be evaluated to determine whether the deviation represents unacceptable accuracy, or variable but acceptable accuracy. Accuracy objectives for MS recoveries and surrogate recovery objectives are identified in the NYSDEC ASP 2005 Revision and contained in Table2.

11.6 Data Completeness Assessment Procedures

Completeness of a field or laboratory data set will be calculated by comparing the number of valid sample results generated to the total number of results generated.

$$\text{Completeness} = \frac{\text{Number valid results}}{\text{Total number of results generated}} \times 100$$

As a general guideline, overall project completeness is expected to be at least 90 percent. The assessment of completeness will require professional judgment to determine data usability for intended purposes.

12. Instrument/Equipment Testing, Inspection and Maintenance Requirements

12.1 General

Testing and maintenance schedules have been developed for both field and laboratory instruments. This section summarizes testing and maintenance activities to be performed during the RI/FS.

12.2 Field Instruments and Equipment

Prior to field sampling, each piece of field equipment will be inspected to ensure that it is operational. If the equipment is not operational, it will be serviced prior to its use. All meters that require charging or batteries will be fully charged and have fresh batteries. If instrument servicing is required, the appropriate Task Manager or field personnel will follow the maintenance schedule and arrange for timely service. Field instruments will be maintained according to the manufacturers' instructions.

Logbooks will be kept for each field instrument. Each logbook will contain records of operation, maintenance, calibration and any problems and repairs. Logbooks for each piece of equipment will be maintained in project records. The Task Managers will review calibration and maintenance logs.

12.2.1 Equipment Maintenance

All measuring and test equipment to be used in support of the RI/FS activities that directly affect the quality of the analytical data will be subject to preventive maintenance measures that minimize equipment downtime. Equipment will be examined to certify that it is in operating condition. This includes checking the manufacturer's operating manual to ensure that all maintenance requirements are being observed. Field notes from previous sampling events will be reviewed to ensure that any prior equipment problems are not overlooked and that any necessary repairs to equipment have been carried out.

Field equipment returned from a site will be inspected to confirm that it is in working order. The inspection will be recorded in the logbook or field notebooks, as appropriate. The last user of the equipment is responsible for recording any problems with that equipment in the logbook. Nonoperational field equipment will either be repaired or replaced. Appropriate spare parts will be made available for field meters.

Consultant-/subcontractor-owned or leased equipment maintenance will be conducted in accordance with the manufacturer's instructions.

12.3 Laboratory Instruments and Equipment

12.3.1 General

Laboratory instrument and equipment documentation procedures include details of any observed problems, corrective measure(s), routine maintenance and instrument repair (which will include information regarding the repair and the individual who performed the repair).

Preventive maintenance of laboratory equipment generally will follow the guidelines recommended by the manufacturer. A malfunctioning instrument will be repaired immediately by in-house staff or through a service call from the manufacturer.

12.3.2 Instrument Maintenance

Maintenance schedules for laboratory equipment will adhere to the manufacturer’s recommendations. Records will reflect the complete history of each instrument and specify the time frame for future maintenance. Major repairs or maintenance procedures will be performed through service contracts with the manufacturer or qualified contractors. Paperwork associated with service calls and preventive maintenance calls will be kept on file by the laboratory.

Laboratory Systems Managers are responsible for the routine maintenance of instruments used in the particular laboratory. Any routine preventive maintenance conducted will be logged into the appropriate logbooks. The frequency of routine maintenance will be dictated by the nature of samples being analyzed, the requirements of the method used and/or the judgment of the Laboratory Systems Manager.

All major instruments will be backed up by comparable (if not equivalent) instrument systems in the event of unscheduled downtime. An inventory of spare parts is also available to minimize equipment/instrument downtime.

12.3.3 Equipment Monitoring

The operation of balances, incubators, ovens, refrigerators and water purification systems will be checked and documented daily. Any discrepancies will be immediately reported to the appropriate laboratory personnel for resolution.

13. Instrument Calibration and Frequency

13.1 Field Instruments and Equipment

The calibration of field instruments is governed by specific SOPs documented in Attachment 3 for the applicable field analysis method, and such procedures take precedence over the following discussion.

Field personnel are responsible for maintaining a master calibration/maintenance log, following the procedures specified for each measuring device. Where applicable, each log will include, at a minimum, the following information:

- name of device and/or instrument calibrated
- device/instrument serial/identification numbers
- calibration method
- tolerance
- calibration standard used
- frequency of calibration
- date(s) of calibration(s)
- name of person(s) performing calibration(s)

Instruments and equipment used to gather, generate or measure environmental data will be calibrated at the intervals specified by the manufacturer or more frequently, and in such a manner that accuracy and reproducibility of results are consistent with the manufacturer’s specifications. If an internally calibrated field instrument fails to meet calibration/checkout procedures, it will be returned to the manufacturer for service. Equipment found to be out of tolerance during the period of use will be removed from the field and measuring and testing activities performed using the equipment will be addressed via the corrective action system described in Section 17.4 of this QAPP.

13.2 Laboratory Instrument and Equipment

Instrument calibration will follow the specifications provided by the instrument manufacturer or specific analytical method used. The analytical methods for target constituents are identified separately below.

13.2.1 VOCs

Equipment calibration procedures will follow guidelines presented in SW-846 methods as modified by laboratory SOPs.

13.2.2 SVOCs

Equipment calibration procedures will follow guidelines presented in SW-846 methods as modified by laboratory SOPs.

13.2.3 Metals

Equipment calibration procedures will follow guidelines presented in SW-846 methods as modified by laboratory SOPs.

13.2.4 TOC

Equipment calibration procedures will follow guidelines presented in Lloyd Kahn Method.

Equipment calibration procedures will follow guidelines presented in NYSDEC ASP 2005 Revision, Exhibit D for the following parameter groups/methods: methane, chloride, nitrogen as ammonia, nitrate, sulfate, sulfide, phosphorous as orthophosphate, total dissolved organic carbon, pH, oxidation-reduction potential, alkalinity, alkalinity-bicarbonate, iron (filtered and unfiltered), manganese (filtered and unfiltered).

When analyses are conducted according to the USEPA SW-846 methods, the calibration procedures and frequencies specified in the applicable method will be followed, as noted in the attached SOPs (Attachment 1). For analyses governed by SOPs, see the appropriate SOP for the required calibration procedures and frequencies. Records of calibrations will be filed and maintained by the laboratory. These records will be subject to QA audit. For all instruments, the laboratory will maintain trained repair staff with in-house spare parts or will maintain service contracts with vendors.

All standards used in the calibration of equipment are traceable, directly or indirectly, to the National Institute of Standards and Technology. All standards received will be logged into standard receipt logs maintained by the individual analytical groups. Each group will maintain a standards log that tracks the preparation of standards used for calibration and QC purposes.

14. Inspection/Acceptance Requirements for Supplies and Consumables

All supplies to be used in the field and laboratory will be available when needed and will be free of target chemicals and interferences. All reagents will be tested prior to use with site samples. All standards will be verified against a second source standard. The laboratory will follow a “first in first out” procedure for the storage and use of all consumables to minimize the risk of contamination and degradation. The various supplies and consumables required on site will be noted in the SOPs included in Attachment 3.

15. Data Acquisition Requirements for Non-Direct Measurements

Historical data generated by outside parties is currently not anticipated to be used to complete the RI/FS, but will be used as guidance in determining sampling locations.

Prior to their use, historical data sets will be reviewed according to the procedures identified in subsequent sections of this QAPP to determine the appropriate uses of such data. The extent to which these data can be validated will be determined by the analytical level and QC data available. The evaluation of historical data for RI/FS purposes requires the following:

- identification of analytical levels
- evaluation of QC data, when available
- development of conclusions regarding the acceptability of the data for intended uses

Acceptability of historical data for intended uses will be determined by application of these procedures and professional judgment. If the historical data quality cannot be determined, its use will be limited to general trend evaluations.

16. Data Management

The purpose of data management is to ensure that all of the necessary data are accurate and readily accessible to meet the analytical and reporting objectives of the project. The field investigations will encompass a large number of samples and analytes from a large geographic area. Due to the large amount of resulting data, a structured, comprehensive and efficient program is necessary to manage the data.

The data management program established for the project includes field documentation and sample QA/QC procedures, methods for tracking and managing the data, and a system for filing all site-related information. Specifically, data management procedures will be employed to efficiently process the information collected such that the data are readily accessible and accurate. This section describes these procedures in detail.

The data management plan has five elements:

1. sample designation system
2. field activities
3. sample tracking and management
4. data management system
5. document control and inventory

These elements are discussed in Sections 16.1 through 16.5, respectively.

16.1 Sample Designation System

A concise and easily understandable sample designation system is an important part of the project sampling activities. It provides a unique sample number that will facilitate both sample tracking and easy resampling of select locations to evaluate data gaps, if necessary. The sample designation system to be employed during the sampling activities will be consistent, yet flexible enough to accommodate unforeseen sampling events or conditions. A combination of letters and numbers will be used to yield a unique sample number for each field sampled collected, as outlined in Section 6.2.1.

16.2 Field Activities

Field activities designed to gather the information necessary to make decisions during the RI/FS process require consistent documentation and accurate record keeping. During site activities, standardized procedures will be used for documentation of field activities, data security and QA. These procedures are described in further detail in the following subsections.

16.2.1 Field Documentation

Complete and accurate record keeping is a critical component of the field investigation activities. When interpreting analytical results and identifying data trends, investigators realize that field notes are an important part of the review and validation process. To ensure that the field investigation is thoroughly documented, several different information records, each with its own specific reporting requirements, will be maintained, including:

- field logs
- COC forms
- instrument calibration records

A description of each of these types of field documentation is provided below.

16.2.1.1 Field Logs

Personnel performing the field activities will keep field logs that detail all observations and measurements made during the RI/FS. Data will be recorded directly into site-dedicated, bound notebooks, with each entry dated and signed. To ensure at any future date that notebook pages are not missing, each page will be sequentially numbered. Erroneous entries will be corrected by crossing out the original entry, initialing it and documenting the proper information. In addition, certain media sampling locations will be surveyed to accurately record their locations. The survey crew will use their own field logs and will supply the sampling location coordinates to the Database Administrator.

16.2.1.2 COC Forms

COC forms are used to document and track sample possession from time of collection to the time of disposal. A COC form will accompany each field sample collected, and one copy of the form will be filed in the field office. All field personnel will be briefed on the proper use of the COC procedure. COC procedures and a sample form are included in Attachment 3A.

16.2.1.3 Instrument Calibration Records

As part of data QA procedures, field monitoring and detection equipment will be routinely calibrated. Instrument calibration ensures that equipment used is of the proper type, range, accuracy and precision to provide data compatible with the specified requirements and desired results. Calibration procedures for the various types of field instrumentation are described in Section 13.1. To demonstrate that established calibration procedures have been followed, calibration records will be prepared and maintained to include, as appropriate, the following information:

- calibration date and time
- type and identification number of equipment
- calibration frequency and acceptable tolerances
- identification of individual(s) performing calibration
- reference standards used
- calibration data
- calibration success or failure

The calibration record will serve as a written account of monitoring or detection equipment QA. All erratic behavior or failures of field equipment will be subsequently recorded in the calibration log.

16.2.2 Data Security

Measures will be taken during the field investigation to ensure that samples and records are not lost, damaged or altered. When not in use, all field notebooks will be stored at the field office or locked in the field vehicle. Access to these files will be limited to the field personnel who use them.

16.3 Sample Management and Tracking

ARCADIS will maintain a record of all field documentation to ensure the validity of data used in the site analysis. To effectively execute such documentation, specific sample tracking and data management procedures will be used throughout the sampling program.

Sample tracking will begin with the completion of COC forms, as summarized in Section 9.2.3. The completed COC forms associated with samples collected will be faxed to the Quality Assurance Coordinator (QAC). Copies of all completed COC forms will be maintained in the field office. The laboratory will verify receipt of the samples electronically (via email) on the following day.

When analytical data are received from the laboratory, the QAC will review the incoming analytical data packages against the information on the COCs to confirm that the correct analyses were performed for each sample and that results for all samples submitted for analysis were received. The QAC will promptly follow up on any discrepancies noted.

16.4 Data Management System

In addition to the sample tracking system, ARCADIS will implement a data management system. The central focus of the data management system will be the development of a personal computer-based project database. Anticipated computer hardware and software components are described in Sections 16.4.1 and 16.4.2.

The Database Administrator will maintain the project database, which will combine pertinent geographical, field and analytical data. Information that will be used to populate the database will be derived from three primary sources: survey of sampling locations, field observations and analytical results. These sources are discussed in Sections 16.4.3, 16.4.4 and 16.4.5.

16.4.1 Computer Hardware

The database will be constructed on personal computer work stations connected through a network server. The network will provide access to various hardware peripherals (e.g., laser printers, backup storage devices, image scanners, modems). Computer hardware will be upgraded to industrial and corporate standards, as necessary, in the future.

16.4.2 Computer Software

The database is web-based database provided by LOCUS Technologies. The laboratory EDDs will be imported into the database and available to the project team through the Database Administrator. Geographic information system (GIS) applications will be developed in ESRI ArcGIS, with additional customization performed using Microsoft Visual Basic. Custom applets, such as for the generation of data tables and other database reports will be generated through Microsoft Access in conjunction with Microsoft Excel, Microsoft Word and/or Seagate Crystal Reports. These software products will be upgraded to current industrial standards, as necessary.

16.4.3 Survey Information

In general, each location sampled as part of the RI/FS will be surveyed to ensure accurate documentation of sample locations for mapping and GIS purposes (if appropriate), to facilitate the resampling of select sample locations during future monitoring programs and remediation activities. Field surveying activities will consist of collecting information that will be used to compute a northing and easting in state plane coordinates for each sample location and collecting information to compute elevations relative to the National Geodetic Vertical Datum of 1988 for select sample locations, as appropriate. All field books associated with the surveying activities will be stored as a record of the project activities.

16.4.4 Field Observations

Field observations are an important part of the information that will ultimately reside in the data management system for use during the project. Following each sampling event, a status memorandum may be prepared by the field personnel who performed the sampling activities. The purpose of the status memorandum is to summarize and record the sampling event. Topics to be discussed include the locations sampled, sampling methodologies used, QA/QC procedures, blind duplicate and MS/MSD sample identification numbers, equipment decontamination procedures, personnel involved in the activity, and any other noteworthy events that occurred.

Tables are typically attached to the memorandum and are used to summarize measurements that were recorded in the field books. ARCADIS anticipates that these tables will be developed using a personal computer spreadsheet program to reduce possible transcription error and to facilitate the transfer of information to the data management system. For example, for soil samples, the table will present the sampling date and time, water depth, soil depth, depth of soil recovered in a given core, depth increment submitted for analysis, and description of the lithology.

Status memorandums are valuable tools to keep project personnel informed on the details of the field activities and for use during the development of the final report. Each status memo will be reviewed for accuracy and completeness by the respective sampling activity manager. Following the approval and finalization of each memo, the status memo will be used to transfer field observations into the data management system.

All pertinent field data will be manually entered into the appropriate database tables from the COC forms and field notebooks.

16.4.5 Analytical Results

Analytical results will be provided by the laboratory in both a digital and a hard copy format. The data packages will be examined to ensure that the correct analyses were performed for each sample submitted and that all of the analyses requested on the COC form were performed. If discrepancies are noted, the QAC will be notified and will promptly follow up with the laboratory to resolve any issues.

Each data package will be validated in accordance with the procedures presented in Section 20. Any data that does not meet the specified standards will be flagged pending resolution of the issue. The flag will not be removed from the data until the issue associated with the sample results is resolved. Although flags may remain for certain data, the use of that data may not necessarily be restricted.

Following completion of the data validation, the digital files will be used to populate the appropriate database tables. An example of the electronic data deliverable (EDD) format is included in Table 5. This format specifies one data record for each constituent for each sample analyzed. Specific fields include:

- sample identification number
- date sampled
- date analyzed
- parameter name
- analytical result
- units
- detection limit
- qualifier(s)

The individual EDDs, supplied by the laboratory in either an ASCII comma-separated value format or in a Microsoft Excel worksheet, will be loaded into the appropriate database table. Any analytical data that cannot be provided by the laboratory in electronic format will be entered manually. After entry into the database, the EDD data will be compared to the field information previously entered into the database to confirm that all requested analytical data have been received.

16.4.6 Data Analysis and Reporting

The data management system will have several functions to facilitate the review and analysis of the RI/FS data. Data entry screens will be developed to assist in keypunching of field observations. Routines will also be developed to permit the user to scan analytical data from a given site for a given media. ARCADIS has developed several output functions that will be appropriately modified for use in the data management system.

A valuable function of the data management system will be the generation of tables of analytical results from the project databases. The capability of the data management system to directly produce tables reduces the redundant manual entry of analytical results during report preparation and precludes transcription errors that may occur otherwise. This data management system function creates a digital comma-delimited ASCII file of analytical results and qualifiers for a given media. The ASCII file is then processed through a spreadsheet, which transforms the comma-delimited file into a table of rows and columns. Tables of analytical data will be produced as part of data interpretation tasks, the reporting of data, and the generation of the RI/FS Report.

Another function of the data management system will be to create digital files of analytical results and qualifiers suitable for transfer to mapping/presentation software. ARCADIS has created a function that creates a digital file consisting of sample location number, state plane coordinates, sampling date and detected constituents and associated concentrations and analytical qualifiers. The file is then transferred to an AutoCAD work station, where another program has been developed to plot a location’s analytical data in a "box" format at the sample location (represented by the state plane coordinates). This routine greatly reduces the redundant keypunching of analytical results and facilitates the efficient production of interpretative and presentation graphics.

The data management system can also produce a digital file of select parameters that exist in one or more of the databases. This type of custom function is accomplished on an interactive basis and is best used for transferring select information into several analysis tools, such as statistical or graphing programs.

16.5 Document Control and Inventory

ARCADIS maintains project files at its Syracuse, New York office. Each client project is assigned a file/job number. Each file is then broken down into the following subfiles:

1. Agreements/Proposals (filed chronologically)

2. Change Orders/Purchase Orders (filed chronologically)

3. Invoices (filed chronologically)
4. Project Management (filed by topic)
5. Correspondence (filed chronologically)
6. Notes and Data (filed by topic)
7. Public Relations Information (filed by topic)
8. Regulatory Documents (filed chronologically)
9. Marketing Documents (filed chronologically)
10. Final Reports/Presentations (filed chronologically)
11. Draft Reports/Presentations (filed chronologically)
12. Documents Prepared by Others (filed chronologically)

Originals, when possible, are placed in the files. These are the central files and will serve as the site-specific files for the RI/FS.

17. Assessment and Response Actions

This section discusses the performance and systems audits that will be completed in the field and in the laboratory during the RI/FS.

17.1 Field Audits

The appropriate Task Manager will monitor field performance. Field performance audit summaries will contain an evaluation of field activities to verify that activities are performed according to established protocols. The ARCADIS QAC will review field reports and communicate concerns to the ARCADIS Project Manager and/or Task Managers, as appropriate. In addition, the ARCADIS QAC will review the rinse and trip blank data to identify potential deficiencies in field sampling and cleaning procedures. Systems audits will be performed to compare scheduled QA/QC activities from this QAPP with actual QA/QC activities completed. The appropriate Task Manager and QAC will periodically confirm that work is being performed consistent with this QAPP, the RI/FS WP and the SOPs provided in Attachment 3.

17.2 Laboratory Audits

The laboratory will perform internal audits consistent with NYSDEC ASP 2005 Revision and in accordance with method and laboratory SOP criteria. Internal laboratory audits are conducted by the laboratory QAC. As part of the audit, the overall performance of the laboratory staff is evaluated and compared to the performance criteria outlined in the laboratory QA manual and SOPs. Results of the audits are summarized and issued to each department supervisor, the Laboratory Manager and the Laboratory Director. The QAC will also perform a systems audit of each laboratory to determine if the procedures implemented by each laboratory comply with the laboratory QA manual and SOPs.

In addition to the laboratory’s internal audits, as participants in state and federal certification programs, the laboratory is audited by representatives of the regulatory agency issuing certification. Audits are usually conducted annually and focus on laboratory conformance to the specific program protocols for which the laboratory is seeking certification. The auditor reviews sample handling and tracking documentation, analytical methodologies, analytical supportive documentation, and final reports. The audit findings are formally documented and submitted to the laboratory for corrective action, if necessary.

ARCADIS reserves the right to conduct an on-site audit of the laboratory prior to the start of analyses for the project. Additional audits may be performed during the project, as deemed necessary.

17.3 Corrective Action

Corrective actions are required when field or analytical data are not within the objectives specified in this QAPP or the RI/FS WP. Corrective actions include procedures to promptly investigate, document, evaluate and correct data collection and/or analytical procedures. Field and laboratory corrective action procedures are described in Sections 17.3.1 and 17.3.2.

17.3.1 Field Procedures

When conducting the field work, if a condition is noted by the field personnel that will have an adverse effect on data quality, corrective action will be taken so as not to repeat this condition. Condition identification, cause and corrective action implemented by the Field Manager or a designee, will be documented on a Corrective Action Form and reported to the appropriate ARCADIS Task Manager, QAC and Project Manager.

Examples of situations that will require corrective actions are provided below:

- Protocols defined in this QAPP and the RI/FS WP have not been followed.
- Equipment is not in proper working order or is not properly calibrated.
- QC requirements have not been met.
- Issues resulting from performance or systems audits have not been resolved.

Project personnel will continuously monitor ongoing performance during daily work activities.

17.3.2 Laboratory Procedures

In the laboratory, when a condition is noted to have an adverse effect on data quality, corrective action will be taken so as not to repeat this condition. Condition identification, cause and corrective action taken will be documented and reported to the appropriate Project Manager and QAC.

Corrective action may be initiated, at a minimum, under the following conditions:

- Specific laboratory analytical protocols have not been followed.
- Protocols defined in this QAPP have not been followed.

- Predetermined data acceptance standards have not been obtained.
- Equipment is not in proper working order or calibrated.
- Sample and test results are not completely traceable.
- QC requirements have not been met.
- Issues resulting from performance or systems audits have not been resolved.

Laboratory personnel will continuously monitor ongoing performance during daily work activities. Corrective action will be initiated when a problem is identified. At whatever level this occurs (analyst, supervisor, data review or QC), it will be brought to the attention of the laboratory QAC and, ultimately, the Laboratory Director. Final approval of any action deemed necessary is subject to the approval of the Laboratory Director.

ARCADIS will implement any corrective action deemed necessary based on system or performance audits or the results of data review. The corrective action may include sample re-extraction, re-preparation, re-analysis, cleanup, dilutions, matrix modifications or other activities.

18. Reports to Management

18.1 Internal Reporting

The laboratory will submit analytical reports to ARCADIS for review. If required, ARCADIS will submit the analytical reports to the data validator for review. Supporting data (i.e., historical data, related field or laboratory data) will also be reviewed to evaluate data quality, as appropriate. The ARCADIS Quality Assurance Manager will incorporate results of the data validation reports (if required) and assessments of data usability into a summary report (if required) that will be submitted to the ARCADIS Project Manager and appropriate Task Managers. If required, this report will be filed in the project file at ARCADIS's office and will include the following:

- assessment of data accuracy, precision and completeness for both field and laboratory data
- results of the performance and systems audits
- significant QA/QC problems, solutions, corrections and potential consequences
- analytical data validation report

18.2 Remedial Investigation/Feasibility Study Reporting

Upon sample transport to the laboratory, a copy of the COC will be forwarded to ARCADIS’s Project Manager. Upon receipt of the ASP - Category B Data Package from the laboratory, the ARCADIS Quality Assurance Manager will determine if the data package has met the required DQOs. The analytical data package will be submitted to the ARCADIS Project Manager and the analytical data will be incorporated into the RI/FS Report in a table.

19. Data Reduction and Review

After field and laboratory data are obtained, the data will be subject to the following:

1. reduction, or manipulation mathematically, or otherwise into meaningful and useful forms
2. review
3. organization, interpretation and reporting
4. data validation

19.1 Field Data Reduction and Review

19.1.1 Field Data Reduction

Information collected in the field through visual observation, manual measurement and/or field instrumentation will be recorded in field notebooks or data sheets, and/or on forms. Such data will be reviewed by the appropriate Task Manager for adherence to the RI/FS WP and this QAPP, and for consistency. Concerns identified as a result of this review will be discussed with the field personnel, corrected if possible, and, as necessary, incorporated into the data evaluation process.

19.1.2 Field Data Review

Field data calculations, transfers and interpretations will be conducted by the field personnel and reviewed for accuracy by the appropriate Task Manager and the QAC. Logs and documents will be checked for:

1. general completeness
2. readability
3. use of appropriate procedures
4. appropriate instrument calibration and maintenance
5. reasonableness in comparison to present and past data collected

6. correct sample locations

7. correct calculations and interpretations

19.2 Laboratory Data Reduction and Review

19.2.1 Laboratory Data Reduction

The calculations used for data reduction will be specified in each of the analytical methods referenced previously. Whenever possible, analytical data will be transferred directly from the instrument to a computerized data system. Raw data will be entered into permanently bound laboratory notebooks. The data entered will be sufficient to document all factors used to arrive at the reported value.

Concentration calculations for chromatographic analyses will be based on response factors. Quantitation will be performed using either internal or external standards.

Inorganic analyses will be based on regression analysis. Regression analysis is used to fit a curve through the calibration standard data. The sample concentrations will be calculated using the resulting regression equations.

Nonaqueous values will be reported on a dry-weight basis. Unless otherwise specified, all values will be reported uncorrected for blank contamination.

19.2.2 Laboratory Data Review

Data will be subject to multilevel review by the laboratory. The group leader will review all data reports prior to release for final data report generation. The QAC will review the final data reports, and the QA Manager will review a cross-section of the final data reports prior to shipment to ARCADIS.

If discrepancies or deficiencies exist in the analytical results, corrective action will be taken as discussed in Section 17. Deficiencies discovered as a result of internal data review, as well as the corrective actions to be used to rectify the situation, will be documented on a Corrective Action Form. This form will be submitted to the ARCADIS Project Manager.

19.3 Data Validation and Verification

All data generated for health and safety and engineering design/control purposes will be subjected to the data validation and verification procedures outlined in Section 20. Data generated for disposal purposes will not be reviewed.

20. Data Validation and Verification

Data validation entails a review of the QC data and raw data to verify that the laboratory was operating within required limits, the analytical results were correctly transcribed from the instrument read outs and which, if any, environmental samples were related to any out-of-control QC samples. The objective of data validation is to identify any questionable or invalid laboratory measurements.

ARCADIS will validate all data generated by producing a NYSDEC data usability summary report for each individual SDG using the most recent versions of the USEPA’s Functional Guidelines (USEPA 1999 and 2002a) and USEPA Region II SOPs for data validation that are available at the time of project initiation, where appropriate. These procedures and criteria may be modified as necessary to address project- and method-specific criteria, control limits, and procedures. Data validation will consist of data screening, checking, reviewing, editing and interpretation to document analytical data quality and to determine whether the quality is sufficient to meet the DQOs.

The data validator will verify that reduction of laboratory measurements and laboratory reporting of analytical parameters is conducted in accordance with the procedures specified for each analytical method and/or as specified in this QAPP. Any deviations from the analytical method or any special reporting requirements apart from that specified in this QAPP will be detailed on COC forms.

Upon receipt of laboratory data, the following procedures will be executed by the data validator:

- Evaluate completeness of data package.
- Verify that field COC forms were completed and that samples were handled properly.
- Verify that holding times were met for each parameter. Document holding time exceedances, if they occur. Flag data for all samples that exceed holding time requirements as either estimated or rejected. The decision as to which qualifier is more appropriate will be made on a case-by-case basis.
- Verify that parameters were analyzed according to the methods specified.
- Review QA/QC data (i.e., make sure duplicates, blanks and spikes were analyzed on the required number of samples, as specified in the method; verify that duplicate and MS recoveries are acceptable).
- Investigate anomalies identified during review. If anomalies are identified, discuss them with the Project Manager and/or Laboratory Manager, as appropriate.

- If data appears suspect, investigate the specific data of concern. Calculations will be traced back to raw data; if calculations do not agree, the cause will be determined and corrected.

Deficiencies discovered as a result of the data review, as well as the corrective actions implemented in response, will be documented and submitted in the form of a written report addressing the following topics as applicable to each method:

- assessment of the data package
- description of any protocol deviations
- failures to reconcile reported and/or raw data
- assessment of any compromised data
- overall appraisal of the analytical data
- table of site name, sample quantities, matrix and fractions analyzed

It should be noted that qualified results do not necessarily invalidate data. The goal to produce the best possible data does not necessarily mean producing data without QC qualifiers. Qualified data can provide useful information.

Resolution of any issues regarding laboratory performance or deliverables will be handled between the laboratory and the data validator. Suggestions for reanalysis may be made by the ARCADIS QAC at this point.

Data validation reports will be kept in the project file at the ARCADIS office in Syracuse, New York.

21. Reconciliation with User Requirements

The data results will be examined to determine the performance that was achieved for each data usability criteria. The performance will then be compared with the project objectives and DQOs. Deviations from objectives will be noted. Additional action may be warranted when performance does not meet performance objectives for critical data. Options for corrective action relating to incomplete information, questionable results or inconsistent data, may include any or all of the following:

- retrieval of missing information
- request for additional explanation or clarification
- reanalysis of sample from extract (when appropriate)
- recalculation or reinterpretation of results by the laboratory

These actions may improve the data quality, reduce uncertainty and eliminate the need to qualify or reject data.

If these actions do not improve the data quality to an acceptable level, the following additional actions may be taken:

- extrapolate missing data from existing data points
- use historical data
- evaluate the critical/noncritical nature of the sample

If the data gap cannot be resolved by these actions, the data bias and potential for false negatives and positives can be evaluated. If the resultant uncertainty level is unacceptable, additional sample collection and analysis must be performed.

22. References

New York State Department of Environmental Conservation. 2005.

United States Environmental Protection Agency. 1980. *Interim Guidance and Specifications for Preparing Quality Assurance Project Plans*. QAMS-005/80. Office of Research and Development (December 1980).

United States Environmental Protection Agency. 1992. *Guide to Management of Investigation-Derived Wastes*. 9345.3-03FS (January 1992).

United States Environmental Protection Agency. 1999. *Contract Laboratory Program National Functional Guidelines for Organic Data Review*. EPA-540/R-99-008 (October 1999).

United States Environmental Protection Agency. 2001. *EPA Requirements for Quality Assurance Project Plans for Environmental Operations*. EPA-QA/R-5. Office of Environmental Information (March 2001).

United States Environmental Protection Agency. 2002a. *Contract Laboratory Program National Functional Guidelines for Inorganic Data Review*. EPA-540/R-01-008 (July 2002).

United States Environmental Protection Agency. 2002b. *Guidance for Quality Assurance Project Plans*. EPA-QA/G-5. Office of Environmental Information (December 2002).

Tables

Table 1. Sample Quantities and Quality Control Frequencies, Quality Assurance Project Plan, Chevron Environmental Management Company, Former Gulf Terminal Site, Oceanside, NY

Parameter	Estimated Environmental Sample Quality	Field QC Analyses						Laboratory QC Sample						Total
		Trip Blank		Rinse Blank		Field Duplicate		Matrix Spike		Matrix Spike Duplicate		Lab Duplicate		
		Freq.	No.	Freq.	No.	Freq.	No.	Freq.	No.	Freq.	No.	Freq.	No.	
Soil														
Volatile Organic Compounds (SW-846 8260-TCL)	45	NA	10	1/day	10	1/20	3	1/20	3	1/20	3	1/20	3	77
Semivolatile Organic Compounds (SW-846 8270-TCL)	45	NA	--	1/day	10	1/20	3	1/20	3	1/20	3	1/20	3	67
TAL Metals (EPA Method 6010B and 7471A)	45	NA	--	1/day	10	1/20	3	1/20	3	1/20	3	1/20	3	67
Ferrous Iron (SM3500-Fe-D)	45	NA	--	1/day	10	NA	3	1/20	3	1/20	3	1/20	3	67
Total Dissolved Organic Carbon (SM5310-D)	45	NA	--	1/day	10	NA	3	1/20	3	1/20	3	1/20	3	67
Soil (Leachate- TCLP)														
TCLP-Volatiles (SW-846 1311/8260-Benzene)	3	NA	--	NA	--	NA	--	NA	--	NA	--	NA	--	3
TCLP-Semivolatile (SW-846 8270C)	3	NA	--	NA	--	NA	--	NA	--	NA	--	NA	--	3
TCLP-Metals (EPA 6010B TCLP)	3	NA	--	NA	--	NA	--	NA	--	NA	--	NA	--	3
Reactivity	3	NA	--	NA	--	NA	--	NA	--	NA	--	NA	--	3
Flash point (Ignitability)	3	NA	--	NA	--	NA	--	NA	--	NA	--	NA	--	3
Groundwater														
Volatile Organic Compounds (SW-846 8260- TCL)	36	NA	10	1/day	10	1/20	3	1/20	3	1/20	3	1/20	3	68
Semivolatile Organic Compounds (SW-846 8270-TCL)	36	NA	--	1/day	10	1/20	3	1/20	3	1/20	3	1/20	3	58
TAL Metals (EPA Method 6010B and 7471A)	36	NA	--	1/day	10	1/20	3	1/20	3	1/20	3	1/20	3	58
Alkalinity (EPA Method 310.2)	12	NA	--	1/day	10	1/20	1	1/20	1	1/20	1	1/20	1	26
Alkalinity-Bicarbonate (EPA Method 310.2)	12	NA	--	1/day	10	1/20	1	1/20	1	1/20	1	1/20	1	26
Ethane (RSK175)	12	NA	--	1/day	10	1/20	1	1/20	1	1/20	1	1/20	1	26
Ethene (RSK175)	12	NA	--	1/day	10	1/20	1	1/20	1	1/20	1	1/20	1	26
Ferrous Iron (SM3500-Fe-D)	12	NA	--	1/day	10	1/20	1	1/20	1	1/20	1	1/20	1	26
Manganese (EPA Method 6010)	12	NA	--	1/day	10	1/20	1	1/20	1	1/20	1	1/20	1	26
Methane (RSK175)	12	NA	--	1/day	10	1/20	1	1/20	1	1/20	1	1/20	1	26
Nitrate (EPA Method 353.2)	12	NA	--	1/day	10	1/20	1	1/20	1	1/20	1	1/20	1	26
Nitrogen as ammonia (350.1)	12	NA	--	1/day	10	1/20	1	1/20	1	1/20	1	1/20	1	26
Phosphorous as orthophosphate (SM4500-P-E)	12	NA	--	1/day	10	1/20	1	1/20	1	1/20	1	1/20	1	26
Sulfate (ASTM D516-90)	12	NA	--	1/day	10	1/20	1	1/20	1	1/20	1	1/20	1	26
Sulfide (SM4500-S-2D)	12	NA	--	1/day	10	1/20	1	1/20	1	1/20	1	1/20	1	26
Total Dissolved Organic Carbon (SM5310-D)	12	NA	--	1/day	10	1/20	1	1/20	1	1/20	1	1/20	1	26
Total Dissolved Solids (SM2540-C)	12	NA	--	1/day	10	1/20	1	1/20	1	1/20	1	1/20	1	26

Notes:

Sample counts are an approximation.

1/day - One rinse blank per day or one per 20 samples, whichever is more frequent. Rinse blanks not required when dedicated sampling equipment is used.

Freq - Frequency.

NA - Not Applicable.

No. - Number.

QC - Quality Control.

Table 2. Analytical Quality Control Limits[1], Quality Assurance Project Plan, Chevron Environmental Management Company, Former Gulf Terminal Site, Oceanside, NY

Parameter	Accuracy - % Recovery			Precision - RPD		
	Surrogate	MS/MSD	LCS	MS/MSD	Lab Duplicate	Field Duplicate
Soil						
Volatile Organics	60-140	60-140	70-140	25	--	100
Semivolatile Organics	20-140	20-140	40-120	40	--	100
Metals	--	80-120	80-120	--	20	100
Total Organic Carbon	--	70-130	70-130	--	30	100
Soil (leachates)						
Volatile Organics	75-115	60-145	70-140	20	--	--
Reactivity	--	--	70-130	--	30	--
Corrosivity	--	--	70-130	--	30	--
Groundwater						
Volatile Organics	75-115	60-145	70-140	20	--	50
Semivolatile Organics	20-140	20-130	40-120	40	--	50
Metals	--	80-120	80-120	--	30	50
Wet Chemistry and Miscellaneous	--	70-130	70-130	--	30	50

Note:

[1] The listed QC limits are based on SW-846 guidance and are advisory. The actual limits are determined based on laboratory performance. Frequent failure to meet the QC limits; however, warrant investigation of the laboratory.

Table 3. Parameters, Methods, and Target Reporting Limits, Quality Assurance Project Plan, Chevron Environmental Management Company, Former Gulf Terminal Site, Oceanside, NY

	Water (ug/L)			Soil (ug/kg)		
	NYS GW	Laboratory	Laboratory	Unrestricted Use	Laboratory	Laboratory
Analyte	**STD./G.V.[3]**	**MDL**	**RL**	**SCOs[4]**	**MDL**	**RL**
Volatile Organic Compounds 8260 [1]						
1,1,1-Trichloroethane	5	0.82	1	680	0.36	5
1,1,2,2-Tetrachloroethane	5	0.21	1	NA	0.81	5
1,1,2-Trichlorotrifluoroethane	5	0.31	1	NA	1.1	5
1,1,2-Trichloroethane	1	0.23	1	NA	0.65	5
1,1-Dichloroethane	5	0.38	1	270	0.61	5
1,1-Dichloroethene	5	0.29	1	330	0.61	5
1,2,4-Trichlorobenzene	5	0.41	1	NA	0.30	5
1,2-Dibromo-3-chloropropane	0.04	0.39	1	NA	2.5	5
1,2-Dibromoethane	0.0006	0.73	1	NA	0.64	5
1,2-Dichlorobenzene	3	0.79	1	1,100	0.39	5
1,2-Dichloroethane	0.6	0.21	1	20 [c]	0.25	5
1,2-Dichloropropane	1	0.72	1	NA	2.5	5
1,3-Dichlorobenzene	3	0.78	1	2,400	0.26	5
1,4-Dichlorobenzene	3	0.84	1	1,800	0.70	5
2-Butanone	50	1.3	10	NA	1.8	25
2-Hexanone	50	1.2	5	NA	2.5	25
4-Methyl-2-pentanone	NA	2.1	5	NA	1.6	25
Acetone	50	3.0	10	50	4.2	25
Benzene	1	0.41	1	60	0.24	5
Bromodichloromethane	50	0.39	1	NA	0.67	5
Bromoform	50	0.26	1	NA	2.5	5
Bromomethane	5	0.69	1	NA	0.45	5
Carbon disulfide	60	0.19	1	NA	2.5	5
Carbon tetrachloride	5	0.27	1	760	0.48	5
Chlorobenzene	5	0.75	1	1,100	0.66	5
Chlorodibromomethane	NA	0.32	1	NA	0.64	5
Chloroethane	5	0.32	1	NA	1.1	5
Chloroform	7	0.34	1	370	0.31	5
Chloromethane	5	0.35	1	NA	0.30	5
cis-1,2-Dichloroethene	5	0.81	1	250	0.64	5
cis-1,3-Dichloropropene	0.4	0.36	1	NA	0.72	5
Cyclohexane	NA	0.18	1	NA	0.70	5
Dichlorodifluoromethane	5	0.68	1	NA	0.41	5
Ethylbenzene	5	0.74	1	1,000	0.34	5
Isopropylbenzene	5	0.79	1	NA	0.75	5
Methyl acetate	NA	0.50	1	NA	0.93	5
Methyl tert-butyl ether	10	0.16	1	930	0.49	5
Methylcyclohexane	NA	0.16	1	NA	0.76	5
Methylene chloride	5	0.44	1	50	2.3	5
Styrene	5	0.73	1	NA	0.25	5
Tetrachloroethene	5	0.36	1	1,300	0.67	5
Toluene	5	0.51	1	700	0.38	5
trans-1,2-Dichloroethene	5	0.90	1	190	0.52	5
trans-1,3-Dichloropropene	0.4	0.37	1	NA	2.2	5
Trichloroethene	5	0.46	1	470	1.1	5
Trichlorofluoromethane	5	0.88	1	NA	0.47	5
Vinyl chloride	2	0.90	1	20	0.61	5
Xylenes (total)	5	0.66	2	260	0.84	10
Semivolatile Organic Compounds 8270 [2]						
2,4,5-Trichlorophenol	NA	0.48	5	NA	37	170
2,4,6-Trichlorophenol	NA	0.61	5	NA	11	170
2,4-Dichlorophenol	5	0.51	5	NA	8.8	170
2,4-Dimethylphenol	50	0.50	5	NA	46	170
2,4-Dinitrophenol	10	2.2	10	NA	59	330
2,4-Dinitrotoluene	5	0.45	5	NA	26	170
2,6-Dinitrotoluene	5	0.40	5	NA	41	170
2-Chloronaphthalene	10	0.46	5	NA	11	170
2-Chlorophenol	NA	0.53	5	NA	8.6	170
2-Methylnaphthalene	NA	0.60	5	NA	2.0	170
2-Methylphenol	NA	0.40	5	NA	5.2	170
2-Nitroaniline	5	0.42	10	NA	54	330
2-Nitrophenol	NA	0.48	5	NA	7.7	170
3,3'-Dichlorobenzidine	5	0.40	5	NA	150	170

See Notes on Page 3.

Table 3. Parameters, Methods, and Target Reporting Limits, Quality Assurance Project Plan, Chevron Environmental Management Company, Former Gulf Terminal Site, Oceanside, NY

	Water (ug/L)			Soil (ug/kg)		
	NYS GW	Laboratory	Laboratory	Unrestricted Use	Laboratory	Laboratory
Analyte	STD./G.V.[3]	MDL	RL	SCOs[4]	MDL	RL
Semivolatile Organic Compounds 8270 [2] (Cont.)						
3-Nitroaniline	5	0.48	10	NA	39	330
4,6-Dinitro-2-methylphenol	NA	2.2	10	NA	58	330
4-Bromophenyl-phenylether	NA	0.45	5	NA	54	170
4-Chloro-3-methylphenol	NA	0.45	5	NA	6.9	170
4-Chloroaniline	5	0.59	5	NA	50	170
4-Chlorophenyl-phenylether	NA	0.35	5	NA	3.6	170
4-Methylphenol	NA	0.36	10	NA	9.4	330
4-Nitroaniline	5	0.25	10	NA	19	330
4-Nitrophenol	NA	1.5	10	NA	41	330
Acenaphthene	20	0.41	5	20,000	2.0	170
Acenaphthylene	NA	0.38	5	100,000 [a]	1.4	170
Acetophenone	NA	0.54	5	NA	8.7	170
Anthracene	50	0.28	5	100,000 [a]	4.3	170
Atrazine	7.5	0.46	5	NA	7.5	170
Benzaldehyde	NA	0.27	5	NA	19	170
Benzo(a)anthracene	0.002	0.36	5	1,000 [c]	2.9	170
Benzo(a)pyrene	ND	0.47	5	1,000 [c]	4.1	170
Benzo(b)fluoranthene	0.002	0.34	5	1,000 [c]	3.3	170
Benzo(g,h,i)perylene	NA	0.35	5	100,000	2.0	170
Benzo(k)fluoranthene	0.002	0.73	5	800 [c]	1.9	170
Biphenyl	NA	0.65	5	NA	11	170
bis(2-Chloroethoxy)methane	5	0.35	5	NA	9.2	170
bis(2-Chloroethyl)ether	NA	0.40	5	NA	15	170
Bis(2-chloroisopropyl) ether	NA	0.52	5	NA	18	170
bis(2-Ethylhexyl)phthalate	5	1.8	5	NA	54	170
Butylbenzylphthalate	50	0.42	5	NA	45	170
Caprolactam	NA	2.2	5	NA	73	170
Carbazole	NA	0.30	5	NA	2.0	170
Chrysene	0.002	0.33	5	1,000 [c]	1.7	170
Dibenz(a,h)anthracene	NA	0.42	5	330 [b]	2.0	170
Dibenzofuran	NA	0.51	10	NA	1.8	170
Diethylphthalate	50	0.22	5	NA	5.1	170
Dimethylphthalate	50	0.36	5	NA	4.4	170
Di-n-butyl phthalate	50	0.31	5	NA	58	170
Di-n-octyl phthalate	50	0.47	5	NA	3.9	170
Fluoranthene	50	0.40	5	100,000 [a]	2.4	170
Fluorene	50	0.36	5	30,000	3.9	170
Hexachlorobenzene	0.04	0.51	5	NA	8.4	170
Hexachlorobutadiene	0.5	0.68	5	NA	8.6	170
Hexachlorocyclopentadiene	5	0.59	5	NA	51	170
Hexachloroethane	5	0.59	5	NA	13	170
Indeno(1,2,3-cd)pyrene	0.002	0.47	5	500 [c]	4.7	170
Isophorone	50	0.43	5	NA	8.4	170
Naphthalene	10	0.76	5	12,000	2.8	170
Nitrobenzene	0.4	0.29	5	NA	7.5	170
N-Nitrosodiphenylamine	50	0.54	5	NA	13	170
N-Nitrosos-di-n-propylamine	50	0.51	5	NA	9.2	170
Pentachlorophenol	1	2.2	10	800 [b]	58	330
Phenanthrene	50	0.44	5	100,000	3.5	170
Phenol	1	0.39	5	330 [b]	18	170
Pyrene	50	0.34	5	100,000	1.1	170
Metals						
Aluminum	100	45	200	NA	580	10000
Antimony	3	6.79	20	NA	540	15000
Arsenic	25	5.55	10	13,000 [c]	220	2000
Barium	1,000	0.3	2	350,000 [c]	10	500
Beryllium	3	0.22	2	7,200	6	200
Cadmium	5	0.33	1	2,500 [c]	30	200
Calcium	NA	100	500	NA	3300	50000
Chromium	50	0.87	4	1,000 [b] (hexavalent) 30,000 [c] (trivalent)	90	500
Cobalt	5	0.63	4	NA	50	500
Copper	200	1.5	10	50,000	60	1000
Iron	300	19.3	50	NA	1100	10000

See Notes on Page 3.

Table 3. Parameters, Methods, and Target Reporting Limits, Quality Assurance Project Plan, Chevron Environmental Management Company, Former Gulf Terminal Site, Oceanside, NY

Analyte	Water (ug/L) NYS GW STD./G.V.[3]	Water (ug/L) Laboratory MDL	Water (ug/L) Laboratory RL	Soil (ug/kg) Unrestricted Use SCOs[4]	Soil (ug/kg) Laboratory MDL	Soil (ug/kg) Laboratory RL
Metals (Cont.)						
Lead	25	3	5	63,000 [c]	120	1000
Magnesium	35,000	43.4	200	NA	927	20000
Manganese	300	0.19	3	1,600,000 [c]	32	200
Mercury	0.7	0.12	0.2	180 [c]	8	20
Nickel	100	1.26	10	30,000	80	5000
Potassium	NA	50	500	NA	3000	30000
Wet Chemistry and Miscellaneous						
Alkalinity (total) (EPA Method 310.2)	NA	400	10000	NA	NA	NA
Alkalinity-Bicarbonate (EPA Method 310.2)	NA	400	10000	NA	NA	NA
Ferrous Iron (SM3500-Fe-D)	300	19.3	50	NA	NA	NA
Manganese (EPA Method 6010)	300	0.19	3	NA	NA	NA
Methane (RSK 175)	NA	22	1	NA	NA	NA
Ethene (RSK 175)	NA	52	1.5	NA	NA	NA
Ethane (RSK 175)	NA	49	1.5	NA	NA	NA
Nitrate (EPA Method 353.2)	10000	11	5	NA	NA	NA
Nitrite(EPA Method 353.2)	NA	20	50	NA	NA	NA
Nitrogen as ammonia (EPA Method 350.1)	2000	9	20	NA	NA	NA
Phosphorous as orthophosphate (SM4500-P-E)	20	6	20	NA	NA	NA
Sulfate (ASTM D516-90)	250000	1490	50000	NA	NA	NA
Sulfide (SM4500-S-2D)	50	52	10000	NA	NA	NA
Total Dissolved Solids (SM2540-C)	NA	400	10000	NA	NA	NA
Total Dissolved Organic Carbon (SM5310-D)	NA	434	1000	NA	NA	NA

Notes:

[1] USEPA. Office of Solid Waste and Emergency Response. *Test Methods for Evaluating Solid Waste SW-846 3rd ed. Washington, D.C. 1996.*

[2] The target reporting limits are based on wet weight. The actual reporting limits will vary based on sample weight and moisture content.

[3] Water guidance values (GV) are as presented in the NYSDEC, Division of Water, Technical and Operation Guidance Series (TOGS) document titled, *Ambient Water Quality Standards and Guidance Values and Groundwater Effluent Limitations* (TOGS 1.1.1), dated June 1998, last revised April 2000.

[4] Unrestricted use Soil Cleanup Objectives (SCOs) are as presented in Table 375-6.89(a) of 6 NYCRR Part 375-6.8 effective as of December 14, 2006. The following footnotes on the unrestricted use SCOs are taken directly from Table 375-6.89(a):

[a] The SCOs for unrestricted use were capped at a maximum value of 100 ppm. See Technical Support Document (TSD), section 9.3.

[b] For constituent where the calculated SCO was lower than the contract required quantitation limit (CRQL), the CRQL is used as the Track 1 SCO value.

[c] For constituents where the calculate SCO was lower than the rural soil background concentration, as determined by the Department and Department of Health rural soil survey, the rural soil background concentration is used as the Track 1 SCO value for this use of the site.

* Based on response to MDL Verification due to non-linearity compound.

** MDL Verification spike did not yield any recovery at calculated MDL. MDL adjusted to reflect concentration which can be detected (spike level of replicates).

Table 4. Sample Containers, Preservation, and Holding Times, Quality Assurance Project Plan, Chevron Environmental Management Company, Former Gulf Terminal Site, Oceanside, NY

Parameter	Method[1]	Bottle Type	Preservation	Holding Time[2]
Soil				
TCL Volatile Organic Compounds	SW-846 8260B	1 - 2 oz glass jar with Teflon®-lined lid	Cool to 4°C	12 days to analysis
TCL Semivolatile Organic Compounds	SW-846 8270C	1 - 4 oz glass jar with Teflon®-lined lid	Cool to 4°C	10 days to extraction
Ferrous Iron	SM3500-Fe-D	1 - 4 oz glass jar with Teflon®-lined lid	Cool to 4°C	immediately after extraction
Total Mercury	EPA Method 7471A	1 - 8 oz poly container	HNO3 to pH<2	28 days to analysis
TAL Metals	EPA Method 6010B	1 - 4 oz glass jar	Cool to 4°C	180 days to analysis
Soil Leachates				
Reactivity	SW846 Sect. 7.3 withdrawn	1 - 4 oz glass jar with Teflon®-lined lid	Cool to 4°C	180 days to analysis
Cyanide (Reactive)	SW846 Sect. 7.3 withdrawn	1 - 4 oz glass jar with Teflon®-lined lid	Cool to 4°C	180 days to analysis
Sulfide (Reactive)	SW846 Sect. 7.3 withdrawn	1 - 4 oz glass jar with Teflon®-lined lid	Cool to 4°C	180 days to analysis
Flash point (Ignitability)	EPA Method 1010	1 - 4 oz glass jar with Teflon®-lined lid	Cool to 4°C	180 days to analysis
TCLP-Volatiles	SW-846 1311/8260-Benzene	1 - 4 oz glass jar with Teflon®-lined lid	Cool to 4°C	5 days to TCLP extraction
TCLP Volatile Organic Compounds	EPA Method 8260B	1 - 4 oz glass jar	Cool to 4°C	12 days to analysis
TCLP Semivolatile Organic Compounds	EPA Method 8270C	1 - 16 oz glass jar	Cool to 4°C	5 days to analysis
TCLP Metals	EPA Method 6010B	1 - 16 oz glass jar	Cool to 4°C	180 days to analysis
TCLP Mercury	EPA Method 7470A	1 - 16 oz glass jar	Cool to 4°C	26 days to analysis
Water				
TCL Volatile Organic Compounds	EPA Method 8260B	2 - 40 ml glass vials with Teflon®-lined lid	HCl to pH<2, Cool to 4°C	12 days to analysis
TCL Semivolatile Organic Compounds	EPA Method 8270	2 - 1 liter amber glass bottle with Teflon®-lined lid	Cool to 4°C	5 days to extraction
Alkalinity (total)	EPA Method 310.2	1 - 8 oz poly container, no headspace	Cool to 4°C	12 days to analysis
Alkalinity-Bicarbonate	EPA Method 310.2	"	"	12 days to analysis
Ferrous Iron	SM3500-Fe-D	1 - 4 oz plastic	Cool to 4°C	immediately
Manganese	EPA Method 6010	1 - 8 oz plastic	HNO3 to pH<2	180 days to analysis
Methane	RSK 175	2 - 40 ml glass vials with Teflon®-lined lid	HCl to pH<2, Cool to 4°C	12 days to analysis
Ethane	RSK 175	2 - 40 ml glass vials with Teflon®-lined lid	HCl to pH<2, Cool to 4°C	12 days to analysis
Ethene	RSK 175	2 - 40 ml glass vials with Teflon®-lined lid	HCl to pH<2, Cool to 4°C	12 days to analysis
Nitrate	EPA Method 353.2	1 - 8 oz plastic	Cool to 4°C	24 hours to analysis
Nitrite	EPA Method 353.2	1 - 8 oz plastic	Cool to 4°C	24 hours to analysis
Nitrogen as ammonia	EPA Method 350.1	1 - 8oz plastic	H2SO4 to pH<2	26 days to analysis
Phosphorous as orthophosphate	EPA Method 4500-PE	1 - 8 oz plastic	Cool to 4°C	24 hours to analysis
Sulfate	ASTM D516-90	1 - 8 oz plastic	Cool to 4°C	26 days to analysis
Sulfide	SM4500-S-2D	1 - 8 oz plastic	Zinc Acetate + NaOH to pH>9	5 days to analysis
TAL Metals	EPA Method 6010B	1 - 8 oz plastic	HNO3 to pH<2	180 days to analysis
Total Mercury	EPA Method7470A	1 - 8 oz plastic	HNO3 to pH<2	26 days to analysis
Total Dissolved Solids	EPA Method 2540C	1 - 4 oz plastic	Cool to 4°C	5 days to analysis
Total Dissolved Organic Carbon	EPA Method 5310D	2 - 40 ml glass vials with Teflon®-lined lid	HCl to pH<2, filtered	26 days to analysis

Notes:

[1] USEPA. Office of Solid Waste and Emergency Response. *Test Methods for Evaluating Solid Waste. SW-846 3rd ed. Washington, D.C. 1996.*
USEPA. Methods for Chemical Analysis of Water and Waste. EMSL-Cincinnati. 1983.
APHA. *Standard Methods for the Examination of Water and Wastewater.* Washington, DC. 1998.
ASTM International. 2003. *Annual Book of ASTM Standards 2003 Section 4 Construction*, Volume 04.08. West Conshohocken, PA. ASTM International.
Department of the Army. 1986. *Engineering Manual Laboratory Soils Testing.* Washington, D.C. Department of the Army, Office of the Chief of Engineers.

[2] All holding times are measured from date of collection.

VTSR = Verified Time of Sample Receipt (ASP 2005 hold times expressed as VTSR).

Table 5. Electronic Data Deliverable (EDD) Format, Quality Assurance Project Plan, Chevron Environmental Management Company, Former Gulf Terminal Site, Oceanside, NY

Field	Required	Must Be Date	Must Be Number	Maximum Length	Has List Of Values	Part of Unique Index	Default Value Available	Populated By EIM	Position In EDD	Description	Comments
FIELD_SAMPLE_ID	Cond			25		Yes			1	ID assigned to the sample by field sampling team or other site, facility, or project personnel.	Must be left blank for lab-originated samples (e.g., lab control samples, method blanks, blank spikes, etc.). Should be populated for lab duplicates and matrix spikes and duplicates (if the sample that is spiked is the client sample).
LAB_ID	Yes			10	Yes	Yes			2	ID assigned to a laboratory. This should be a meaningful value as it is used in many drop-down lists.	
ANALYTICAL_METHOD	Yes			30	Yes	Yes			3	Number/ID of an analytical method.	
ANALYSIS_DATE	Yes	Yes				Yes			4	Date sample was analyzed.	
PARAMETER_CODE	Yes			12	Yes	Yes	Yes		5	ID/Code assigned to the analytical parameter. This should be the CAS Number unless the parameter does not have one.	
RESULT_TYPE_CODE	Yes			5	Yes	Yes	Yes		6	Coded value for type of result. Associated with list of valid values. Typical entries here are TRG (Target), SPK (Spike), and TIC (Tentatively Identified Compound.	Generally, the entry in the sample purpose field determines whether a result ends up in the Field or Lab Sample Result tables. The one exception is Surrogates. Those associated with Field Samples are loaded into the Lab Sample Result table.
LAB_RESULT	Cond		Yes	10				Yes	7	Result reported by the lab. If the parameter was not detected, the value in this field should be the same as the entry in the LAB_DETECTION_LIMIT column.	Required of all samples except surrogates and spikes. If not detected, enter the laboratory reporting limit here unless project requirements dictate that nondetects be reported at the method detection limit. Results for spikes and surrogates are reported in the SPIKE_ADDED, SPIKED_RESULT, and SPIKE_RECOVERY fields.
LAB_UNITS	Yes			10	Yes				8	Units associated with the entries in the LAB_RESULT, ORIGINAL_LAB_RESULT, and LAB_DETECTION_LIMIT columns in the FIELD_SAMPLE_RESULT and LAB_SAMPLE_RESULT tables.	Enter the units associated with the entry in the LAB_RESULT or SPIKED_RESULT column.
LAB_DETECTION_LIMIT	Yes			10			Yes		9	The lab reporting limit should be recorded in this field. This is the lowest concentration at which an analyte can be detected in a sample and its concentration reported with a reasonable degree of accuracy and precision.	
METHOD_DETECTION_LIMIT				10					10	Standard method detection limit (MDL) as defined by US EPA.	

See Notes on Page 3.

Table 5. Electronic Data Deliverable (EDD) Format, Quality Assurance Project Plan, Chevron Environmental Management Company, Former Gulf Terminal Site, Oceanside, NY

Field	Required	Must Be Date	Must Be Number	Maximum Length	Has List Of Values	Part of Unique Index	Default Value Available	Populated By EIM	Position In EDD	Description	Comments
LAB_REPORTING_LIMIT_TYPE				10	Yes				11	Coded value identifying the type of reporting limit (e.g., practical quantitation limit, instrument detection limit, etc.). Required for Geotracker exports (equivalent column in Geotracker is REPDLVQ). Associated with a list of values.	
LAB_MATRIX	Yes			10	Yes				12	Matrix of the lab sample.	
LAB_SAMPLE_ID	Yes			20					13	Internal ID assigned to the sample by the lab.	Often historical data do not have a lab sample ID, and as such, this field can be left blank in EIM. However, all new data should have an entry in this field. The validation module will not work without it.
ANALYSIS_TIME	Yes			5		Yes	Yes		14	Time that a sample was analyzed.	
LAB_QUALIFIER				10					15	Qualifier assigned to the analytical result by the lab.	In the Validation module, entries with a U in them are assumed to be nondetects and those with a J estimated values.
DILUTION_FACTOR	No			7					16	Dilution factor.	Should be provided when sample is diluted.
PREP_METHOD	No			30					17	Method used to extract or otherwise prepare the sample prior to analysis.	Report a value only if sample was extracted or otherwise prepped before it was analyzed.
PREP_DATE	No	Yes							18	Date that sample was extracted or otherwise prepared.	Report a value only if sample was extracted or otherwise prepped before it was analyzed.
SAMPLE_DELIVERY_GROUP				20					19	ID/Name of Sample Delivery Group.	Although Sample Delivery Group is not a required field, it must be populated for EIM's validation module to work.
PARAMETER_NAME	Yes			60					20	Name of the analytical parameter.	
ANALYSIS_TYPE_CODE	Yes			5	Yes	Yes	Yes		21	Coded value designating the type of analysis. Associated with list of valid values. Typical entries here are INIT (Initial), REEXT (Reextraction), and REANL (Reanalysis).	
FILTERED_FLAG	Yes			1	Yes	Yes	Yes		22	Flag (generally Y/N) indicating whether the sample was filtered by lab. At a project's/site's discretion, this value can be updated if sample was initially filtered in the field.	
LEACHED_FLAG	Yes			1	Yes	Yes	Yes		23	Flag (generally Y/N) indicating whether the sample was leached. Associated with list of valid values.	
SAMPLE_DATE	Yes	Yes						Yes	24	Date field sample collected in field or date or origin of lab sample.	This field must be filled in for all samples.

See Notes on Page 3.

Table 5. Electronic Data Deliverable (EDD) Format, Quality Assurance Project Plan, Chevron Environmental Management Company, Former Gulf Terminal Site, Oceanside, NY

Field	Required	Must Be Date	Must Be Number	Maximum Length	Has List Of Values	Part of Unique Index	Default Value Available	Populated By EIM	Position In EDD	Description	Comments
SAMPLE_PURPOSE	Yes			5	Yes				25	Coded value identifying purpose of sample. Associated with list of values. Typical entries in this column are REG (Regular Sample), FD (Field Duplicate), and TB (Trip Blank).	This field must be filled in for all samples.
LOCATION_ID	Yes			30	Yes				26	ID/Name assigned to the sampling location. Should be same as well ID if well has only one screen. If well has multiple screens, each different screened interval should be assigned its own unique Location ID.	
SAMPLE_NAME				40					27	Descriptive name assigned to sample by field/project/site personnel.	
SAMPLE_TIME	Yes			5			Yes		28	Time sample was collected/prepared in field.	
SAMPLE_MATRIX	Yes			10	Yes				29	Matrix of sample. Associated with list of valid values. Typical entries here are WATER and SOIL.	
SAMPLE_START_DEPTH			Yes	8					30	Starting depth of sampling interval.	
SAMPLE_END_DEPTH			Yes	8					31	Ending depth of sampling interval.	
SAMPLE_DEPTH_UNITS				10	Yes				32	Units associated with entries in the SAMPLE_START_DEPTH and SAMPLE_END_DEPTH columns.	
SAMPLE_TYPE	Yes			10	Yes				33	Coded value for type of sample. Associated with list of valid values. Typical entries here are GW (Groundwater), SW (Surface water), and BW (Blank water).	

Notes:

1. The information provided is based on LOCUS/EIM formatting.
2. Depth-related fields may be left blank for samples and matrices for which they are not applicable.

ARCADIS

Attachment 1

Laboratory Standard Operating Procedures

Title: Thermo Jarrell Ash 61E Trace Analysis
Method No(s). 6010B/200.7/CLP

Once printed, this is considered an uncontrolled document

Approvals (Signature/Date):

E. Scott Wagner 02/08/10
E. Scott Wagner Date
Department Manager

Kenneth E. Kasperek 02/08/10
Kenneth Kasperek Date
Health & Safety Manager

Peggy Gray-Erdmann 02/08/10
Peggy Gray-Erdmann Date
Quality Assurance Manager

02/08/10
Christopher Spencer Date
Laboratory Director

Facility Distribution No. ___________ **Distributed To:**________________________

1.0 Scope and Application

1.1 This SOP is specific for methods (SW-846) 6010B, 200.7, and CLP. This SOP discusses the procedures as they are performed at TestAmerica Buffalo. Table 17.15 summarizes the actual method criteria.

1.2 At TestAmerica Buffalo, there are two ICP Analyzers. They are designated as Trace #1 and Trace #2.

1.3 Table 17.1 lists the elements that are analyzed on each Trace.

1.4 Tables 17.2 and 17.3 list the approximate instrument detection limits (IDL's) which can be achieved on each Trace. IDL's are recalculated quarterly or when a significant instrumentation change occurs.

1.5 Table 17.4 lists the wavelengths and typical background points used on each Trace.

1.6 The linear range is the concentration range over which the instrument response to an analyte is linear. Table 17.5 lists the approximate linear ranges of each Trace. Linear ranges are recalculated quarterly or when a significant instrumentation change occurs.

1.7 Interelement correction factors (IECs) are used to correct for interferences caused by spectral overlap of elemental lines. At TestAmerica Buffalo, IECs are verified and calculated quarterly or when an instrumentation change occurs.

1.8 All samples, standards, and blanks are matrix matched to achieve an aqueous solution containing 6% HNO_3 and 5% HCl by volume.

1.9 Analytes, Matrix(s), and Reporting Limits

1.9.1 Soluble water samples and digestates of waters, TCLPs, total recoverables, soils, sludges, sediments, and other wastes.

1.9.2 Tables 17.2 and 17.3 list achievable Instrumental Detection Limits (IDLs) and Method Detection Limits. The laboratory IDLs are updated quarterly and the MDLs are updated annually. The current IDLs and MDLs are maintained in the laboratory LIMs system.

1.9.3 The laboratory standard Practical Quantitation Limits (PQLs) are also listed in Tables 17.2 and 17.3. The standard laboratory PQLS remain static and are only changed if there is a major update to the analytical system.

1.9.4 On occasion clients may request modifications to this SOP. These modifications are handled following the procedures outlined in the Quality Assurance Manual.

2.0 Summary of Method

2.1 Samples are nebulized and the aerosol that is produced is transported to the plasma torch where excitation occurs. Characteristic atomic-line emission spectra are produced by radio frequency inductively coupled plasma (ICP). The spectra are dispersed by a grating spectrometer and the intensities of the emission lines are monitored by photomultiplier tubes. The photocurrents from the photomultiplier tubes are processed and controlled by a computer system. A background correction technique is required to compensate for variable background contribution to the determination of trace elements. Background must be measured adjacent to analyte lines during analysis. The position selected for the background intensity measurement, on either or both sides of the analytical line, will be determined by the complexity of the spectrum adjacent to the analyte line. The position used must be free of spectral interferences and reflect the same change in background intensity as occurs at the analyte wavelength measured.

2.2 This SOP contains the procedures for the daily operation of the ICAP 61E Trace Analyzer. This SOP also contains procedures for calibration, standard and sample preparation, maintenance, data handling, and quality control. This SOP is based on methods 6010B (SW-846), 200.7 and CLP.

2.3 On a given day the normal steps in operating the Trace include:

- Perform any routine maintenance, if required.
- Instrument start-up and warm-up, if instrument is not already conditioned
- Preparation of standards. All standards and quality control standards are prepared from stock solutions, as needed (6-month expiration date.) The Calibration Standards are made every 3-7 days.
- Type-up a run to analyze. A run is simply a sequence of samples with all required quality control that is analyzed as a single unit.
- Set-up the autosampler.
- Prepare all the samples for analysis including the required spikes, serial dilutions, and other quality control samples.
- Analyze the samples.
- When the analysis is complete, check the data for compliance with 6010B, 200.7, or CLP whichever is applicable.
- Log in compliant data.
- Dispose of samples and standards appropriately. Clean-up lab area.

2.4 If the instrument is not operating properly or requires any maintenance, refer to Section 10.3 for help with routine maintenance and troubleshooting.

3.0 Definitions

3.1 Trace - Abbreviation for Thermo Jarrell Ash ICAP 61E Trace Analyzer. The Trace ICP has a viewing angle along the long axis of the torch.

3.2 IECs - Interelement correction factors. Used to correct for interferences caused by spectral overlap of element lines. See Section 9.0.16 for procedures on determining IECs.

3.3 Linear Range - Also referred to as linear dynamic range. The linear range is the concentration range over which the instrument response to an analyte is linear. Refer to Section 9.16 for the determination of linear ranges.

3.4 IDL - Instrument detection limit. The IDL of an element is the lowest calculated concentration that the instrument can measure. See section 9.0.16 for procedures on determining IDLs.

3.5 MDL – Method Detection Limit. The minimum amount of a substance that can be measured with a specified degree of confidence that the amount is greater than zero.

3.6 PQL – Practical Quantitation Limit. The minimum amount of a substance that can be *quantitatively* measured with a specified degree of confidence and within *accuracy and precision guidelines*.

3.7 Calibration Standards - A series of solutions containing known amounts of each element with a matrix similar to samples. These solutions are used to calibrate the instrument.

.3.8 ICV - Initial calibration verification, which must be from a source different from that of the calibration standard

3.9 ICB - Initial calibration blank.

3.10 IFA - Interference check sample containing only high levels of Al, Fe, Ca, and Mg.

3.11 IFB - Interference check sample containing high levels of Al, Fe, Ca, and Mg, and low levels of all other elements that are analyzed by the Trace.

3.12 CCV - Continuing calibration verification.

3.13 CCB - Continuing calibration blank.

3.14 BS - Blank Spike. A quality control sample containing known concentration of analytes that is taken through the entire digestion and analysis procedure.

3.15 BLK- Method Blank - A blank sample that is taken through each step of the analytical procedure, including the digestion procedure if it is used.

3.16 Method of Standard Addition - Involves the analysis of an unknown sample and the analysis of an unknown sample with a known amount of a standard added. This procedure may be used when matrix interference is suspected.

3.17 Calibration Blank - A blank solution containing 6% HNO_3 and 5% HCl for calibration.

3.18 Total Metals – The concentration determined on an unfiltered sample following vigorous digestion.

3.19 Soluble or Dissolved Metals – The concentration determined on a sample after passing through a 0.45 um membrane. Acidification and digestion are performed after filtration.

3.20 ELGA water – This is blank reagent water that is deionized, filtered, and has a resistivity of 18 $M\Omega cm^{-1}$.

3.21 Calibration Curvefit- This process allows individual element wavelengths to be standardized using an extended calibration with more standards to produce a more linear and precise response over a greater range. The calibration is then fitted to a curve, which can be resloped during the instrument standardization.

4.0 Interferences

There are four main types of interferences. They are - spectral, physical, chemical and memory interferences.

4.1 Spectral Interferences

These types of interferences are caused primarily from the overlap of elemental lines and background contributions. Interferences from spectral overlap are eliminated by the use of interelement correction factors. Interferences caused by background contributions are eliminated by the use of background correction. Table 17.4 lists the typical background points.

4.2 Physical Interferences

These types of interferences are caused by differences between the physical properties of standards and samples. The major source of these interferences is a high dissolved solids concentration in a sample. Physical interferences are minimized by using an internal standard, diluting the samples and/or performing the method of standard addition.

Additionally, high salt concentrations can cause a buildup of salt at the tip of the nebulizer. This effect is minimized on the Trace by use of an Argon Saturator and a Noordermeer V-Groove nebulizer designed for high dissolved solid use.

4.3 Chemical Interferences

These are generally caused by molecular compound formation, ionization effects, and solvent evaporization effects. These effects can be minimized by careful selection of the operating conditions, by buffering the sample, or by standard addition procedures. At TestAmerica Buffalo, buffer solution of Li (NO_3) is added on-line to minimize the ionization effects of the high level of easily ionized elements such as K and Na.

4.4 Memory Interferences

Memory interferences result when analytes in a previous sample contribute to the signals measured in a new sample. To minimize memory effects, a rinse period of at least 60 seconds is used between samples and standards. If memory interference is suspected, the sample must be reanalyzed after a rinse period of sufficient length.

4.5 The following tests may be performed to check for physical and chemical interferences. A serial dilution is performed on a representative sample from each sample batch. A post digestion spike is performed based upon client requirements. The sample batch does not exceed twenty samples.

4.5.1 Serial Dilution (SRD)

A serial dilution (1:5) is performed on a representative sample of each matrix of each sample group. See Table 17.15 for recovery criteria for each method. If the analyte concentration is high enough, the serial dilution must agree within 10% of the original sample. If the serial dilution is outside the 10% limit, a chemical or physical interference effect should be suspected.

4.5.2 Spike Addition

A post-spike is performed when required per method being analyzed. A representative sample within the sample group (client job) is spiked. Generally, the spike is performed on the same sample as the one on which the serial dilution is performed, unless there is limited volume. Spiking a sample consists of adding a specified amount of four separate spike solutions to the unknown sample. Each spike solution contains various elements of interest. See Table 17.15 for recovery criteria for each analyte.

4.5.3 The four spike solutions for Non-CLP samples are:

- **Spike 1 (Custom Inorganic Standard) Made by Ultra Scientific**
  This ULTRAgrade ™ standard was gravimetrically prepared and the true value listed is the concentration calculated from gravimetric and volumetric measurements performed during the preparation of the standard.

ANALYTE	TRUE VALUE
Antimony	40.0 µg/mL
Arsenic	40.0 µg/mL
Beryllium	40.0 µg/mL
Cadmium	40.0 µg/mL
Chromium	40.0 µg/mL
Cobalt	40.0 µg/mL
Copper	40.0 µg/mL
Lead	40.0 µg/mL
Manganese	40.0 µg/mL
Molybdenum	40.0 µg/mL
Nickel	40.0 µg/mL
Selenium	40.0 µg/mL
Thallium	40.0 µg/mL
Vanadium	40.0 µg/mL
Zinc	40.0 µg/mL
Titanium	40.0 µg/mL
Calcium	2000.0 µg/mL
Iron	2000.0 µg/mL
Magnesium	2000.0 µg/mL

Matrix: 5% HNO_3 in water

All weights are traceable to NIST traceable weights CAT#ICUS-1370.

NOTE: These concentrations might be slightly different between different lots. Current concentrations may be found in the binder of the Certificates of Analysis and in Element. This NOTE is also applicable to Spike 2, Spike 3 and Spike 4.

- **Spike 2 (Custom Inorganic Standard) Made by Ultra Scientific**

  This ULTRAgrade ™ standard was gravimetrically prepared and the true value listed is the concentration calculated from gravimetric and volumetric measurements performed during the preparation of the standard.

ANALYTE	TRUE VALUE
Barium	40.0 µg/mL
Boron	40.0 µg/mL
Aluminum	2000.0 µg/mL
Potassium	2000.0 µg/mL
Sodium	2000.0 µg/mL

Matrix: 5% HNO_3 in water

All weights are traceable to NIST traceable weights CAT# ICUS-574

- **Spike 3**

ANALYTE	TRUE VALUE
Silver	10 µg/mL

See 7.10 for preparation.

- **Spike 4**

ANALYTE	TRUE VALUE
Tin	40 µg/mL

See 7.11 for preparation.

Table 17.6 lists the final concentration of each element spiked.

To prepare a spike, add 0.05 mL of Spike 1, Spike 2, Spike 3 and Spike 4 to 9.80 mL of sample. Mix thoroughly and analyze.

The three spike solutions for CLP samples are:

CLP-1 Made by ULTRA SCIENTIFIC

ANALYTE	TRUE VALUE (4.1)	TRUE VALUE (5.2)
Aluminum	2000 µg/mL	2000 µg/mL
Barium	2000 µg/mL	2000 µg/mL
Beryllium	50.0 µg/mL	50.0 µg/mL
Chromium	200.0 µg/mL	200.0 µg/mL
Cobalt	500.0 µg/mL	500.0 µg/mL
Copper	250.0 µg/mL	250.0 µg/mL
Iron	1000 µg/mL	1000 µg/mL
Manganese	500.0 µg/mL	500.0 µg/mL
Nickel	500.0 µg/mL	500.0 µg/mL
Silver	50.0 µg/mL	50.0 µg/mL
Vanadium	500.0 µg/mL	500.0 µg/mL
Zinc	500.0 µg/mL	500.0 µg/mL

CLP-2 Made by ULTRA SCIENTIFIC

ANALYTE	TRUE VALUE (4.0)	TRUE VALUE (5.0)
Antimony	500.0 µg/mL	100.0 µg/mL

CLP-3 Made by ULTRA SCIENTIFIC

ANALYTE	TRUE VALUE (4.0)	TRUE VALUE (5.0)
Arsenic	2000 µg/mL	40 µg/mL
Cadmium	50 µg/mL	50 µg/mL
Thallium	2000 µg/mL	50 µg/mL
Selenium	2000 µg/mL	10 µg/mL
Lead	500 µg/mL	20 µg/mL

Refer to sample preparation SOPs for the preparation of matrix spikes for CLP samples. The spike recovery criteria may be found in table 17.15

5.0 Safety

Many of the metallic elements analyzed for in this method are known to be hazardous to health. Care should be taken in the handling and disposing of all standards and samples. See section 14.0 for procedures on the disposal of standard and sample waste.

5.1 The matrix of all ICP standards and samples is 6% HNO3, 5% HCl by volume. Gloves must be used when handling all standards and samples. Safety glasses must be worn at all times. Extra care should be taken when dispensing concentrated acids. Concentrated acids should be dispensed only in the fume hood.

5.2 The plasma emits strong UV light and is harmful to vision. **AVOID LOOKING DIRECTLY AT THE PLASMA.**

5.3 The RF generator produces strong radio frequency waves, most of which are unshielded. People with pacemakers should not go near the instrument while in operation.

Employees must abide by the policies and procedures in the Corporate Environmental Health and Safety Manual (CW-E-M-001), Radiation Safety Manual and this document. This procedure may involve hazardous material, operations and equipment. This SOP does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of the method to follow appropriate safety, waste disposal and health practices under the assumption that all samples and reagents are potentially hazardous. Safety glasses, gloves, lab coats and closed-toe, nonabsorbent shoes are a minimum.

5.4 Primary Materials Used

The following is a list of the materials used in this method, which have a serious or significant hazard rating. **Note: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the MSDS for each of the materials listed in the table.** A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the MSDS for each material before using it for the first time or when there are major changes to the MSDS.

Material (1)	Hazards	Exposure Limit (2)	Signs and symptoms of exposure
Nitric Acid	Corrosive Oxidizer Poison	2 ppm-TWA 4 ppm-STEL	Nitric acid is extremely hazardous; it is corrosive, reactive, an oxidizer, and a poison. Inhalation of vapors can cause breathing difficulties and lead to pneumonia and pulmonary edema, which may be fatal. Other symptoms may include coughing, choking, and irritation of the nose, throat, and respiratory tract. Can cause redness, pain, and severe skin burns. Concentrated solutions cause deep ulcers and stain skin a yellow or yellow-brown color. Vapors are irritating and may cause damage to the eyes. Contact may cause severe burns and permanent eye damage.
Hydrochloric Acid	Corrosive Poison	5 ppm-Ceiling	Inhalation of vapors can cause coughing, choking, inflammation of the nose, throat, and upper respiratory tract, and in severe cases, pulmonary edema, circulatory failure, and death. Can cause redness, pain, and severe skin burns. Vapors are irritating and may cause damage to the eyes. Contact may cause severe burns and permanent eye damage.
1 – Always add acid to water to prevent violent reactions.			
2 – Exposure limit refers to the OSHA regulatory exposure limit.			

6.0 Equipment and Supplies

6.1 Instrumentation

Thermo Jarrell Ash ICAP 61E Trace Analyzer is equipped with an autosampler, computer, printer, and source of argon. There are two Trace Analyzers at TestAmerica Buffalo. They are designated as Trace #1 and Trace #2.

- Spare parts for the Trace:
  - nebulizers
  - torches
  - spray chambers
  - platens

- Red/Red/Red pump tubing

- Orange/Green/Orange pump tubing

- Orange/Orange/Orange pump tubing

- Internal Standard tubing mixing kit

- o Autosampler sample probes (TJA #4097-30)

6.2 Supplies

- Volumetric flasks in various sizes from 50 mL to 1000 mL. These are used for standard preparation and sample dilution.

- Eppendorfs in various sizes. These are used for standard and sample preparation. The Eppendorfs and re-pipettors are verified using an analytical balance on a daily basis. They are calibrated on a quarterly basis along with the re-pipettors. An electronic spreadsheet contains the calibration results. At least one Eppendorf in each of the following ranges are used:

- 10 µL → 100 µL
- 50 µL → 200 µL
- 50 µL → 250 µL
- 100 µL → 1000 µL
- 500 µL → 2500 µL
- 2000 µL → 10000 µL

- Disposable polypropylene pipette tips for the Eppendorfs in various sizes.

- Disposable 17x100 mm polypropylene culture tubes used in the autosampler as the sample containers.

- 28 mL Nalgene brand disposable sample vials used to hold standards and quality control samples in the autosampler.
- 500 mL graduated cylinder.

- Parafilm
- Repipettors for acids and dilutions.

7.0 <u>Reagents and Standards</u>

All standards and samples are prepared such that the matrices are matched.

7.1 All standards and samples are prepared using 18 $M\Omega cm^{-1}$ ELGA water. The metals lab has an ELGA water system attached to a deionized water system. The ELGA water is monitored daily by the Wet Chemistry department and maintenance is performed as needed.

7.2 All standards are prepared with volumetric flasks, and calibrated Eppendorfs.

7.3 All standards and samples are prepared with Trace Metals Grade Nitric and Hydrochloric Acids.

7.4 All the standards and samples are prepared in the same matrix containing 6%HNO_3 and 5% HCl by volume.

7.5 Standards are prepared as needed, every 3 to 7 days for Calibration Standards.

Table 17.7 lists all the reagents and stock solutions that are purchased as starting materials. All stock solutions are certified and the certifications are kept for a record. All stock solutions are logged into an incoming logbook that is stored in the lab.

The multi-element calibration standards and other solutions required (except the quality control sample used for ICVs and CCVs) are prepared from stock solutions purchased from ULTRA SCIENTIFIC. The quality control sample used for ICVs and CCVs are prepared from stock solutions purchased from HIGH PURITY. The use of two vendors ensures a second source verification of standards. The ionization buffer $LiNO_3$ is purchased as a solid from MALLINCKRODT.

7.6 There are two types of solutions that are prepared from the purchased stock standards. They are prepared stock solutions and the working standards. Prepared stock solutions are used as intermediate standards for preparing the working standards. Prepared stock solutions are recorded in Element. They expire in six months or when the original starting stock standards expire, whichever is first. Prepared stock solutions are labeled with their name, the preparation date, the expiration date, and the initials of the analyst preparing the solution.

The working standards are prepared from the purchased stock standards and the prepared stock solutions. The working standards are also recorded in Element.

Solutions prepared by the analyst are recorded in the LIMS. The following information is recorded:

- Name or concentration of the solution
- Date prepared
- Initials of analyst preparing the solution
- The manufacturer of the starting stock solution
- The lot number of the starting stock solution
- The name or concentration of the starting stock solution
- The volume of the starting stock solution used
- The final volume of the solution being prepared
- The source of the HNO3
    - The source of the HCl

7.7 Blank solutions contain 6% HNO_3 and 5% HCl in ELGA water. The blank solution is used for the following:

- Calibration blank
- ICB
- CCB’s
- Sample dilutions

7.8 The Blank Solution is prepared by adding 1200 mL concentrated HNO_3 and 1000 mL concentrated HCl to a 20 liter plastic carboy half filled with ELGA water. Bring up to volume with ELGA water. This procedure may be scaled up or down. Use a 500 mL graduated cylinder to add the acids. Be extremely careful when handling conc. acids in these amounts (work in the fume hood wearing lab coat, gloves and safety glasses).

7.8.1 The instrument rinse is prepared in a 20-liter plastic carboy. The rinse blank is prepared by adding 1200 mL of concentrated HNO_3 and 1000 ml of concentrated HCl to the carboy half filled with ELGA water. Fill the carboy to the 20-liter mark with ELGA water.

7.9 The following stock solutions are prepared:

- Spike 3, containing 10 μg/mL Ag and Spike 4, containing 40 μg/mL Sn
- 200 g/L $LiNO_3$ solution

Begin with ULTRA SCIENTIFIC stock standards.

7.10 Spike 3 is prepared by adding 1.0 mL of 1,000 μg/mL Ag to a 100 mL volumetric flask half filled with 2% HNO_3 Blank Solution. Bring up the final volume with 2% HNO_3 Blank Solution. This spike is used for the post-spike.

7.11 Spike 4 is prepared by adding 4.0 ml of 1,000 ug/ml Sn to a 100 ml volumetric flask filled with blank solution. Bring up the final volume with Blank solution. This spike is used for the post-spike.

7.12 The 200.0 g/L $LiNO_3$ (0.1%) solution is prepared by weighing out 200.0 g of $LiNO_3$ and dissolving in a 1000 mL volumetric flask half filled with Blank Solution. Dilute to one liter with Blank Solution. This is the buffer for the internal standard.

7.13 The following calibration standards and solutions are to be prepared in the laboratory:

- CAL2
- CAL3
- CAL4
- IFA
- IFB
- CCV
- Internal Standard
- LCV
- NAKCAMG100
- NAKCAMG400

These standards and solutions are prepared from ULTRA SCIENTIFIC stock standards and prepared stock solutions.

7.13.1 CAL2 is prepared by adding 20 mL of CAL4 (Section 7.13.3) to a 200 mL volumetric flask half filled with Blank Solution. Bring up to final volume with Blank Solution. See Table 17.8 for concentrations of elements in CAL2.

7.13.2 CAL3 is prepared by adding 100 mL of CAL4 (Section 7.13.3) a 200 mL volumetric flask half filled with Blank Solution. Bring up to final volume with Blank Solution. See Table 17.8 for concentrations of elements in CAL3.

7.13.3 CAL4 is prepared by adding 5.0 mL ICUS-575, 5.0 mL ICUS-576, 0.5 mL 1000 μg/mL Sn and 0.5 mL 1000 μg/mL Ag to a 500 mL volumetric flask half filled with Blank Solution. Bring up to final volume with Blank Solution. See Table 17.8 for concentrations of elements in CAL4.

7.13.4 The IFA is prepared by adding 50.0 mL of IFA stock solution (ICM-441) to a 500 mL volumetric flask half filled with Blank Solution. Bring up to final volume with Blank Solution. See Table 17.9 for concentrations of elements in the IFA.

7.13.5 The IFB is prepared by adding 50.0 mL of stock solution (ICUS-919) to a 500 mL volumetric flask half filled with Blank Solution. Bring up to final volume with Blank Solution. See Table 17.9 for concentrations of elements in the IFB.

7.13.6 The NAKCAMG100 is prepared by adding 1.0 mL of each 10,000ppm stock solutions Na (ICP-111-5), K (ICP-119-5), Ca (ICP-120-5), Mg (ICP-112) to a 100 mL volumetric flask half filled with Blank Solution. See Table 17.9 for concentrations of elements in the NAKCAMG100.

7.13.7 The NAKCAMG400 is prepared by adding 4.0 mL of each 10,000ppm stock solutions Na (ICP-111-5), K (ICP-119-5), Ca (ICP-120-5), Mg (ICP-112) to a 100 mL volumetric flask half filled with Blank Solution. See Table 17.9 for concentrations of elements in the NAKCAMG400.

7.14 The following standards are prepared from HIGH PURITY

7.14.1 A quality control sample (called the CCV) is prepared from HIGH PURITY stock standards. See Table 17.10 for true values for the CCV. The CCV is prepared by adding 5.0 mL CAL STD. #2 –R Solution A, 5.0 mL CAL STD.#2-R Solution B, 0.5 mL of 1000 μg/mL Ag and 0.5 mL of 1000 μg/mL Sn to a 1000 mL volumetric flask half filled with Blank Solution. Bring up to volume with Blank Solution. Final concentrations can be found in Table 17.10.

7.14.2 The Initial Calibration Verification (ICV) is prepared using the same stock as the CCV. It is prepared by adding 75.0ml of the CCV to a 100ml volumetric flask and bringing it up to volume with Blank Solution. Final concentrations can be found in Table 17.10.

7.14.3 Yttrium – 5 mg/l: The internal standard/profile is prepared by adding 0.5 mL of 10,000 μg/mL Y stock solution to a 1000 mL volumetric flask half filled with Blank Solution. Add 50.0 mL of the 200 g/L $LiNO_3$ solution. Bring up to final volume with Blank Solution.

7.14.4 Low Level Verification Standard:

The LCV is prepared by adding 50 ml ICUS-1932 to a 500 mL volumetric flask half filled with Blank Solution. Bring up to final volume with Blank Solution.

8.0 Sample Collection, Preservation, Shipment and Storage

8.1 The maximum holding time for metals samples is 180 days from sample collection. Aqueous samples are preserved with nitric acid to a pH<2. Soil samples do not require additional preservation.

8.2 Soil and total water samples are prepared by a digestion procedure in the digestion lab. The digestates are brought to the instrumental lab by the Digestion analyst. The digestates are stored on a shelf in the instrumental lab. When analysis on the digestates is complete, the digestates are placed in a main sample storage area. The main storage area is located near the digestion lab. The main storage area is used to store the original total samples, digestates, and soluble samples. The main storage area is kept locked when unattended. The digestates are kept for 6 months before they are finally disposed of. For CLP work the digestates are stored for 365 days after delivery of the data package. For all CLP samples, samples must be refrigerated at 4 degrees C from the time of collection until digestion. CLP samples may be disposed of after 60 days in a manner that complies with all applicable regulations.

8.3 **Controlled Access Storage**: CLP samples require controlled access storage with strict Chain-of-Custody procedures. Digestates for these samples are obtained from and returned to the cooler custodian. The custodian maintains both the original samples and the digestates in the locked controlled access storage cooler.

8.3.1 The original samples are kept for 60 days following delivery of the final report package.

8.3.2 Digestates are maintained for 365 days for CLP samples;

8.4 Most total and dissolved samples have already been preserved by sample control when they were received or in the field when the samples were taken. Preservation is required by the laboratory analyst in cases that samples have not been filtered and preserved. A comment, listing lot numbers of the acid and filter used, is placed in the particular job affected

Sample container, preservation techniques and holding times may vary and are dependent on sample matrix, method of choice, regulatory compliance, and/or specific contract or client requests. Listed below are the holding times and the references that include preservation requirements.

Matrix	Sample Container	Min. Sample Size	Preservation	Holding Time[1]	Reference
Waters	HDPE	50 mLs	HNO_3, pH < 2; Cool 4 ± 2°C	180 Days	40 CFR Part 136.3
Soils	Glass	3 grams	Cool 4 ± 2°C	180 Days	N/A

[1] Inclusive of digestion and analysis.

9.0 Quality Control

Overview: This section provides the guidelines of the quality control that are used to determine if data are useable or not. Depending on the clients' requests and each specific protocol, some QC samples may not be prepared and/or analyzed to each job. Any observed deviations must be documented for future references. If the analyst cannot make a decision about the usability of data, the supervisor must be consulted and the resolution must be documented. If data are unusable, the samples must be re-digested and/or re-analyzed depending on the situation. For details on how an actual analytical run is laid out see Section 10.0 - Procedures. For details on any of the calculations which are required in this section see Section 11.0 - Calculations.

9.1 Standards - To insure quality data, all working standards are prepared from high quality certified stock standards. All prepared standards are logged into the LIMS to insure traceability. Stock solutions are purchased as often as necessary to insure a fresh source.

9.2 Instrument Calibration - The instrument is calibrated daily at the beginning of each analytical run. All relevant information is printed for reference. A blank and three levels of standards are used to calibrate each element. A linear plot of each element is produced. The correlation coefficient (which is printed right after the calibration standards have been analyzed) for each element must be 0.995 or greater. If the correlation is less than 0.995 for a particular element, then the data for that element may not be used from that particular analytical run.

9.3 ICV - The ICV is prepared from a separate source other than the calibration standards. Table 17.15 for ICV control limit criteria. If the ICV is outside the control limits for an element, then the instrument must be recalibrated or that element cannot be used from that analytical run. The measured values must be within +/- 10% of the true value for CLP and method 6010B. The measured values must be within +/- 5% of the true value for method 200.7. See Table 17.10 for the true values of the ICV.

9.4 CCV's - The CCV is prepared from a separate source other than the calibration standards. It is analyzed after every ten samples and at the end of the analytical run. See Table 17.15 for CCV control limit criteria. If the CCV is outside the control limits for an element, the ten samples before and after that CCV should be reanalyzed for that element. See Table 17.10 for the true values of the CCV.

9.5 ICB and CCB's - After analyzing the ICV, analyze an ICB. After analyzing each CCV, analyze a CCB. See Table 17.15 for blank control criteria.

9.6 IFA – See Table 17.15 for recovery criteria for the IFA standard. If the IFA is outside the control limit for an element, that element cannot be used from that analytical run. See Table 17.9 for the true values of the IFA.

9.7 IFB -- After analyzing the IFA, analyze an IFB. See Table 17.15 for recovery criteria. If the IFB is outside the control limits for an element, that element cannot be used from that analytical run. See Table 17.9 for the true values of the IFB.

9.8 Method Blank (BLK) – For each batch of digestions, one method blank is digested for every 20 samples. Table 17.15 summarizes method blank compliance criteria.

9.9 BS - For each batch of digestions of water samples, a BS (Blank Spike) is prepared. Refer to Table 17.15 for compliance criteria. If the BS for an element is outside the control limits, then all the samples for that element must be re-digested. See Table 17.6 for the concentrations of each element.

9.10 SRM - For each batch of digestions of soil samples, a SRM (Standard Reference Material) is prepared. The certified values are different from lot to lot, and the certified values can be found in the binder of the Certificate of Analysis and in Element. The acceptance limits are provided by the supplier. If the SRM for an element is outside the control limits, all the samples for that element must be re-digested.

9.11 Matrix Duplicate (MD) - For CLP + per client request, one matrix duplicate is performed per digestion batch. See table 17.15 for duplicate compliance criteria. If the RPD is outside the control limits for an element, the data should be reviewed to determine cause. If lab error suspected, reanalyze or re-digest. Generally MD is performed only for CLP digestions.

9.12 Matrix Spike (MS) and Matrix Spike Duplicate (MSD) - For each batch of samples, two matrix spikes are performed (one is MS and the other is MSD). See Table 17.6 for the concentrations of the matrix spikes for each element. See table 17.15 for criteria for spike recovery and precision. If the RPD is outside the control limits for an element, the data should be reviewed to determine cause. If lab error suspected, reanalyze or re-digest. If the recovery for an element is outside the control limits, matrix effect is suspected for digestion and/or the determination. Generally MSD is performed for SW-8463 and CFR protocols.

9.13 Post Spike - A post digestion spike is performed based on client requirements. It is performed on the base sample that has an MS associated with it. The spike recovery must agree within the limits specified in Table 17.15. If the post spike for an element is outside the control limits, the matrix effect is suspected in the ICP determination.

9.14 Serial Dilution (SRD) - A serial dilution is performed on the base sample in the batch of 20 that has a matrix spike. If the serial dilution is outside the control limit, the matrix effect is suspected in the ICP determination. The dilution is a 1:5 (one part of the sample to four parts of the blank solution).

9.15 IEC, IDL, AND LINEAR RANGE DETERMINATION

9.16.1 IEC's - The IEC's (interelement correction factors) are determined by first analyzing the following solutions:

- 100 μg/mL Cr
- 100 μg/mL Mn
- 100 μg/mL V
- 1000 μg/mL Fe
- 1000 μg/mL Al
- 1000 μg/mL Ca
- 100 μg/mL Tl
- 100 μg/mL Ti
- 100 μg/mL Cu

100 μg/mL Sn
100 μg/mL Be
100 μg/mL Ba
100 μg/mL Ni
100 μg/mL Cd
1000 μg/mL Mg
100 μg/mL Co
100 μg/mL Mo

Each of the above (except Sn, which is 1000 ug/ml) solutions is prepared from individual 10,000 ug/ml (Ultra Scientific) stock standards at the above concentrations.

Next, check to see whether there are any false positive or negative readings for the other elements in each of the solutions. If there are any, then an IEC calculation is necessary.

To calculate the IEC factor, divide the false reading for an element by the actual reading of the interfering element.

Sample Calculation:
The following results are obtained after running a 200.0 μg/mL Fe solution:

Fe = 208.0 μg/mL
Cd = 1.21 μg/mL

The IEC factor for Cadmium would be:

$$\frac{1.21}{208.0} = 0.00582$$

9.15.2 IDL's – For non-CLP protocols, the IDL is determined by analyzing a blank solution 7 times. Calculate the standard deviation of the 7 readings for each element. The IDL is 3 times the standard deviation. For CLP protocol, the IDL is determined by multiplying 3, the average of the standard deviations obtained on three nonconsecutive days from the analysis of a standard solution at a concentration 3x-5x the estimated IDL's, with seven consecutive measurements per day.

9.15.3 Linear Ranges - The linear range is the highest standard the instrument can read which is ±5% of the known value. Analyze a series of standards for each element. The highest that is within ±5% establishes the linear range.

9.16 The internal standard counts are monitored for every analysis. The internal standard counts must fall between 50 and 150 percent of the counts of the internal standard in the initial calibration blank. If the internal standard fails to fall between 50 and 150 percent of the initial blank the data from that particular sample may not be used from that analytical run. Recalibrate and reanalyze the sample.

9.17 Sample QC - Refer to Table 17.15 for acceptance criteria for QC measurements

9.18 Instrument QC Refer to Table 17.15 for acceptance criteria for QC measurements

9.19 Contingencies for Handling Out-of –Control or Unacceptable Data

9.19.1 Contingencies for unacceptable data will have to be evaluated on a client-by -client or even by a sample-by-sample basis by the supervisor, the lab manager or the QA manager. Corrective action will be prescribed accordingly.

9.19.2 In the event acceptable data can not be obtained, a Job Exception Form must be filed with the project manager and the client notified.

9.19.3 If calibration fails, i.e., correlation coefficient is lower than 0.995, ICV and/or ICB are out of control limit, IFA and/or IFB are not recovered quantitatively, the analysis procedure must be terminated, the problems must be solved, and re-calibration must be started over.

9.19.4 If CCV and/or CCB fail, affected analytes in the 10 samples before and after that CCV/CCB pair must be reanalyzed.

9.19.5 If the BS or SRM do not meet criteria, the whole digestion batch must be reanalyzed. If the reanalysis still fails, that whole digestion batch must be re-prepared.

9.18.6 If the Method Blank fails for an analyte, but samples do not contain that analyte higher than the reporting limit or samples contain that analyte higher than 10x the Method Blank, the data is usable and reportable. Otherwise, the digestion batch must be re-prepared for that analyte.

9.18.7 If RPD for MSD or MD is out of control limits, the data should be reviewed to determine cause. If redigestion and reanalysis are still out of limits, the sample might be inhomogeneous and the data should be reported with qualification. Refer to table 17.15 for RPD criteria.

9.18.8 If Post Spike or Serial Dilution are outside of control limits, matrix effects in determination are suspected.

9.18.9 If BS, Post Spike and Serial Dilution are within QC limits but MS fail, matrix interference can be assumed and corrective action is not required.

9.18.10 For CLP if the percent recovery of the LCV falls outside the control limits of 70-130% (50-150%for Sb, Pb, Tl), the LCV must be re-analyzed for the outlying analytes. 9.20.11

9.18.11If, the internal standard counts for any analysis fail to fall between 50 and 150 percent of the counts of the internal standard in the initial calibration blank, recalibrate and reanalyze the affected sample/samples.

10.0 Procedure

The matrix of all standards and samples for ICP are acidic. Purple nitrile gloves must be worn when handling all standards and samples. Safety glass must be worn at all times in the laboratory. Extra care will be taken when dispensing concentrated acids and are to be dispensed only in a fume hood.

10.1 Sample Preparation

10.1.1 All samples are checked for the proper preservation at time of sample receipt in the sample receiving area. If the samples were not preserved, they are acidified and held for 18 hours. A sticker is affixed to the sample bottles. The pH is rechecked prior to digestion/analysis following the 18 hour waiting period.

10.1.2 Refer to the sample digestion sop's for details: BF-ME-002, BF-ME-003, BF-ME-005, BF-ME-007, and BF-ME-008.

10.2 Calibration

10.2.1 The daily standardization of the 61E Trace analyzer is done automatically at the beginning of each run and approximately every 60 samples. The calibration standards are programmed into the autosampler table. The programming procedure and the standards used are outlined in section 10.

10.2.2 Calibration Curvefit is used to calibrate the Na, K, Ca, and Mg wavelengths. This procedure uses an extended calibration with more standards to produce a more linear and precise response over a greater range. The calibration is then fitted to a curve, which can be resloped during an instrument standardization using the low and high standards of the daily calibration. A Calibration Curvefit is preformed bi-annually or if significant instrument changes occur. The procedures below explain the steps to run a calibration curvefit.

10.2.3 Calibration Curvefit Sample Table Setup and Modification

The following procedure is used to select the elements to be standardized and the concentrations of the standards used during a curvefit, or to modify an existing standards table.

- Starting from the main menu, go to “Development”.
- Go to “Calibration-Standards”.
- Type table name to be create or modified.
- To add a standard press <F1>.
- Add elements to the standard by pressing <F1>.
- To add an element on the periodic table highlight the element to be added and press <F1>. To remove an existing element press <F2>.
- Press <F9> when done.
- Type in the concentration of the each element in the standard.

- Press <F9> when done.
- To modify an existing standard in the table, highlight it, and press <F1>. This will allow you to change the elements and concentrations in the standard by following the instructions above.
- To delete a standard press <F5>.

NOTE: The blank and highest standard of the daily curve must be represented exactly in the curvfit table including the same concentrations. If this is not done the fitted curve will not automatically be resloped during daily calibration.

10.2.4 Analysis of the Calibration Curvefit Standards

This procedure is used when analyzing a curve to be fitted.

- Starting from the main menu go to “Development”.
- Go to “Calibration –Analysis”
- Enter the standards table name and designate a calibration data file using a one digit run number, three letter month abbreviation, two number day date, and the last two digits of the year (example: 1MAR0705).
- Press <F9 Done to continue.
- To analyze a standard, begin by manually aspirating the standard, then press<F1>Run Standard, and skip comments section by pressing <F1>Run.
- When analysis is complete press <F9>Done.
- Repeat above two steps for each standard in the table.
- When all standards have been analyzed press <F9>Done.

10.2.5 Fitting the Curve

These steps are used to fit the analyzed standards or refit an existing data set.

- Starting at the main menu, go to “Development”.
- Go to “Calibration-Curvefit”.
- Select or enter the standards table and data file used during analysis.
- Press <F9>Done to continue.
- Select the element/line to be fitted.
- Press <F1>Fit Element.
- Press <F1>Fit Element.
- Under fit type select “Full Fit”.
- Under Weight select “Concentration Zero Factor “100.000.
- Press <F9>Done Calc.
- Check that the Correlation Coefficient > 0.995.
- Press <F9>Done.
- Repeat for the rest of the elements to be fitted.

10.2.6 Integrating the Calibration Curvefit into the Instrument Method

The following procedure is used to integrate the Calibration Curvefit into the chosen method. This will allow the Calibration Curvfit to be resloped with the daily curve using the blank and high standard.

- Starting on the main menu, go to “Development”.

- Go to “Methods”.
- Type the method name.
- Press <F5>Element Info
- Highlight the fitted element. The drop down menu below the line marked “Stdzn Method” should read “2-pt Calib” and the high and low standards names should match both the daily curves high and low standards as well as the two matching standards in the curve fit table. If these are correct check the remaining curvefit elements.
- If any of the criteria are incorrect press <PgDwn> twice.
- Highlight the incorrect item and type correction or press spacebar for drop down.
- Press <F9>Done to save.
- Repeat for the remaining curvefit elements.
- Press <F9>Done/Keep to save changes.
- Next from “Modifying Existing Method” page press <F3>Method Info.
- At the bottom left of the page enter the “Calibration Data File” and the “Calibration Stds” table name.
- Press <F9>Done/Keep.

These steps should have completed Calibration Curvefit standardization and integration into the method. Run a daily curve to confirm.

10.3 Sample Analysis

The following is a daily checklist for the operation of 61E Trace analyzer. This is only a summary of the basic steps. Each step is followed by a reference for further information.

1. Empty the main drain waste and the autosampler drain waste, if necessary. See Section 10.3.6.1
2. Fill the autosampler rinse, if necessary. See Section 10.3.6.2
3. Refill the internal standard, if necessary. See Section 10.3.6.3
4. Inspect the pump tubing daily. Either change tubing or change side to side. See section 10.3.6.7
5. Change the torch, if necessary. The torch should be changed when necessary. See Section 10.3.6.4. The procedure for cleaning the torch is described in Section 10.3.6.5
6. Check the argon pressure, if necessary. See Section 10.3.6.6
7. Fill the Argon Saturator if necessary. See Section 10.3.6.12
8. Ignite the plasma. See Section 10.3.7.1
9. Start the peristaltic pump. See Section 10.3.7.1
10. Prepare the standards and QC samples. These must be re-poured daily. See Section 7.0
11. Type the run into an autosampler table and print the table. See section 10.4
12. Change the data file name for each run. See Section 10.4.4
13. Place the standards and QC samples in the autosampler. See Section 10.4.7
14. Run a profile before each run. See Section 10.4.6
15. Prepare the samples and place them into the autosampler. See Section 10.4.7
16. Start the analysis. See Section 10.5
17. When the analysis is complete, print the data in a condensed format. See Section 10.6

18. Turn off the plasma unless you want to perform another analysis. See Section 10.7
19. Empty the samples and standards into a waste receptacle.
20. Perform validation of the data. See Section 10.8

Instrument Layout and Maintenance

This section contains information on the layout of the instrument and any maintenance that the analyst might perform. Any maintenance beyond what is stated will be performed by Thermo Jarrell Ash service personnel.

10.3.1 There are two 61E Trace Analyzers at TestAmerica Buffalo. They are both set up in a similar manner.

10.3.2 See Section 17.11 for a diagram of the main instrument layout.

10.3.4 See Section 17.12 for a diagram of the tubing layout.

10.3.5 See Section 17.13 for a diagram of the torch/spray chamber/nebulizer assembly.

10.3.5.1 See Section 17.14 for a diagram showing the layout of the autosampler. Rack #1 holds the 28 mL disposable polypropylene sample vials and are used for the calibration standards and quality control standards.

10.3.5.2 Racks #2, #3, #4, and #5 hold the disposable polypropylene culture tubes and are used for the samples.

10.3.6 The following is a list of maintenance topics and problems that need troubleshooting:

- Emptying the waste (See Section 10.3.6.1).

- Filling the autosampler rinse (See Section 10.3.6.2).

- Filling the Internal Standard (See Section 10.3.6.3).

- Filling Argon Saturator (See Section 10.3.6.12).

- Changing the torch (See Section 10.3.6.4).

- Cleaning the torch (See Section 10.3.6.5).

- Check the argon pressure (See Section 10.3.6.6).

- Changing the pump tubing (See Section 10.3.6.7).

- The cooling water level is low (See Section 10.3.6.8).
- The red warning light on the power supply comes on (See Section 10.3.6.9).
- Droplets form on the wall on the inside of the spray chamber (See Section 10.3.6.10).
- The "By Pass" light comes on (See Section 10.3.6.11).

10.3.6.1 Emptying the Waste

There is one waste container for the 61E Trace Analyzer. One waste line comes from the excess sample draining from the spray chamber (this is called main instrument drain). The other waste line comes from the autosampler rinse station. Both lines run into a central container and are acid wastes and must be disposed of properly. The analyst is responsible for disposal of the acid wastes. Make sure that the 5 gal. waste container is labeled for nitric acid (AN) waste.

The following steps are used when emptying the instrument waste container:

- Unscrew the instrument waste cap.
- Replace full carboy container with an empty one.
- Replace the cap being sure not to tangle the tubing.

CAUTION: Always wear gloves and safety glasses when handling wastes.

10.3.6.2 Filling the Autosampler Rinse

- Pull the tubing from the 20 liter rinse container.
- Refill the rinse container with reagent blank prepared
- Replace the tubing into the rinse container.
- Place the lid of the rinse container on to keep dust out.

10.3.6.3 Filling the Internal Standard

- Pull the tubing out of the internal standard flask.
- Fill the flask with the internal standard prepared according to Section 7.0.7.3.
- Place the tubing back into the internal standard flask.
- Place a piece of parafilm on the top of the flask to keep dust out.

10.3.6.4 **Changing the Torch** - Refer to the diagram in Section 17.13 to aid in changing the torch. Use the following steps:

- Turn the plasma off.
- Pull the spray chamber out.
- Remove the two screws holding the white cover in place.
- Remove the white cover.
- Pull out the gray collar. Be careful not to bump the quartz end.
- Remove the two argon lines from the gray collar by unscrewing the connectors.
- Remove the black O ring from the end of the gray collar.
- Tip the gray collar until the torch slides out.
- Place a clean torch in the instrument and follow steps 1 through 8 in reverse order.
- Ignite the plasma

10.3.6.5 **Cleaning the Torch** – Rinse the torch with a 1:1 HNO_3 solution (equal parts of HNO_3 acid and ELGA water). Rinse the torch with ELGA water and allow to dry.

Prepare the 1:1 HNO_3 solution (equal parts of HNO_3 acid and ELGA water) by adding 100 mL of ELGA water using a graduated cylinder to a 400 mL beaker. Carefully add 100 mL of conc. HNO_3 acid. Be very careful when working with concentrated acids in this quantity. Work in the fume hood and wear gloves, lab coat, and safety glasses.

10.3.6.6 **Checking the Argon Pressure**

- The argon regulator is installed on the wall next to the door. Check the pressure to make sure it is 80 psi.

10.3.6.7 **Changing the Pump Tubing**

There are three types of pump tubing used (use the diagram in Section 17.12 for reference):

- orange/orange/orange - used for the samples.
- orange/green/orange - used for the internal Standard
- red/red/red - used for the autosampler rinse and main instrument waste.

To replace the pump tubing, first pull the tubing from each end of the pump tubing. Insert the tubing into the ends of the new pump tubing.

10.3.6.8 **The Cooling Water Level is Low**

If the cooling system light comes on, the water level is low. Use the following steps to add water to the cooling system:

- Remove the cover from the top of the cooling system.
- Remove the cap inside.
- Add DI water.
- Replace the inside cap.
- Replace the top cover.

10.3.6.9 **The Red Warning light on the Power Supply Comes On**

This indicates that the filter on the back of the power supply is dirty. Use the following steps to clean the filter.

- Remove the filter from the power supply.
- Rinse the filter with water.
- Pat the filter dry with a paper towel.
- Allow the filter to dry.
- Replace the filter on the power supply.

10.3.6.10 **Droplets Form on the Wall on the Inside of the Spray Chamber**

Aspirate a solution of 1:1 Nitric acid (equal parts of HNO_3 acid and ELGA water). Do not aspirate for more than 10 seconds at a time. Prepare the 50% Nitric acid solution by adding 50 mL of conc. Nitric acid to ~50 mL of ELGA water.

WARNING! Conc. Nitric acid is extremely hazardous! Work in the fume hood and wear gloves, lab coat, and safety glasses. Pull the hood sash down to protect your face.

10.3.6.11 **The "By Pass" Light Comes On**

The "By Pass" light comes on if the instrument loses the vacuum due to a power outage or pump failure. If the pump fails, it needs to be fixed first. Use the following steps to regain the vacuum:

- Go to the back of the instrument and turn the switch labeled HV (high voltage) to the off position.
- Dial the Red needle on the vacuum gauge all the way to the right.
- Push the start button.
- When the black needle reads less than 20, dial the Red needle to 20.
- Turn the HV (high voltage) to on.
- Ignite the plasma.

10.3.6.12 **Filling the Argon Saturator**

If the water level is below “min” line, refilling is necessary with the following steps:

- Turn plasma off.
- Unscrew Argon Saturator cap.
- Fill with Elga Water to about ½ inch below the red “Max” line.
- Replace cap
- Restart plasma.

Instrument Start-Up

10.3.7 The instrument should always be left on. This is required to maintain the vacuum. If it ever becomes necessary to turn off the instrument, make sure that the high voltage (HV) switch on the back of the instrument is turned to off. If the instrument is not being used on a given day, it is only necessary to turn the computer, monitor, and printer off.

If the computer has been turned off, and if only the C:\ prompt comes up when it is turned on, then type STNRUN and press 〈Enter〉 to get into the software. Once in the software the plasma is ready to ignite.

10.3.7.1 The following steps are used to ignite the plasma and start the peristaltic pump.

- Starting from the main menu. Go to "Setup".
- Go to "Plasma Control Panel".
- Press 〈Enter〉.
- Press 〈F1〉 for plasma on.
- Press 〈F9〉 to continue.
- If the plasma does not ignite, then press 〈F1〉 and try again. Make sure there is sufficient argon first and that the valve on the argon tank is open.
- When the plasma ignites, press 〈Enter〉 for O.K.
- Press 〈ESC〉 to go back to the main menu.
- Go to "Operation".
- Press 〈Enter〉.
- Type in the name of the method you want to run.
- Press 〈Enter〉. This will start the peristaltic pump. The instrument is now warming up. Allow the instrument to warm up for 30 to 60 minutes.

10.4 Typing an Analytical Run

10.4.1 Each Non-CLP analytical run is typed in the following format:

		SEQ-CAL1
Calibration	⎧ ⎨ ⎩	**SEQ-CAL2** **SEQ-CAL3** **SEQ-CAL4**
		SEQ-ICV
		SEQ-ICB
		SEQ-LCV
		SEQ-IFA
		SEQ-IFB
		SEQ-CCV
		<u>SEQ-CCB</u>

↑
10 samples
↓
SEQ-CCV
SEQ-CCB
↑
10 samples
↓
SEQ-CCV
SEQ-CCB
↑
10 samples
↓
SEQ-CCV
SEQ-CCB
↑
10 samples
↓
SEQ-CCV
SEQ-CCB
↑
10 samples
↓
SEQ-CCV
SEQ-CCB
SEQ-LCV **Client specific.**
SEQ-IFA ↓
SEQ-IFB ↓
SEQ-CCV ↓
SEQ-CCB ↓

Run a CCV and CCB after every 10 samples and at the end of the analytical run. Run the LCV, IFA, and IFB at the beginning and end of the analytical run if Client specific.

NOTE: To be compliant with all protocols and clients' particular requests, extensive QC samples are routinely prepared and run. However, not all these QC samples are required for a particular protocol. For example, the ending LCV, IFA and IFB are not required by SW-864 and 40 CFR protocol. Therefore, a particular run may not include ending LCV, IFA and IFB if that procedure only involves standard SW-864 and 40 CFR protocols. This note is also applicable to CLP procedure.

10.4.2 Each CLP analytical batch is typed in the following format:

SEQ-CAL1
Calibration { **SEQ-CAL2**
SEQ-CAL3
SEQ-CAL4
SEQ-ICV
SEQ-ICB
SEQ-LCV
SEQ-IFA
SEQ-IFB
SEQ-CCV
SEQ-CCB
↑
10 samples
↓
SEQ-CCV
SEQ-CCB
↑
7 samples
SEQ-LCV

SEQ-IFA
SEQ-IFB
↓
SEQ-CCV
SEQ-CCB
↑
10 samples
↓
SEQ-CCV
SEQ-CCB
↑
7samples
SEQ-LCV
SEQ-IFA
SEQ-IFB
↓
SEQ-CCV
SEQ-CCB

10.4.3 Typing the Autosampler Table

Type the analytical run into the autosampler table according to the following steps:

- Starting from the main menu, go to "Exit".
- Go to "ASEDIT.EXE".
- Press ⟨Enter⟩.
- Type in the name of the autosampler table. Use one of the saved templates.
- Press ⟨Enter⟩.
- Press ⟨F1⟩ to edit set.
- Press ⟨F1⟩ to edit samples.
- Use the arrow keys to toggle down to the CCB.
- Holding the ⟨ALT⟩ key down, press:
  - ⟨F1⟩ - to add a sample,
  - ⟨F3⟩ - to add a QC,
  - ⟨F5⟩ - to add calibration standards, or
  - ⟨F7⟩ - to add a blank.
- Type in the ID's of the samples and QC as written in the analytical run log.
- Under the check table column type:
  - ICV - for each ICV,
  - CCV – for each CCV
  - FUCRI – for each LCV,
  - B - for each ICB and CCB,
  - ICSA - for each IFA
  - ICSAB - for each IFB
- After all the samples have been typed in, note the position of the last sample.
- Press ⟨F9⟩ for Done/Keep.
- Press ⟨F5⟩ for modify set.
- Type in the last sample position (from step 12).
- Press ⟨F9⟩ for Done/Keep.
- Press ⟨F9⟩, again, for Done/Keep.
- Press ⟨F2⟩ to print the autosampler table.
- Press ⟨F9⟩ for Done/Keep.

10.4.4 Entering the Data File Name

Each analytical run requires its own specific data file name. Use the following procedure to enter the data file name prior to the start of the analytical run:

- Starting from the main instrument menu, go to "Development".
- Go to "Methods".
- Press 〈Enter〉.
- Type in the method name of the method you are going to run.
- Press 〈Enter〉.
- Press 〈Enter〉 for method info.
- Use the arrow keys to toggle down to "Analysis Data File".
- Enter the new data file name.

Examples:

For Trace #1

1100500

Key: 1 = run# (1, 2, 3, etc.)
10 = month
05 = day
00= the last two digits of the year

For Trace #2

A100500

Key: A = run # (A,B,C, etc.)
10 = month
05 = day
00 = the last two digits of the year

- Press 〈F9〉 for Done/Keep.
- Press 〈F9〉, again, for Done/Keep.

10.4.5 Using the autosampler printout and the autosampler layout diagram (See 17.14), set-up the calibration standards and the quality control samples. Use the 28 mL disposable polypropylene sample vials to hold the calibration standards and the quality control samples.

10.4.6 Instrument Profile

Before each analytical run, a profile of the instrument must be performed. The profile is performed using the Internal Standard, which is a 5 PPM Yitrium solution according to the following steps:

- Starting from the main menu, go to "Operation".
- Go to "analysis".
- Press 〈Enter〉.
- Type in the name of the method you are going to run.
- Press 〈Enter〉.
- Press 〈F5〉 for profile.
- Press 〈F3〉 for automatic.

- Press 〈F1〉 to run scan.
- When the scan comes up, check the "Peak Position". The "Peak Position" should be between negative 0.05 and positive 0.05.
  If yes, then press 〈F9〉 for done/keep.
  If no, then press 〈F1〉 to calc. SS. Check the vernier position on the computer. This should be the same as the vernier position on the instrument. Press 〈Enter〉. Dial in the new vernier position.
- Press 〈F9〉 for Done/Keep.

10.4.7 Preparing Samples for the Autosampler

Using the autosampler table printout and the autosampler layout diagram (See Table 17.14, set-up the samples in the autosampler. Use the disposable polypropylene culture tubes. Pour the samples into the culture tubes and place in the autosampler.

For 'Total Metals' and 'Soluble Metals', the samples consist of the digestates received from the metals preparation department.

10.4.7.1 To prepare post spikes, add the following amounts of each spike solution to 9.80 mL of sample:

50 µL - Spike 1 (Section 4.1.2)
50 µL - Spike 2 (Section 4.1.2)
50 µL – Spike 3 (Section 4.1.2)
50 µL – Spike 4 (Section 4.1.2)

Mix each post spike thoroughly and place in autosampler.

10.4.7.2 To prepare the 1:5 serial dilution, add 2.0 mL of sample to 8.0 mL of calibration blank.

10.5 Starting an Analysis

The determination parameters, such as integration time (13 seconds), rinsing time (60 seconds), number of replicates (2), standard concentrations are already entered, and in very rare cases these parameters may be changed. Refer to the instrument operation manual for modifying these values when necessary.

Once the autosampler table has been prepared, the samples, standard and quality control samples have been placed in the autosampler, and the instrument has been profiled, you are ready to begin the analysis. Use the following steps to begin the analysis.

- From the main menu, go to "Operation".
- Go to "Analysis".
- Press 〈Enter〉.
- Enter the method name of the method you wish to run.
- Press 〈Enter〉.
- Press 〈F9〉 for autosampler.
- Type in the autosampler table name under "Sample Name:".

- Press 〈Enter〉.
- If you want to have the instrument shut off automatically then press 〈F7〉 until the "Terminating Action:" reads "shutdown".
- Press 〈F1〉 to start the analysis.

10.6 Printing Data in Condensed Format

The trace instrument will print data during an analysis. A simplified, condensed version of the data may be prepared after the analysis is completed according to the following procedure:

- Starting from the main instrument menu, go to "IMS".
- Go to "Report Writer".
- Press 〈Enter〉.
- Enter the file name of the file you wish to print according to the following steps:
  - Press 〈F6〉 for a list of files.
  - Press 〈F2〉 to deselect all files.
  - Enter the number preceding the file you wish to print.
  - Press 〈Enter〉.
  - Press 〈F9〉 for Done/Keep.
- Use the arrow keys to go to "Method name".
- Enter the name of the method used to generate the data file.
- Press 〈Enter〉.
- Using the arrow keys to toggle back and forth, enter the start date and the end date of the data file you wish to print. You don't need to enter anything for the times.
- Use the arrow keys to go to "Sample type".
- Press the space bar to bring up a list of options.
- Use the arrow keys to toggle up to "All Types".
- Press 〈Enter〉.
- Press 〈F9〉 to continue.
- Use the arrow keys to toggle down to "Show Internal Standards".
- Change the No to Yes by pressing the "y" key.
- Use the arrow keys to toggle down to "Report format".
- Press the space bar to bring up a list of options.
- Use the arrow keys to toggle down to "vertical".
- Press 〈Enter〉.
- Press 〈F9〉 for Done/Go to start printing the data file.
- After printing the data file, press 〈ESC〉 to return to the main menu.

10.7 Plasma Shutoff

To turn the plasma off, use the following steps:

- From the main menu, go to "Setup".
- Go to "Plasma Control Panel".
- Press 〈Enter〉.
- Press 〈F5〉.
- Press 〈Enter〉.
- After about one minute, press 〈ESC〉.

10.8 Validation of the Data

When the analytical run is complete, the data must be checked for compliance with the method. Using Section 9.0 - Quality Control - check all the quality control samples (ICV, ICB, CCV's, CCB's, IFA, IFB, CAL Standards, LCV, and digested blank and BS/SRM) for compliance. If a quality control sample falls outside the required limits for an element, then that element must be rerun on another analytical run.

Also check the spikes and serial dilution for any matrix effects that might require a diluted sample run.

11.0 Calculations / Data Reduction

Refer to Table 17.15 to determine if data are valid for each element. Any sample reading over the linear range must be diluted. Diluted samples must be run on required samples. Analyzing the sample and a series of spiked aliquots of the sample at different known concentrations performs an MSA.

11.1 The following calculations are illustrated:

- Relative Percent Difference (RPD) (See Section 11.1.1).
- Post spike calculation (See Section 11.1.2).
- Method of Standard Addition (MSA) calculation (See Section 11.1.3).

11.1.1 The formula for calculating the relative percent difference is:

$$\% RPD = \frac{D_1 - D_2}{(D_1 + D_2)/2} X\ 100$$

Where,

RPD = relative percent difference
D_1 = first sample value
D_2 = second sample value (replicate)

Sample calculation: A sample gave a reading of 2.51 μg/mL and the replicate reading was 2.39 μg/mL.

$$\%RPD = \frac{2(2.51-2.39)}{2.51+2.39} \text{X100}$$

$$RPD = 4.90\%.$$

11.1.2 The formula for calculating the post spike recovery is:

$$\% \, Recovery = \frac{S_2 - S_1}{SA} \, X \, 100$$

Where, S_2 = the post spiked sample reading
S_1 = the sample reading
SA = the spike added

Sample Calculations:
A sample gave a reading of 0.250 µg/mL. The sample was post spiked with 2.000 µg/mL and gave a reading of 2.289 µg/mL.

$$\% \, Recovery = \frac{2.289 - .250}{2.000} \, X \, 100$$

$$\% \, Recovery = \frac{2.039}{2.000} \, X \, 100$$

% Recovery =102.0%

11.1.3 The formula for calculating the simplest version of MSA (single-addition method) is:

Where,

$$C_x = \frac{S_A V_S C_S}{(S_B - S_A) V_x}$$

S_B = the concentration of the spiked sample
S_A = the concentration of the unspiked sample
V_S = volume of spike solution added.
C_S = concentration of spike solution
V_x = volume of sample before adding spike
C_x = the unknown sample concentration

Sample calculation:
A sample gave a reading of 0.792 µg/mL. 50 µL of a 200 µg/mL spike solution was added to 10.0 mL of the sample. The spiked sample reading was 1.512 µg/mL.

$$C_x = \frac{(0.792)(0.05)(200.0)}{1.512 - 0.792)(10.0)}$$

$$C_x = \frac{7.92}{7.20}$$

$$C_x = 1.10ppm$$

12.0 Method Performance

This SOP is applicable to digested sample matrices and soluble water samples.

12.1 Extensive quality control is used to insure compliance with method 6010B, 200.7 and CLP protocol.

12.2 Thorough documentation is employed to insure traceability of reagents and standards.

12.3 Approximate detection and reporting limits for Trace #1 and Trace #2 are found in Tables 17.2 and 17.3.

12.4 Samples that read above the instrument's linear range must be diluted.

12.5 Method Detection Limit Study (MDL)

The method detection limit (MDL) is the lowest concentration that can be detected for a given analytical method and sample matrix with 99% confidence that the analyte is present. The MDL is determined according to the laboratory's MDL procedure in the QA Manual. MDLs reflect a calculated (statistical) value determined under ideal laboratory conditions in a clean matrix, and may not be achievable in all environmental matrices. The laboratory maintains MDL studies for analyses performed; these are verified at least annually unless method requirements require a greater frequency.

12.6 Demonstration of Capabilities

Reference the corporate QA Manual. All employees analyzing the methods listed in the sop have documented Initial demonstration of capabilities, as well as demonstration of capabilities each year after. This documentation is forwarded to QA for approval and record keeping.

12.7 Training Requirements

The QA Manual or a Training SOP may be referenced for training requirements. If applicable, state required concentration of samples prepared for Precision and Accuracy study or alternate training procedure.

13.0 Pollution Control

It is TestAmerica's policy to evaluate each method and look for opportunities to minimize waste generated (i.e., examine recycling options, ordering chemicals based on quantity needed, preparation of reagents based on anticipated usage and reagent stability). Employees must abide by the policies in Section 13 of the Corporate Environmental Health and Safety Manual (CW-E-M-001) for “Waste Management and Pollution Prevention.”

14.0 Waste Management

Waste management practices are conducted consistent with all applicable rules and regulations. Excess reagents, samples and method process wastes are disposed of in an accepted manner. Waste description rules and land disposal restrictions are followed. Waste disposal procedures are incorporated by reference AWM-HAZ.MG-01. The following waste streams are produced when this method is carried out.

14.1 All acidic waste consisting of sample and rinse solution: Dispose of as HNO_3 waste in an “AN” waste container.

15.0 References / Cross-References

15.1 Method 6010B, Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW-846, 3rd Edition, Final Update III, Revision 2, December 1996.

15.2 ICAP 61E Trace Analyzer Operator's Manual.

15.3 ILM04.1, USEPA Contract Laboratory Program, Statement of Work for Inorganic Analysis and Classical Chemistry Parameters.

15.4 ILM05.2, USEPA Contract Laboratory Program, Statement of Work for Inorganic Analysis and Classical Chemistry Parameters.

15.5 ILM05.3, USEPA Contract Laboratory Program, Statement of Work for Inorganic Analysis and Classical Chemistry Parameters.

15.6 Method 200.7, “Determination of Metals and Trace Elements in Water and Wastes by Inductively Coupled Plasma-Atomic Emission Spectrometry”, Revision 3.3, 40CFR Part 136, Appendix C, April 1991. (Approved for CWA compliance testing)

15.7 Method 200.7, “Determination of Metals and Trace Elements in Water and Wastes by Inductively Couple Plasma-Atomic Emission Spectrometry”, Revision 4.4, US EPA / EMSL, May 1994. (Approved for SDWA compliance testing)

16.0 Method Modifications:

Item	Method xx	Modification
1	3005A	Adopted prep method for preparation of water samples for 6010B and 200.7. See SOP BF-ME-002 for modifications.

17.0 Attachments

17.1 Elements which are analyzed on ICP's
17.2 Approximate Water Detection Limits for the ICAP 61E Trace Analyzers
17.3 Approximate Soil Detection Limits for the ICAP 61E Trace Analyzers
17.4 Wavelengths and Background Points Used for Each Element on the ICAP 61E Trace Analyzer
17.5 Approximate Linear Dynamic Range of Each Element on the ICAP 61E Trace Analyzer
17.6 Concentration of each analyte for BS, SRM, Post-digestion Spike, Non-CLP matrix spike and CLP matrix spike.
17.7 Reagents and Stock Solution which are purchased as Starting Materials for Preparation of Trace Standards
17.8 Concentration of Calibration Standards
17.9 Values for IFA and IFB
17.10 Values for CCV and ICV
17.11 Main Instrument Layout
17.12 Tubing Layout
17.13 Torch/Spray Chamber/Nebulizer Assembly
17.14 Auto sampler Layout
17.15 Method Summary
17.16 CLP Contract Required Detection Limits (CRDLs)
17.17 Concentration of Each Element in the LCV Standard Solutions
17.18 Data Review Summary for Metals
17.19 Certificates of Analysis for Custom Blend Standards

18.0 Revision History

- Revision 2, Dated January 18, 2010
    - Removed 3.8 ICL-HCV-The highest calibration standard re-run directly after calibrating the instrument.
    - Section 4.5.1 Removed "such that the analyte in the diluted sample is at least a factor of 10 above the IDL"
    - Section 4.5.3 Added in the Note under the Table " Current concentrations may be found in the binder of the Certificates of Analysis and also in Element."
    - Section 6.1 Changed STL: Buffalo to TestAmerica Buffalo
    - Section 7.6 Removed standard logbook and added in Element
    - Remove 8.3 " Soluble samples are stored in the main storage area with the digestates and the original total samples. All samples taken from the storage area must be logged out in the sample custody logbook that is kept in the digestion lab. Samples are logged back in when complete. The main storage area is kept locked when unattended. The key to the storage area can be obtained from the sample control personnel and returned to them when finished."
    - Remove 9.3 "ICL-The ICL is the highest calibration standard that is analyzed after the instrument is calibrated."
    - Section 9.4 Removed "It is analyzed after the ICL."
    - Section 9.10 Changed LFB to BS. Changed Laboratory Fortified Blank to Blank Spike.

- Section 9.11 Changed LCS to SRM. Changed Laboratory Control Sample to Sample Reference Material.
- Section 9.21.5 Changed LFB and LCS to BS and SRM.
- Section 9.21.9 Changed LCS to BS.
- Section 10.3.1 Changed STL Buffalo to TestAmerica Buffalo
- Section 10.3.6.2 Changed “a piece of parafilm” with the lid.
- Section 10.4.1 Removed HCV
- Section 10.4.2 Removed HCV
- Section 16.0 Added method modification
- Removed Tables 17.19 and 17.20
- Table 17.4 Added nm next to wavelength
- Section 2.3 Removed AFCEE reference
- Section 10.4.1 removed AFCEE references
- Section 12.4 removed USACE reference
- Section 7.6 changed ‘standards logbook’ to LIMS
- Section 9.1 changed ‘stndards logbook’ to LIMS
- Section 10.3.6.5 reword the procedure to clean the torch.
- Section 17.7 removed ICUS-573 standard. No longer used

- Revision 1, Dated July 07, 2009
  - New LIMS nomenclature changes:
  - BLANK became CAL1
  - Std.1 became CAL2
  - Std.2 became CAL3
  - Std.3 became CAL4
  - Std.3 VER became HCV
  - CRI became LCV
  - ICSA became IFA
  - ICSAB became IFB
  - MBLK became BLK
  - LCS became SRM
  - LFB became BS
  - SD became MSD
  - 10.3.6.12 Filling Argon Saturator: “Lower neb. pressure” deleted, replaced with “Turn plasma off.” “Turn neb. pressure on” deleted, replaced with “Restart plasma.”
  - 10.4 Typing an Analytical Run: “SEQ-“ added before all calibration and QC standards, names updated.
  - 17.7 1,000 ug/ml Y replaced with 10,000 ug/ml Y under “From ULTRA SCIENTIFIC;” 10,000 ug/ml Y deleted from “From HIGH PURITY.”
  - 17.0 Example of a Data Review Summary Form for Metals replaced with an Example of a Bench Sheet for Metals.
  - Metals Department manager change, signature.

- Revision 0, Dated June 09, 2008
  - Integration for TestAmerica operations
  - Quality Manager change, signature

17.1 Elements Which are Analyzed on the ICAP 61E Trace Analyzer:

Aluminum	Al	Magnesium	Mg
Arsenic	As	Manganese	Mn
Antimony	Sb	Molybdenum	Mo
Barium	Ba	Nickel	Ni
Beryllium	Be	Sodium	Na
Boron	B	Potassium	K
Cadmium	Cd	Selenium	Se
Calcium	Ca	Silver	Ag
Chromium	Cr	Thallium	Tl
Cobalt	Co	Vanadium	V
Copper	Cu	Zinc	Zn
Iron	Fe	Tin	Sn
Lead	Pb	Titanium	Ti

17.2 Approximate Water Detection Limits for the ICAP 61E Trace Analyzers.

Element	Estimated IDL (mg/L)	Estimated MDL (mg/L)	Lab PQL (mg/L)
Al	0.020	0.025	0.2
Sb	0.004	0.005	0.02
As	0.003	0.005	0.01
Ba	0.0002	0.0002	0.002
Be	0.0001	0.0003	0.002
B	0.001	0.005	0.05
Cd	0.00032	0.0006	0.001
Ca	0.014	0.02	0.5
Cr	0.0008	0.001	0.004
Co	0.0008	0.001	0.004
Cu	0.001	0.002	0.01
Fe	0.02	0.030	0.05
Pb	0.002	0.004	0.006
Mg	0.015	0.02	0.2
Mo	0.001	0.002	0.01
Ni	0.00090	0.002	0.01
K	0.052	0.075	0.5
Se	0.003	0.005	0.015
Na	0.250	0.3	1.0
Ag	0.00075	0.0008	0.003
Tl	0.003	0.005	0.02
V	0.001	0.002	0.005
Zn	0.004	0.007	0.02
Sn	0.003	0.004	0.01
Ti	0.00035	0.0008	0.005
Mn	0.0005	0.0003	0.003

17.3 Approximate Soil Detection Limits for the ICAP 61E Trace Analyzers.

Element	Estimated IDL (mg/kg)	Estimated MDL (mg/kg)	Lab PQL (mg/kg)
Al	2.0	2.0	10.0
Sb	0.4	5	15.0
As	0.3	0.5	2.0
Ba	0.02	0.1	0.5
Be	0.01	0.03	0.2
B	0.1	0.5	2.0
Cd	0.03	0.06	0.2
Ca	1.0	2.0	10.0
Cr	0.08	0.1	0.5
Co	0.1	0.1	0.5
Cu	0.1	0.2	1.0
Fe	2.0	3.0	10.0
Pb	0.2	0.4	1.0
Mg	2.0	2.0	20.0
Mo	0.1	0.2	1.0
Ni	0.1	0.2	0.5
K	5.0	8.0	30.0
Se	0.3	0.5	4.0
Na	25	30	140.0
Ag	0.1	0.1	0.5
Tl	0.3	1.0	6.0
V	0.1	0.2	0.5
Zn	0.4	0.7	2.0
Sn	0.3	0.4	2.0
Ti	0.05	0.1	0.5
Mn	0.05	0.05	0.2

17.4 Wavelengths and Background Points Used for Each Element on the ICAP 61E Trace Analyzer.

		Background Points	
Element	Wavelength (nm)	Trace #1	Trace #2
Al	3082.15	+10	+10
Sb	2068.38	+10	+10
As	1890.42	-10	-10
Ba	4934.09	+10	+10
Be	3130.42	-10	+10
B	2946.78	+10	+10
Cd	2265.02	-10	-10
Ca	3179.33	-10	-10
Cr	2677.16	-10	+10
Co	2286.16	+10	-10
Cu	3247.53	+10	+10
Fe	2714.41	+10	+10
Pb	2203.53	+10/-10	+10/-10
Mg	2790.78	-10	+10
Mo	2020.30	NA	-10
Ni	2316.04	+10	+10
K	4047.35/7664.35	-28	-28
Se	1960.26	+10/-10	+10/-10
Na	3302.32	+10	-10
Ag	3280.68	-10	-10
Tl	1908.64	+10	-10
V	2924.02	+10	+10
Zn	3062.00	+10	+10
Sn	2899.89	-10	-10
Ti	3372.80	+10	-10
Mn	2576.10	-10	+10

17.5 Approximate Linear Dynamic Range of Each Element on the ICAP 61E Trace Analyzer.

Element	Trace #1 (mg/L)	Trace #2 (mg/L)
Al	500	500
Sb	25	25
As	50	50
Ba	10	10
Be	10	10
B	25	25
Cd	5	5
Ca	500	500
Cr	25	25
Co	5	5
Cu	20	20
Fe	200	200
Pb	10	25
Mg	500	500
Mo	NA	25
Ni	10	10
K	500	500
Se	10	50
Na	500	500
Ag	10	10
Tl	25	50
V	200	200
Zn	200	200
Sn	10	10
Ti	5	5
Mn	50	50

17.6 Concentration of each analyte for BS, SRM, Post-digestion Spike, Non-CLP matrix spike and CLP matrix spike:

Element	LFB, Post-digestion Spike and Non-CLP Matrix Spike (mg/L)	CLP Matrix Spike (4.1) (mg/L)	CLP Matrix Spike (5.2,3) (mg/L)	Soil Post-digestion Spike and Non-CLP Matrix Spikes (mg/kg)	Estimated Soil LCS (mg/kg) Changes with new lot	CLP Matrix Spike Soil (4.1) (mg/kg)	CLP Matrix Spike Soil (5.2,3) (mg/kg)
Al	10.0	2.00	2.00	100	6340		
Sb	0.20	0.500	0.100	20	34	20	20
As	0.20	2.00	0.040	20	192	8	8
Ba	0.20	2.00	2.00	20	417	400	400
Be	0.20	0.050	0.050	20	99.9	10	10
B	0.20			20	131		
Cd	0.20	0.050	0.050	20	125	10	10
Ca	10.0			100	3370		
Cr	0.20	0.200	0.200	20	133	40	40
Co	0.20	0.500	0.500	20	56.8	100	100
Cu	0.20	0.250	0.250	20	93.9	50	50
Fe	10	1.00	1.00	100	11600		
Pb	0.20	0.500	0.020	20	160	4	4
Mg	10.0			100	2000		
Mo	0.20			20	62.9		
Ni	0.20	0.500	0.500	20	174	100	100
K	10.0			100	1890		
Se	0.20	2.00	0.010	20	97	2	10
Na	10.0			100	241		
Ag	0.20	0.050	0.050	20	115	10	10
Tl	0.20	2.00	0.050	20	79.1	10	10
V	0.20	0.500	0.500	20	92.7	100	100
Zn	0.20	0.500	0.500	20	246	100	100
Sn	0.20			20	117		
Ti	0.20			20	327		
Mn	0.20	0.500	0.500	20	320	100	100

17.7 Reagents and Stock Solutions which are Purchased as Starting Materials for Preparation of Trace Standards.

From ULTRA SCIENTIFIC:

ICUS-575
ICUS-576
ICM-441
ICUS-574
ICUS-919
ICUS-1932
1,000 μg/mL Ag
10,000 ug/ml Y
1,000 μg/mL Sn
CLP-1
CLP-2
CLP-3

10,000 μg/mL	Al
10,000 μg/mL	Sb
10,000 μg/mL	As
10,000 μg/mL	Ba
10,000 μg/mL	Be
10,000 μg/mL	Cd
10,000 μg/mL	Ca
1,000 μg/mL	Cr
10,000 μg/mL	Co
1,000 μg/mL	Cu
10,000 μg/mL	Fe
10,000 μg/mL	Pb
10,000 μg/mL	Mg
10,000 μg/mL	Mn
10,000 μg/mL	Mo
10,000 μg/mL	Ni
10,000 μg/mL	K
10,000 μg/mL	Se
10,000 μg/mL	Na
10,000 μg/mL	Ag
10,000 μg/mL	Tl
10,000 μg/mL	Zn
10,000 μg/mL	Sn
10,000 μg/mL	Ti

Certificates of Analysis are attached for the custom blend standards listed as ICUS-(…) above.

From JT-BAKER

Concentration HCl (Trace Metals Grade)
Concentration HNO_3 (Trace Metals Grade)

From HIGH PURITY:

Solid $LiNO_3$
1,000 μg/mL Ag
1,000 μg/mL Sn
CAL STD #2-R Solution A

CAL STD #2-R Solution B

Table 17.8 Concentrations of Calibration Standards: (in mg/L)

Element	Std. 1	Std. 2	Std. 3	NAKCAMG100	NAKCAMG400
Al	5.0	25.0	50.0		
Sb	0. 1	0.5	1.0		
As	0. 1	0.5	1.0		
Ba	0. 1	0.5	1.0		
Be	0. 1	0.5	1.0		
Cd	0. 1	0.5	1.0		
Ca	5	25	50	100	400
Cr	0.1	0.5	1.0		
Co	0.1	0.5	1.0		
Cu	0.1	0.5	1.0		
Fe	5	25	50		
Mg	5	25	50	100	400
Mn	0.1	0.5	1.0		
Ni	0.1	0.5	1.0		
Ag	0.1	0.5	1.0		
Tl	0.1	0.5	1.0		
Zn	0.1	0.5	1.0		
V	0.1	0.5	1.0		
B	0.1	0.5	1.0		
Mo	0.1	0.5	1.0		
Ti	0.1	0.5	1.0		
Sn	0.1	0.5	1.0		
Se	0.1	0.5	1.0		
Na	5	25	50.0	100	400
K	5	25	50.0	100	400
Pb	0.1	0.5	1.0		

Table 17.9 Values for IFA and IFB (in mg/L)

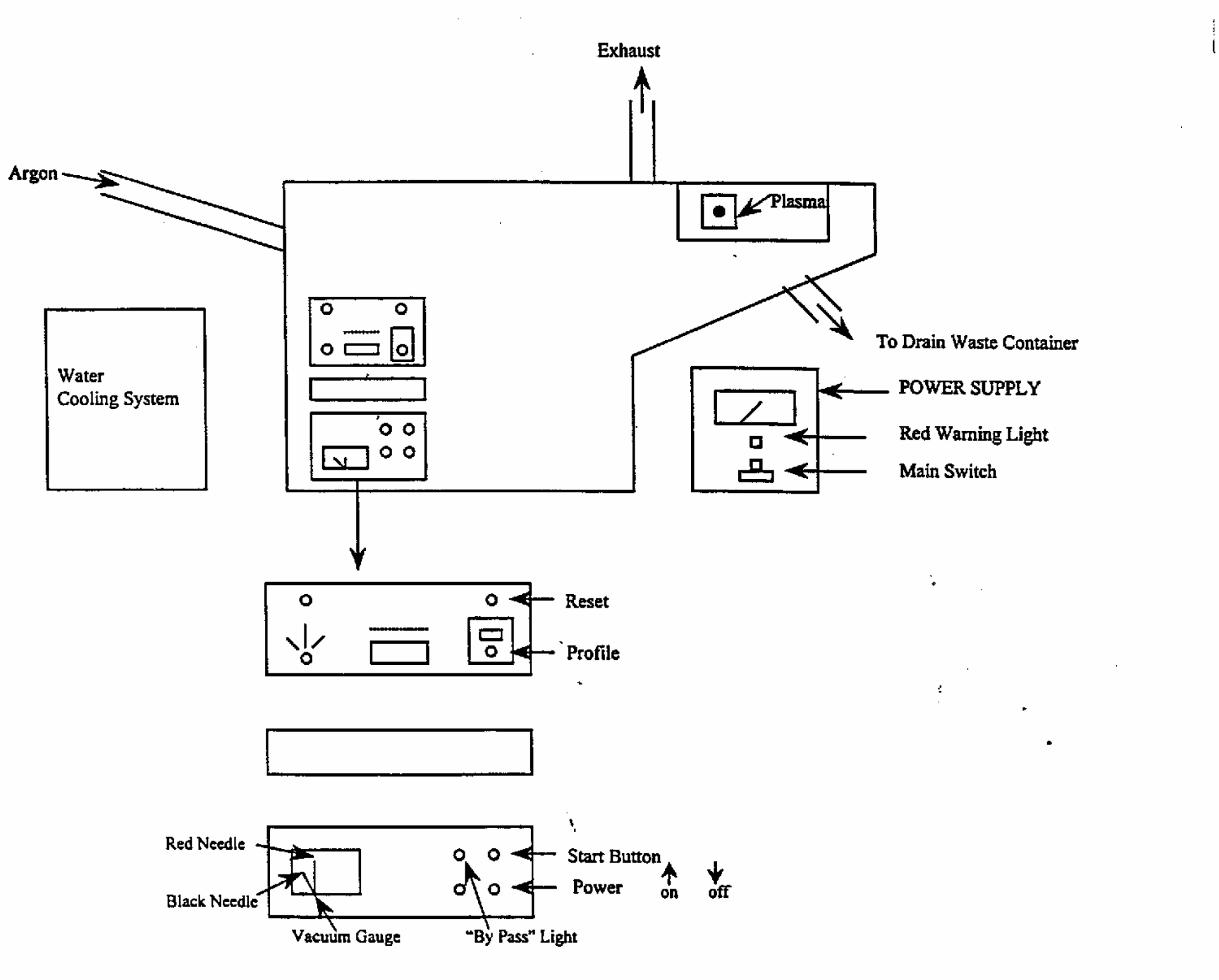
Element	IFB	IFA
Al	500.0	500
Ca	500.0	500
Fe	100.0	200
Mg	500.0	500
Ba	0.5	-
Be	0.5	-
Cd	1.0	-
Co	0.5	-
Cr	0.5	-
Cu	0.5	-
Mn	0.5	-
Ni	1.0	-
Pb	0.05	-
V	0.5	-
Zn	1.0	-
Sb	0.6	-
As	0.1	-
Tl	0.1	-
Se	0.05	-
Ag	0.2	-

Table 17.10 <u>Values for CCV and ICV (in mg/L)</u>:

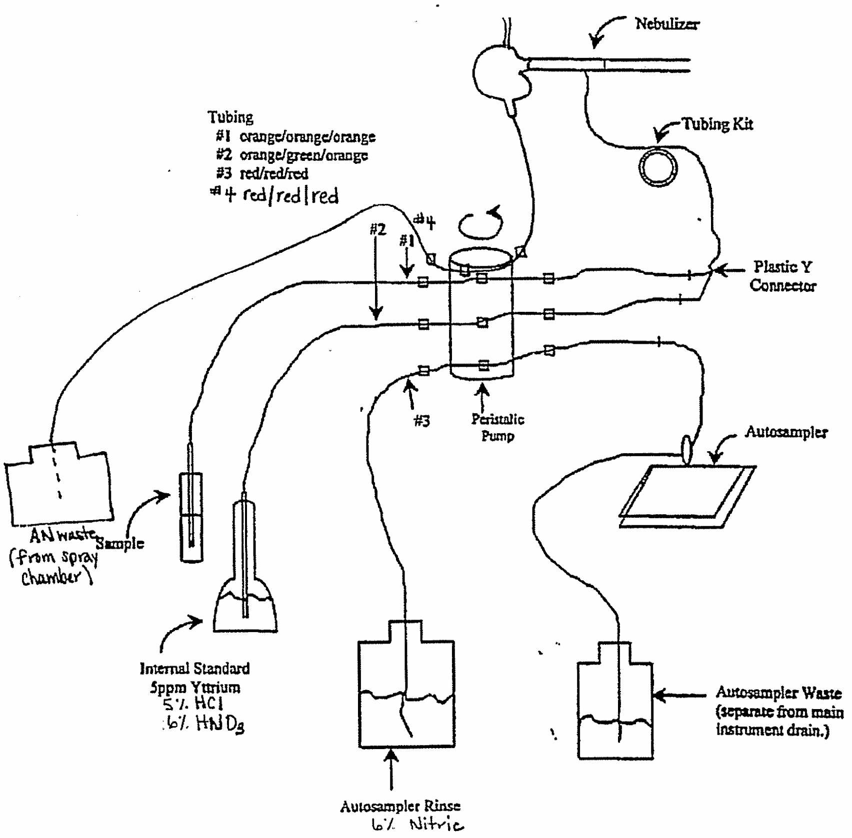
Element	CCV	ICV
Al	25.0	18.75
Sb	0.5	0.375
As	0.5	0.375
Ba	0.5	0.375
Be	0.5	0.375
B	0.5	0.375
Cd	0.5	0.375
Ca	25.0	18.75
Cr	0.5	0.375
Co	0.5	0.375
Cu	0.5	0.375
Fe	25.0	18.75
Pb	0.5	0.375
Mg	25.0	18.75
Mn	0.5	0.375
Mo	0.5	0.375
Ni	0.5	0.375
K	25.0	18.75
Se	0.5	0.375
Na	25.0	18.75
Ag	0.5	0.375
Tl	0.5	0.375
V	0.5	0.375
Zn	0.5	0.375
Sn	0.5	0.375
Ti	0.5	0.375
Mn	0.5	0.375

17.11 Main Instrument Layout:

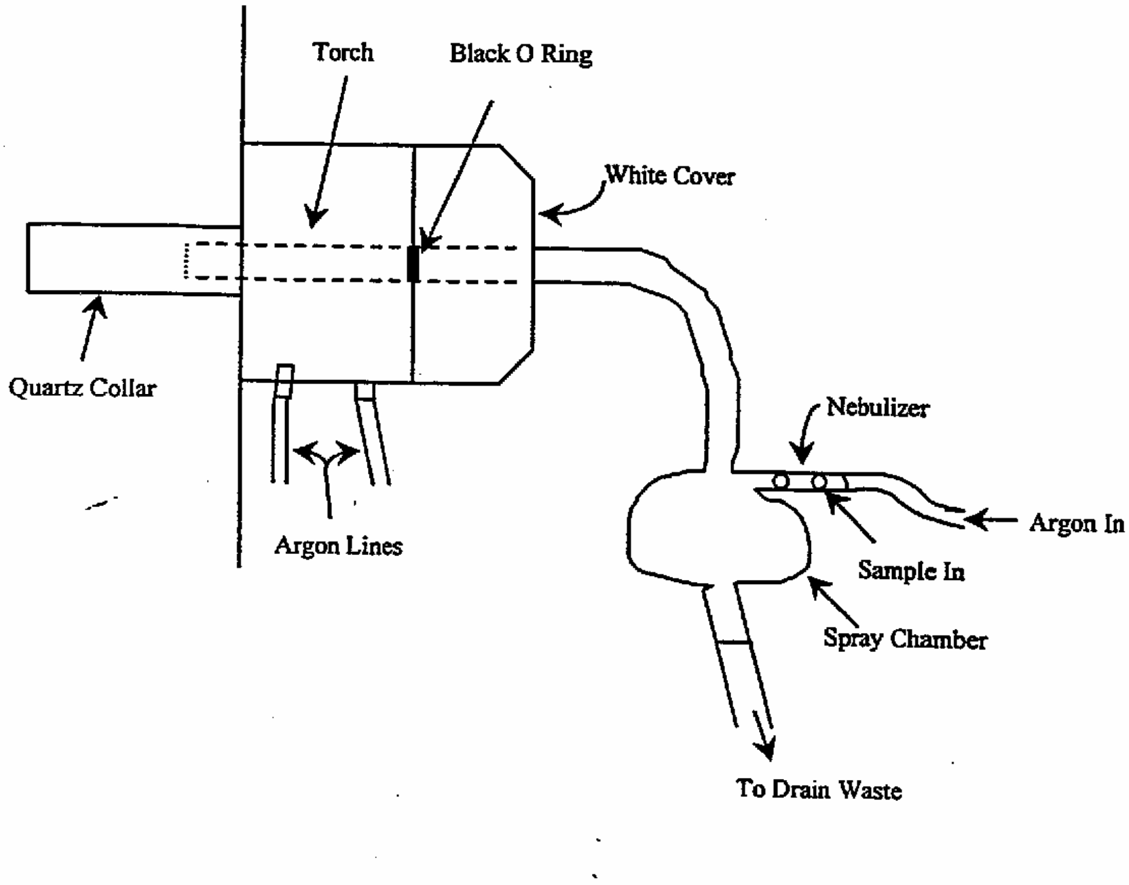
The vacuum pump –not shown- is located behind the instrument. The auto sampler – not shown- is placed in front of the instrument.



17.12 Tubing Layout:



17.13 Torch/Spray Chamber/Nebulizer Assembly:



17.14 Autosampler Layout:

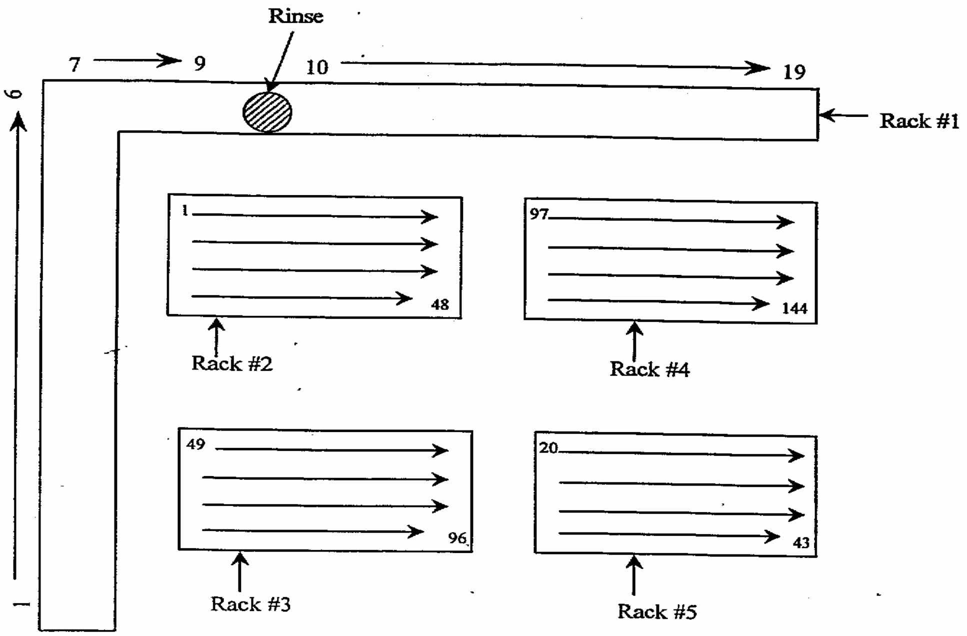


Table 17.15: Method Summary

Method ⇒ Parameter ⇓	EPA Series Method 200.7	***SW-846B*** Method 6010B	CLP
Method Validation (2)	Initial demonstration of performance: 1. Determination of the linear dynamic range. Verify linear calibration range limit by analyzing a high concentration standard. Results must be within 5% of the true value. 2. Analyze LCS within ±10% of stated value. 3. Establish MDLs using Laboratory fortified blanks. MDLs must meet regulatory levels.	Same	Same
QC Check Standards/Samples (ICV)	Verify each element calibration with a Control Sample (ICV) prepared from a source different than the calibration standards at the following concentrations: Silver at a maximum of 0.5mg/L, other elements >1.0 mg/L. The ICV must be within 3% RSD. %R = 95-105	Verify each element calibration with an Instrument Check Standard prepared from a source different from the calibration standards at a concentration equivalent to the midpoint of the calibration curves. The ICV must be within 5% RSD. %R=90-110	Initial calibration verified with independent standard. %R=90-110
Method Detection Limit	IDL's are determined quarterly. MDLs are determined annually	IDL's are determined quarterly. MDLs are determined annually	IDL's are determined quarterly. MDLs are determined annually.
Standard Solution Expiration(3)	Stocks: yearly or specified by supplier Intermediate: 6 mos. Working: 3 days to 7 days	(same)	(same)
Initial Calibration	Per instrument manufacturer's specification. Minimum of a blank and one standard.	Per instrument manufacturer's specifications (should consist of 3 levels and a blank).	Per instrument manufacturer's specifications. Minimum of a blank and one standard.
Continuing Calibration	Analyze instrument performance check (ICV) solution immediately following calibration, after every 10 samples and at the end of the run. For initial analysis, %R=95-105, for subsequent analyses of standard. %R=90-110.	Analyze instrument check standard after every 10 samples and at the end of the run. %R=90-110.	Calibration checked after every 10 samples or 2 hours whichever is more frequent with a mid-range calibration standard. %R=90-110.
Accuracy/Precision	-One MS/MD per 10 samples or each batch %R=75-125%. %RPD < 20% - A laboratory fortified blank (LFB) prepared with each batch of samples digested. %R=85-115.	-One MS/MD per 10 samples or each batch %R=75-125%. %RPD < 20% - A laboratory fortified blank (LFB) prepared with each batch of samples digested. %R=80-120	One MS and one duplicate per sample delivery group or per similar matrix type.%R=75-125. %RPD<20. Analyze a post-digestion spike if the pre-digestion spike recovery is outside control limits and the sample result does not exceed 4 times the spike added.

Method ⇒ Parameter ⇓	EPA Series Method 200.7	***SW-846B*** Method 6010B	CLP
Blanks	One method blank with each batch of samples. When values constitute 10% or more of the analyte level or are 2.2 times the analyte MDL, whichever is greater, the entire preparation and analysis is repeated. Analyze calibration blank after each instrument check standard (CCV). Blank < 3x IDL.	One method blank per batch of samples processed at the same time. Analyze calibration blank after each Instrument Check Standard (CCV). Blank < 3x IDL.	One method blank per sample delivery group or per sample process batch, whichever is more frequent. Analyze calibration blanks after initial and continuing calibration verification or every 10 samples. If absolute value of blank for any analyte >CRDL, terminate analysis, correct problem, and reanalyze all samples since last compliant blank.
Interference Check Standard	Interference Check Solutions containing known concentrations of interfering elements and the elements of interest are analyzed at the beginning of each analytical run. %R=80-120. Reanalyze highest standard after calibration for checking (not required by the method). %R=95-105.	Interference Check Solutions containing known concentrations of interfering elements and the elements of interest are analyzed at the beginning of each analytical run. %R=80-120. Reanalyze highest standard after calibration for checking (not required by the method). %R=95-105.	Analyze IFA and IFB solutions at a frequency of not greater than 20 analytical samples. Must be followed immediately by CCV/CCB pair. IFA: For target analytes with CRDL ≤10 µg/L, results should fall within ± CRDL of the analyte's true value, otherwise use alternate method to quantitate results for affected analytes. IFB: %R=80-120
IDL Standard (LCV)	Run LCV Standard at the beginning of each analytical run directly following ICB. %R=50-150. Not required per method..	Run LCV Standard at the beginning of each analytical run directly following ICB.. %R=50-150. Not required per method.	Run LCV Standard every 20 analytical samples and at the beginning and end of each analytical run. %R=50-150 For Antimony, Lead, and Thallium %R=70-130 All other elements
Serial Dilution	1:5 Dilution on each new matrix per job. % Difference=10%	1:5 Dilution on each new matrix per job. % Difference=10%	1:5 Dilution on each new matrix per SDG. % Difference=10%
Matrix Spike	%R= 75-125	%R=75-125	%R=75-125
Post Digestion Spike	%R=85-115	%R=75-125	%R=75-125
Holding Time (4)	180 days from collection	180 days from collection	180 days from receipt (VTSR)

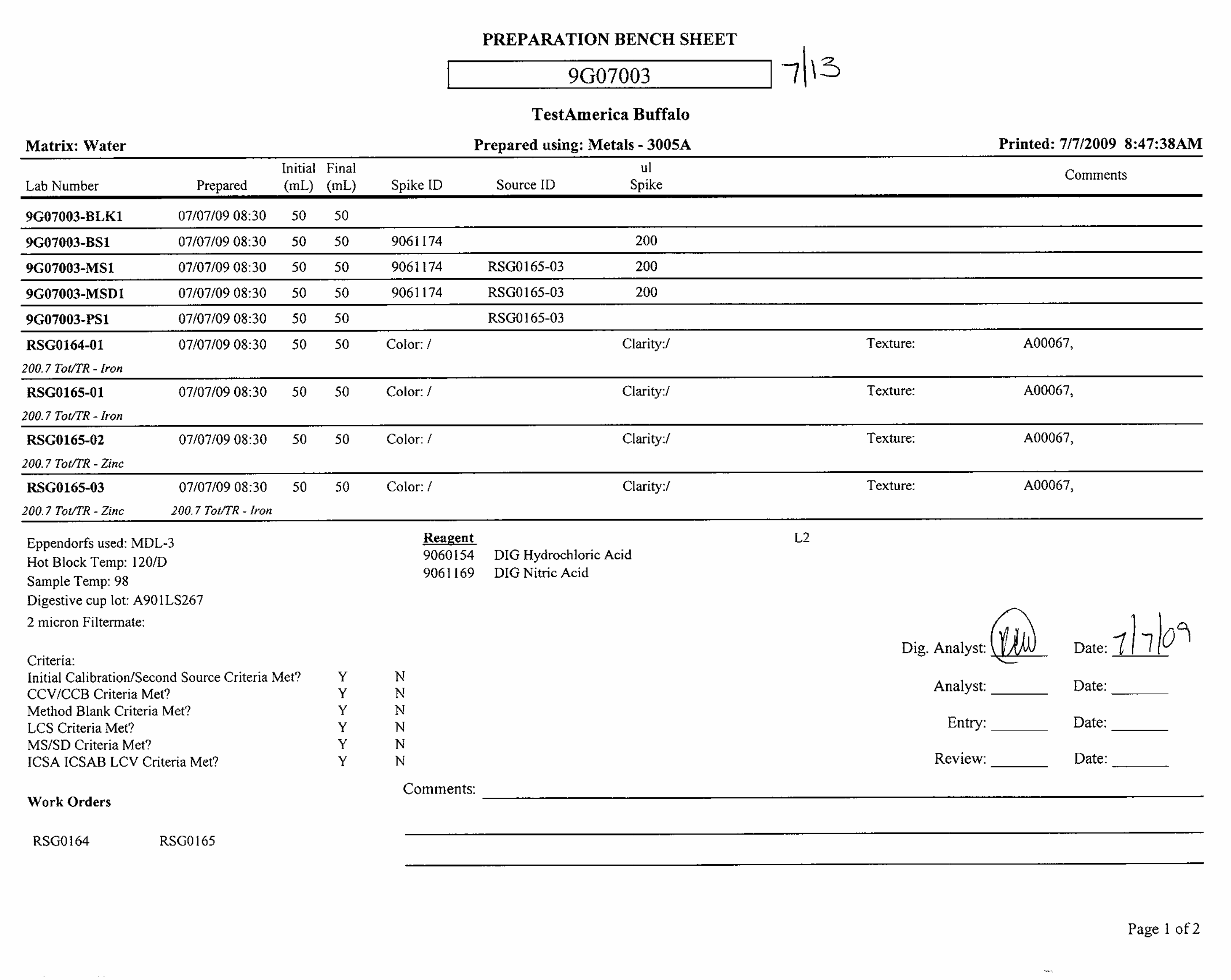
Table 17.16 Contract Required Detection Limits (CRDL)

Analyte	CRDL (4.0) (ng/mL)	CRDL (5.0) (ng/mL)
Aluminum	200	200
Antimony	60	5
Arsenic	10	5
Barium	200	20
Beryllium	5	1
Cadmium	5	2
Calcium	5000	5000
Chromium	10	5
Cobalt	50	5
Copper	25	5
Iron	100	100
Lead	3	3
Magnesium	5000	5000
Manganese	15	10
Mercury	0.2	0.1
Nickel	40	20
Potassium	5000	5000
Selenium	5	5
Silver	10	5
Sodium	5000	5000
Thallium	10	5
Vanadium	50	10
Zinc	20	10

17.17 Concentration of each element in the LCV standard solution and reading on the instrument.

Analyte	LCV Stock Std	LCV
Aluminum	2.0	0.2
Antimony	0.2	0.02
Arsenic	0.1	0.01
Barium	0.02	0.002
Beryllium	0.02	0.002
Boron	0.2	0.02
Cadmium	0.01	0.001
Calcium	5.0	0.05
Chromium	0.04	0.004
Cobalt	0.04	0.004
Copper	0.1	0.01
Iron	0.5	0.05
Lead	0.05	0.005
Magnesium	2.0	0.2
Manganese	0.03	0.003
Nickel	0.1	0.01
Potassium	5.0	0.5
Selenium	0.15	0.015
Silver	0.03	0.003
Sodium	10.0	1.0
Thallium	0.2	0.02
Tin	0.1	0.01
Vanadium	0.05	0.005
Zinc	0.1	0.01

17.18 Example of a Bench Sheet for Metals



PREPARATION BENCH SHEET

9G07003 7/13

TestAmerica Buffalo

Matrix: Water **Prepared using: Metals - 3005A** **Printed: 7/7/2009 8:47:38AM**

Lab Number	Prepared	Initial (mL)	Final (mL)	Spike ID	Source ID	ul Spike		Comments
9G07003-BLK1	07/07/09 08:30	50	50					
9G07003-BS1	07/07/09 08:30	50	50	9061174		200		
9G07003-MS1	07/07/09 08:30	50	50	9061174	RSG0165-03	200		
9G07003-MSD1	07/07/09 08:30	50	50	9061174	RSG0165-03	200		
9G07003-PS1	07/07/09 08:30	50	50		RSG0165-03			
RSG0164-01 *200.7 Tot/TR - Iron*	07/07/09 08:30	50	50	Color: /		Clarity:/	Texture:	A00067,
RSG0165-01 *200.7 Tot/TR - Iron*	07/07/09 08:30	50	50	Color: /		Clarity:/	Texture:	A00067,
RSG0165-02 *200.7 Tot/TR - Zinc*	07/07/09 08:30	50	50	Color: /		Clarity:/	Texture:	A00067,
RSG0165-03 *200.7 Tot/TR - Zinc*	07/07/09 08:30 *200.7 Tot/TR - Iron*	50	50	Color: /		Clarity:/	Texture:	A00067,

Eppendorfs used: MDL-3
Hot Block Temp: 120/D
Sample Temp: 98
Digestive cup lot: A901LS267
2 micron Filtermate:

Reagent	
9060154	DIG Hydrochloric Acid
9061169	DIG Nitric Acid

L2

Criteria:

Initial Calibration/Second Source Criteria Met?	Y	N
CCV/CCB Criteria Met?	Y	N
Method Blank Criteria Met?	Y	N
LCS Criteria Met?	Y	N
MS/SD Criteria Met?	Y	N
ICSA ICSAB LCV Criteria Met?	Y	N

Dig. Analyst: ______ Date: 7/7/09
Analyst: ______ Date: ______
Entry: ______ Date: ______
Review: ______ Date: ______

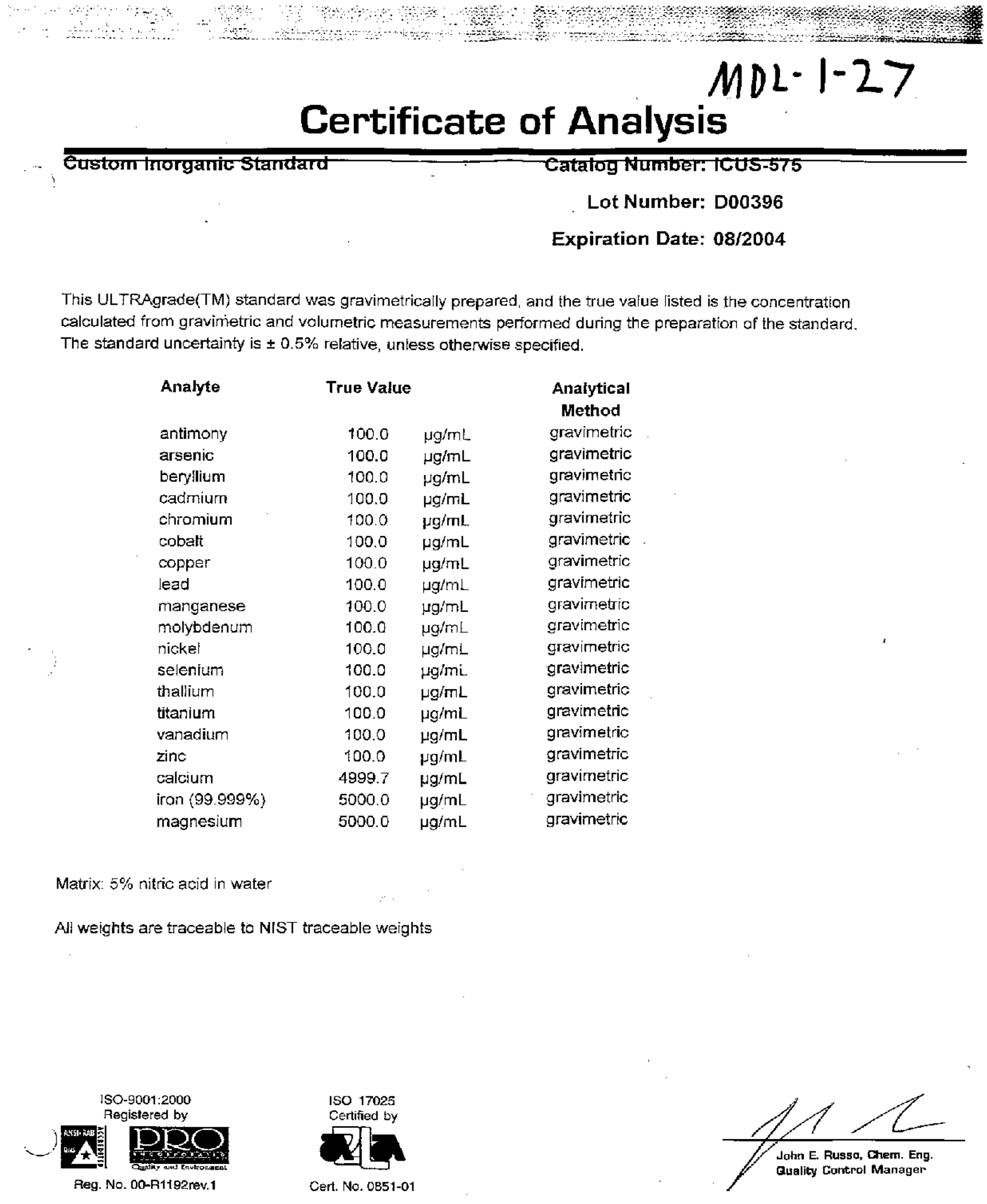
Comments: ______

Work Orders

RSG0164 RSG0165

Page 1 of 2

17.19 Certificates of Analysis for Custom Blend Standards



MDL-1-27

Certificate of Analysis

Custom Inorganic Standard **Catalog Number: ICUS-575**

Lot Number: D00396

Expiration Date: 08/2004

This ULTRAgrade(TM) standard was gravimetrically prepared, and the true value listed is the concentration calculated from gravimetric and volumetric measurements performed during the preparation of the standard. The standard uncertainty is ± 0.5% relative, unless otherwise specified.

Analyte	True Value		Analytical Method
antimony	100.0	µg/mL	gravimetric
arsenic	100.0	µg/mL	gravimetric
beryllium	100.0	µg/mL	gravimetric
cadmium	100.0	µg/mL	gravimetric
chromium	100.0	µg/mL	gravimetric
cobalt	100.0	µg/mL	gravimetric
copper	100.0	µg/mL	gravimetric
lead	100.0	µg/mL	gravimetric
manganese	100.0	µg/mL	gravimetric
molybdenum	100.0	µg/mL	gravimetric
nickel	100.0	µg/mL	gravimetric
selenium	100.0	µg/mL	gravimetric
thallium	100.0	µg/mL	gravimetric
titanium	100.0	µg/mL	gravimetric
vanadium	100.0	µg/mL	gravimetric
zinc	100.0	µg/mL	gravimetric
calcium	4999.7	µg/mL	gravimetric
iron (99.999%)	5000.0	µg/mL	gravimetric
magnesium	5000.0	µg/mL	gravimetric

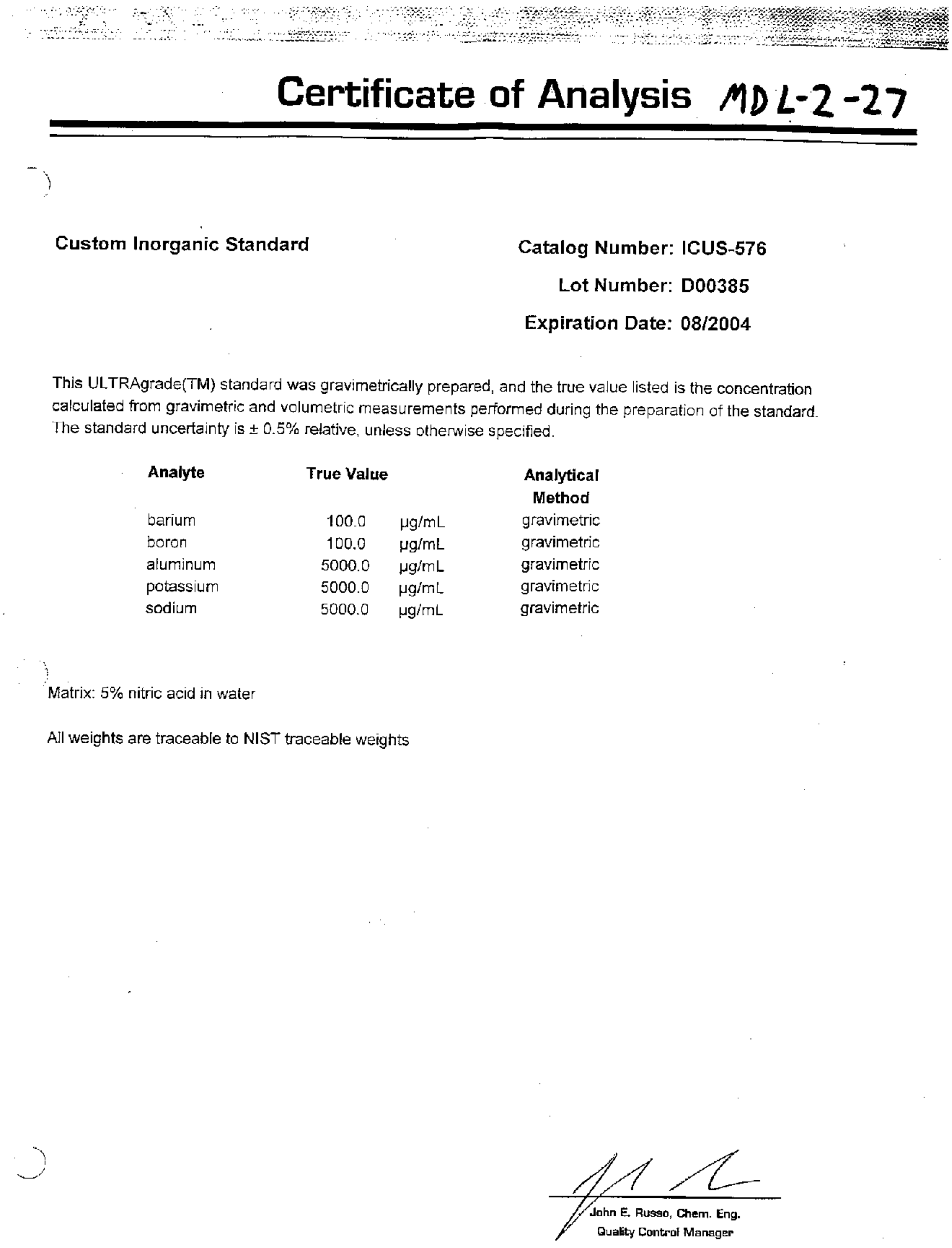
Matrix: 5% nitric acid in water

All weights are traceable to NIST traceable weights

ISO-9001:2000
Registered by
Reg. No. 00-R1192rev.1

ISO 17025
Certified by
Cert. No. 0851-01

John E. Russo, Chem. Eng.
Quality Control Manager



Certificate of Analysis MDL-2-27

Custom Inorganic Standard

Catalog Number: ICUS-576
Lot Number: D00385
Expiration Date: 08/2004

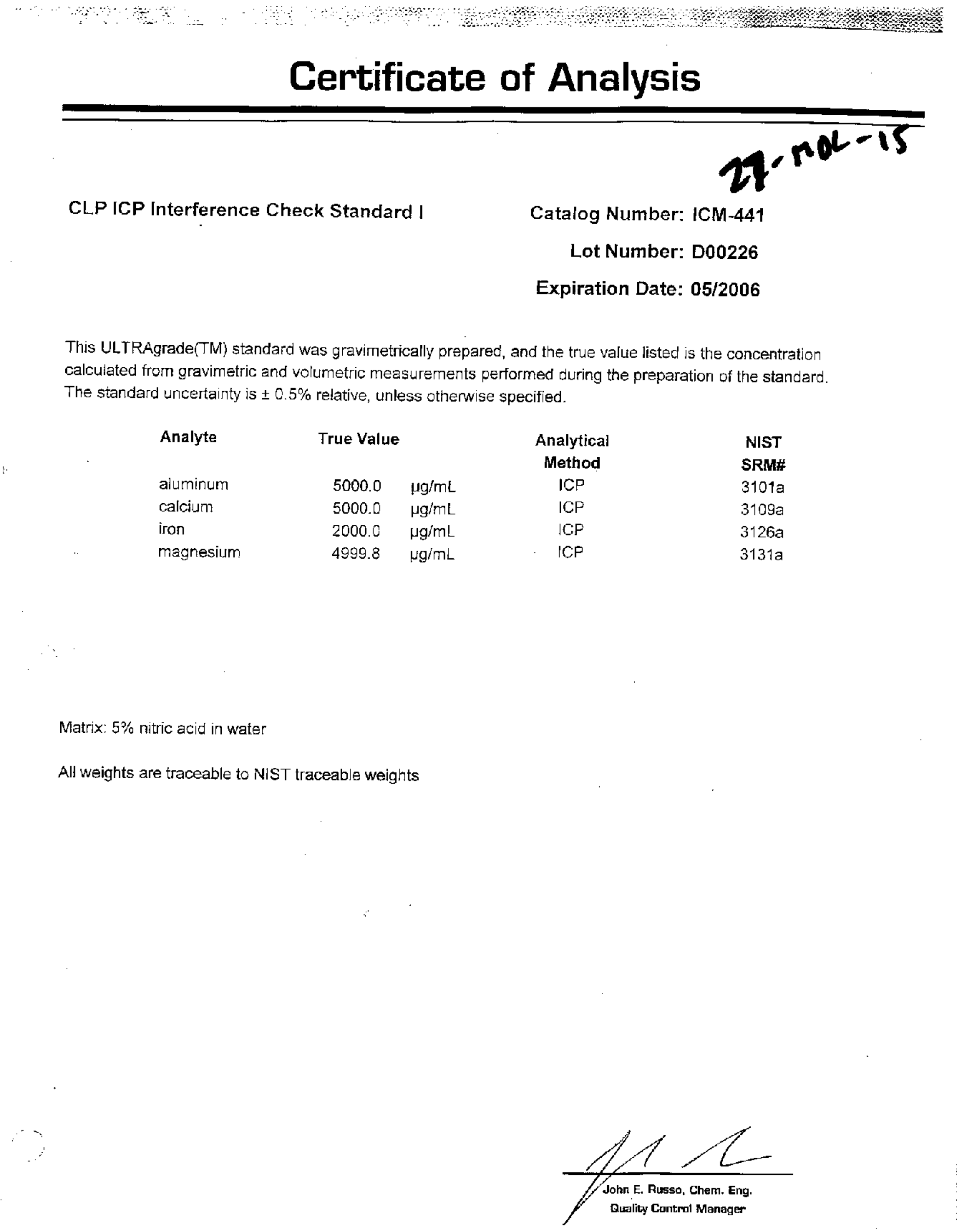
This ULTRAgrade(TM) standard was gravimetrically prepared, and the true value listed is the concentration calculated from gravimetric and volumetric measurements performed during the preparation of the standard. The standard uncertainty is ± 0.5% relative, unless otherwise specified.

Analyte	True Value		Analytical Method
barium	100.0	µg/mL	gravimetric
boron	100.0	µg/mL	gravimetric
aluminum	5000.0	µg/mL	gravimetric
potassium	5000.0	µg/mL	gravimetric
sodium	5000.0	µg/mL	gravimetric

Matrix: 5% nitric acid in water

All weights are traceable to NIST traceable weights

John E. Russo, Chem. Eng.
Quality Control Manager



Certificate of Analysis

27-NOV-15

CLP ICP Interference Check Standard I

Catalog Number: ICM-441

Lot Number: D00226

Expiration Date: 05/2006

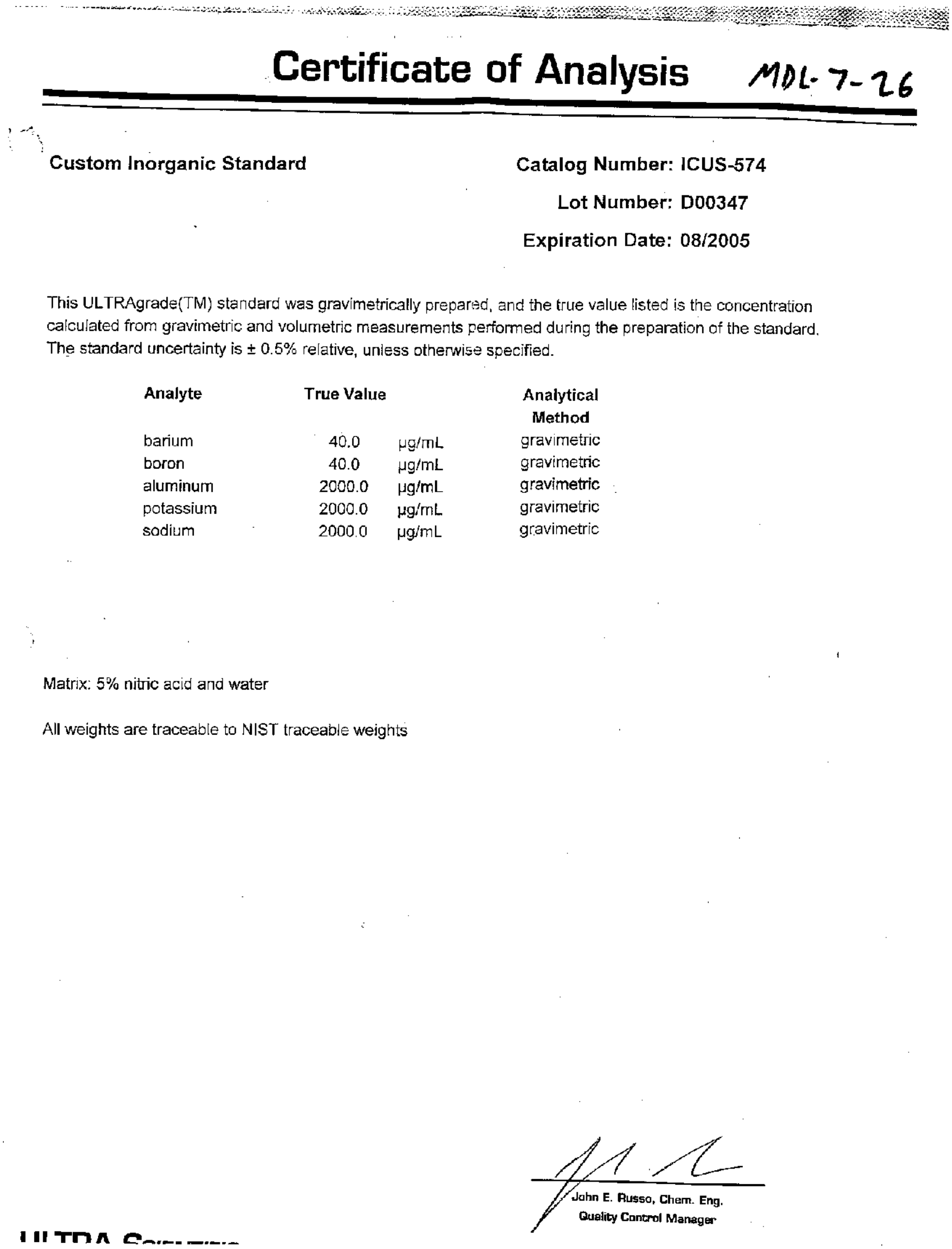
This ULTRAgrade(TM) standard was gravimetrically prepared, and the true value listed is the concentration calculated from gravimetric and volumetric measurements performed during the preparation of the standard. The standard uncertainty is ± 0.5% relative, unless otherwise specified.

Analyte	True Value		Analytical Method	NIST SRM#
aluminum	5000.0	µg/mL	ICP	3101a
calcium	5000.0	µg/mL	ICP	3109a
iron	2000.0	µg/mL	ICP	3126a
magnesium	4999.8	µg/mL	ICP	3131a

Matrix: 5% nitric acid in water

All weights are traceable to NIST traceable weights

John E. Russo, Chem. Eng.
Quality Control Manager



Certificate of Analysis

MDL-7-26

Custom Inorganic Standard

Catalog Number: ICUS-574

Lot Number: D00347

Expiration Date: 08/2005

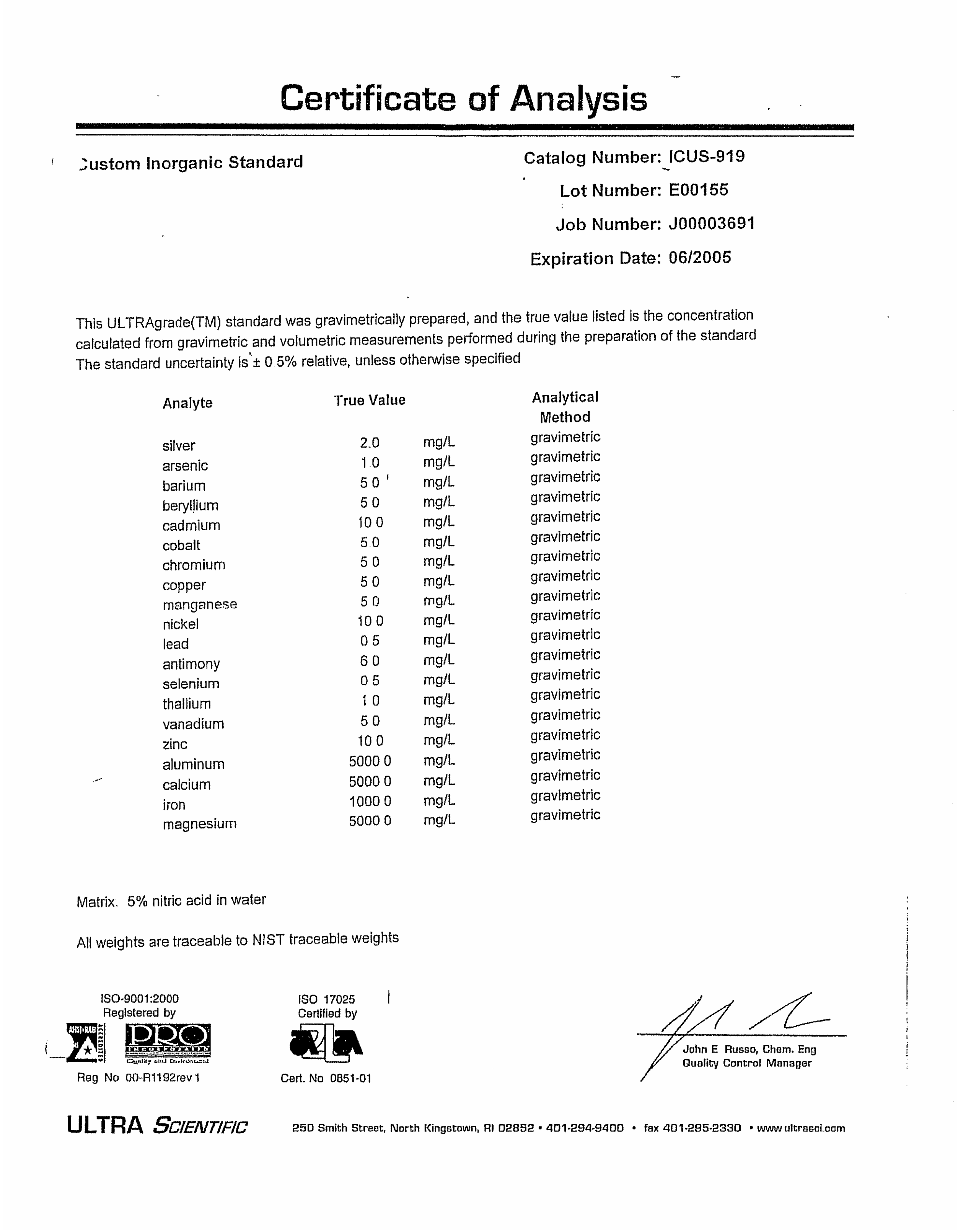
This ULTRAgrade(TM) standard was gravimetrically prepared, and the true value listed is the concentration calculated from gravimetric and volumetric measurements performed during the preparation of the standard. The standard uncertainty is ± 0.5% relative, unless otherwise specified.

Analyte	True Value		Analytical Method
barium	40.0	µg/mL	gravimetric
boron	40.0	µg/mL	gravimetric
aluminum	2000.0	µg/mL	gravimetric
potassium	2000.0	µg/mL	gravimetric
sodium	2000.0	µg/mL	gravimetric

Matrix: 5% nitric acid and water

All weights are traceable to NIST traceable weights

John E. Russo, Chem. Eng.
Quality Control Manager



Certificate of Analysis

Custom Inorganic Standard

Catalog Number: ICUS-919
Lot Number: E00155
Job Number: J00003691
Expiration Date: 06/2005

This ULTRAgrade(TM) standard was gravimetrically prepared, and the true value listed is the concentration calculated from gravimetric and volumetric measurements performed during the preparation of the standard The standard uncertainty is ± 0 5% relative, unless otherwise specified

Analyte	True Value		Analytical Method
silver	2.0	mg/L	gravimetric
arsenic	1 0	mg/L	gravimetric
barium	5 0	mg/L	gravimetric
beryllium	5 0	mg/L	gravimetric
cadmium	10 0	mg/L	gravimetric
cobalt	5 0	mg/L	gravimetric
chromium	5 0	mg/L	gravimetric
copper	5 0	mg/L	gravimetric
manganese	5 0	mg/L	gravimetric
nickel	10 0	mg/L	gravimetric
lead	0 5	mg/L	gravimetric
antimony	6 0	mg/L	gravimetric
selenium	0 5	mg/L	gravimetric
thallium	1 0	mg/L	gravimetric
vanadium	5 0	mg/L	gravimetric
zinc	10 0	mg/L	gravimetric
aluminum	5000 0	mg/L	gravimetric
calcium	5000 0	mg/L	gravimetric
iron	1000 0	mg/L	gravimetric
magnesium	5000 0	mg/L	gravimetric

Matrix. 5% nitric acid in water

All weights are traceable to NIST traceable weights

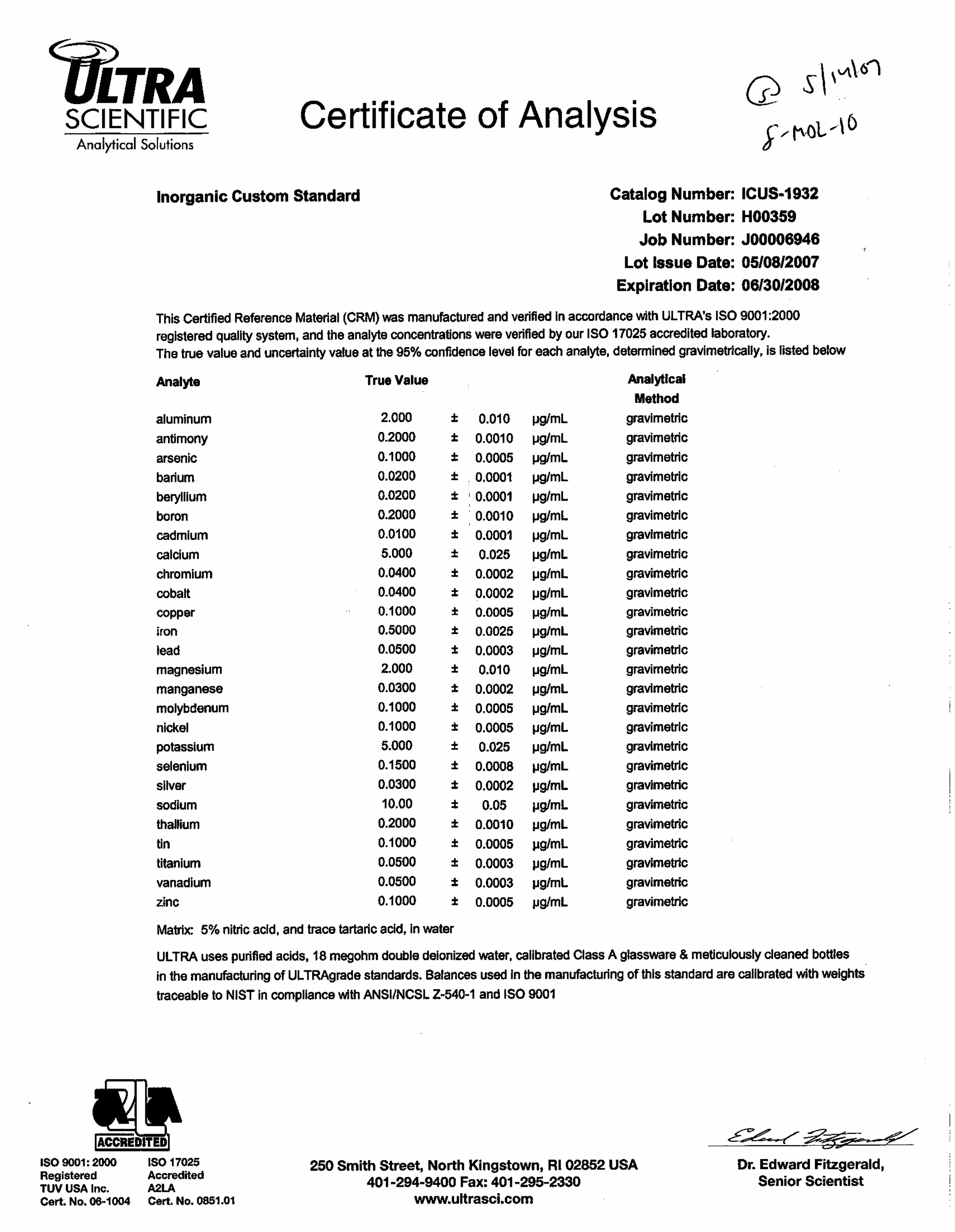
ISO-9001:2000
Registered by
Reg No 00-R1192rev 1

ISO 17025
Certified by
Cert. No 0851-01

John E Russo, Chem. Eng
Quality Control Manager

ULTRA *SCIENTIFIC*

250 Smith Street, North Kingstown, RI 02852 • 401-294-9400 • fax 401-295-2330 • www ultrasci.com



ULTRA SCIENTIFIC
Analytical Solutions

Certificate of Analysis

Ⓢ 5/14/07
S-MOL-10

Inorganic Custom Standard

Catalog Number: ICUS-1932
Lot Number: H00359
Job Number: J00006946
Lot Issue Date: 05/08/2007
Expiration Date: 06/30/2008

This Certified Reference Material (CRM) was manufactured and verified in accordance with ULTRA's ISO 9001:2000 registered quality system, and the analyte concentrations were verified by our ISO 17025 accredited laboratory. The true value and uncertainty value at the 95% confidence level for each analyte, determined gravimetrically, is listed below

Analyte	**True Value**				**Analytical Method**
aluminum	2.000	±	0.010	µg/mL	gravimetric
antimony	0.2000	±	0.0010	µg/mL	gravimetric
arsenic	0.1000	±	0.0005	µg/mL	gravimetric
barium	0.0200	±	0.0001	µg/mL	gravimetric
beryllium	0.0200	±	0.0001	µg/mL	gravimetric
boron	0.2000	±	0.0010	µg/mL	gravimetric
cadmium	0.0100	±	0.0001	µg/mL	gravimetric
calcium	5.000	±	0.025	µg/mL	gravimetric
chromium	0.0400	±	0.0002	µg/mL	gravimetric
cobalt	0.0400	±	0.0002	µg/mL	gravimetric
copper	0.1000	±	0.0005	µg/mL	gravimetric
iron	0.5000	±	0.0025	µg/mL	gravimetric
lead	0.0500	±	0.0003	µg/mL	gravimetric
magnesium	2.000	±	0.010	µg/mL	gravimetric
manganese	0.0300	±	0.0002	µg/mL	gravimetric
molybdenum	0.1000	±	0.0005	µg/mL	gravimetric
nickel	0.1000	±	0.0005	µg/mL	gravimetric
potassium	5.000	±	0.025	µg/mL	gravimetric
selenium	0.1500	±	0.0008	µg/mL	gravimetric
silver	0.0300	±	0.0002	µg/mL	gravimetric
sodium	10.00	±	0.05	µg/mL	gravimetric
thallium	0.2000	±	0.0010	µg/mL	gravimetric
tin	0.1000	±	0.0005	µg/mL	gravimetric
titanium	0.0500	±	0.0003	µg/mL	gravimetric
vanadium	0.0500	±	0.0003	µg/mL	gravimetric
zinc	0.1000	±	0.0005	µg/mL	gravimetric

Matrix: 5% nitric acid, and trace tartaric acid, in water

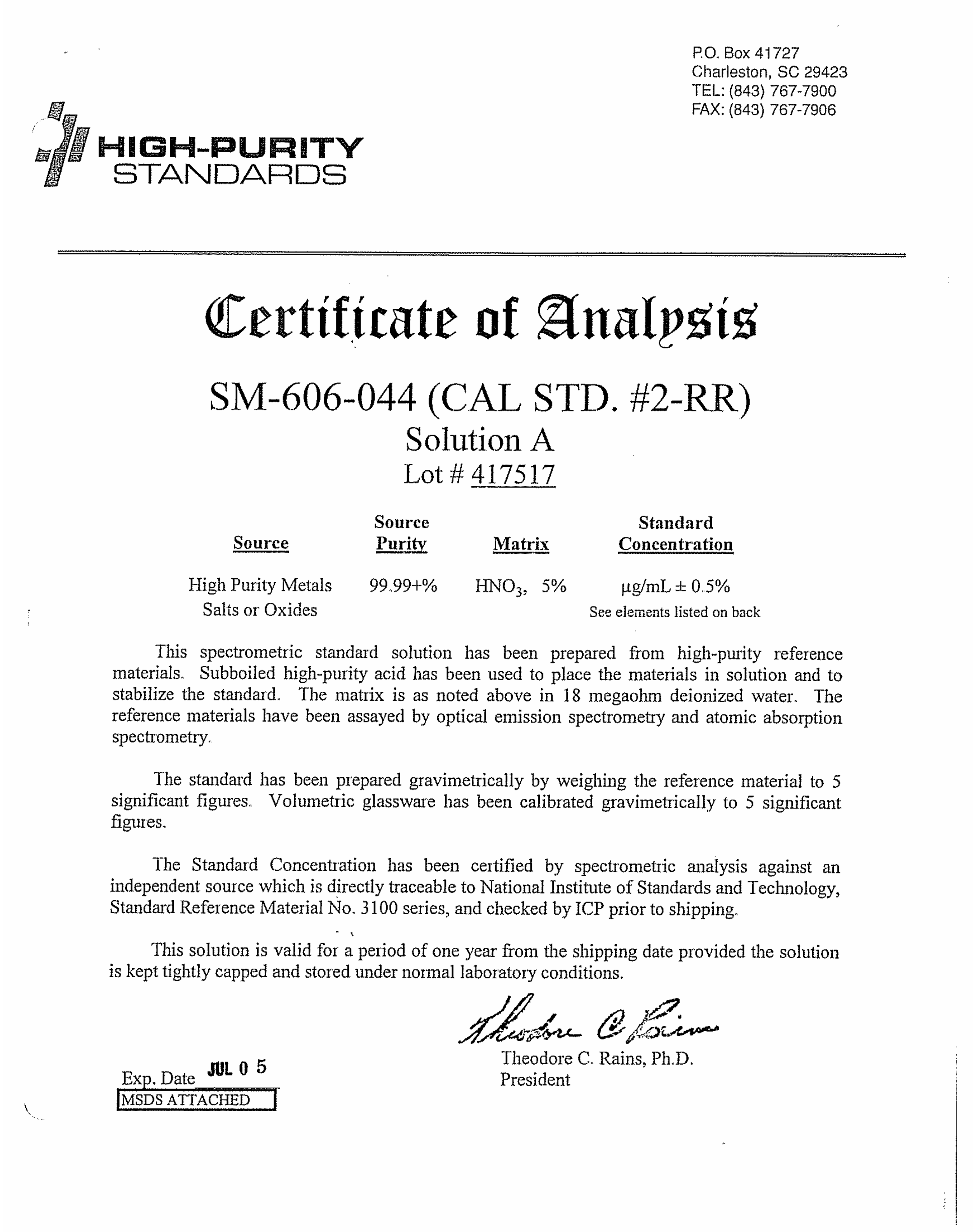
ULTRA uses purified acids, 18 megohm double deionized water, calibrated Class A glassware & meticulously cleaned bottles in the manufacturing of ULTRAgrade standards. Balances used in the manufacturing of this standard are calibrated with weights traceable to NIST in compliance with ANSI/NCSL Z-540-1 and ISO 9001

A2LA ACCREDITED

ISO 9001: 2000	ISO 17025
Registered	Accredited
TUV USA Inc.	A2LA
Cert. No. 06-1004	Cert. No. 0851.01

250 Smith Street, North Kingstown, RI 02852 USA
401-294-9400 Fax: 401-295-2330
www.ultrasci.com

Edward Fitzgerald
Dr. Edward Fitzgerald,
Senior Scientist



P.O. Box 41727
Charleston, SC 29423
TEL: (843) 767-7900
FAX: (843) 767-7906

**HIGH-PURITY
STANDARDS**

Certificate of Analysis

SM-606-044 (CAL STD. #2-RR)

Solution A

Lot # 417517

Source	Source Purity	Matrix	Standard Concentration
High Purity Metals Salts or Oxides	99.99+%	HNO_3, 5%	µg/mL ± 0.5% See elements listed on back

This spectrometric standard solution has been prepared from high-purity reference materials. Subboiled high-purity acid has been used to place the materials in solution and to stabilize the standard. The matrix is as noted above in 18 megaohm deionized water. The reference materials have been assayed by optical emission spectrometry and atomic absorption spectrometry.

The standard has been prepared gravimetrically by weighing the reference material to 5 significant figures. Volumetric glassware has been calibrated gravimetrically to 5 significant figures.

The Standard Concentration has been certified by spectrometric analysis against an independent source which is directly traceable to National Institute of Standards and Technology, Standard Reference Material No. 3100 series, and checked by ICP prior to shipping.

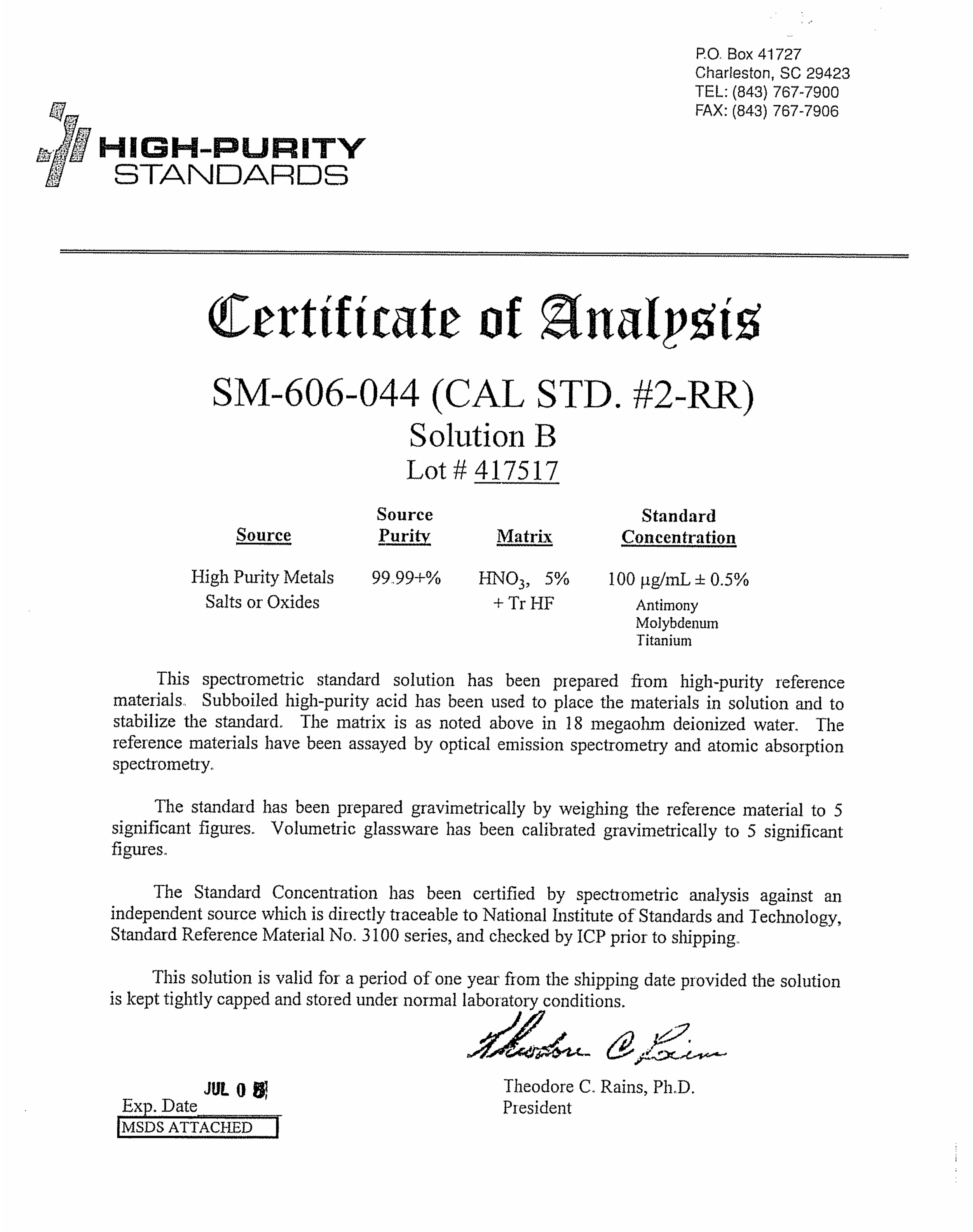
This solution is valid for a period of one year from the shipping date provided the solution is kept tightly capped and stored under normal laboratory conditions.

Theodore C Rains

Theodore C. Rains, Ph.D.
President

Exp. Date JUL 0 5

MSDS ATTACHED



P.O. Box 41727
Charleston, SC 29423
TEL: (843) 767-7900
FAX: (843) 767-7906

HIGH-PURITY STANDARDS

Certificate of Analysis

SM-606-044 (CAL STD. #2-RR)

Solution B

Lot # 417517

Source	Source Purity	Matrix	Standard Concentration
High Purity Metals Salts or Oxides	99.99+%	HNO_3, 5% + Tr HF	100 µg/mL ± 0.5% Antimony Molybdenum Titanium

This spectrometric standard solution has been prepared from high-purity reference materials. Subboiled high-purity acid has been used to place the materials in solution and to stabilize the standard. The matrix is as noted above in 18 megaohm deionized water. The reference materials have been assayed by optical emission spectrometry and atomic absorption spectrometry.

The standard has been prepared gravimetrically by weighing the reference material to 5 significant figures. Volumetric glassware has been calibrated gravimetrically to 5 significant figures.

The Standard Concentration has been certified by spectrometric analysis against an independent source which is directly traceable to National Institute of Standards and Technology, Standard Reference Material No. 3100 series, and checked by ICP prior to shipping.

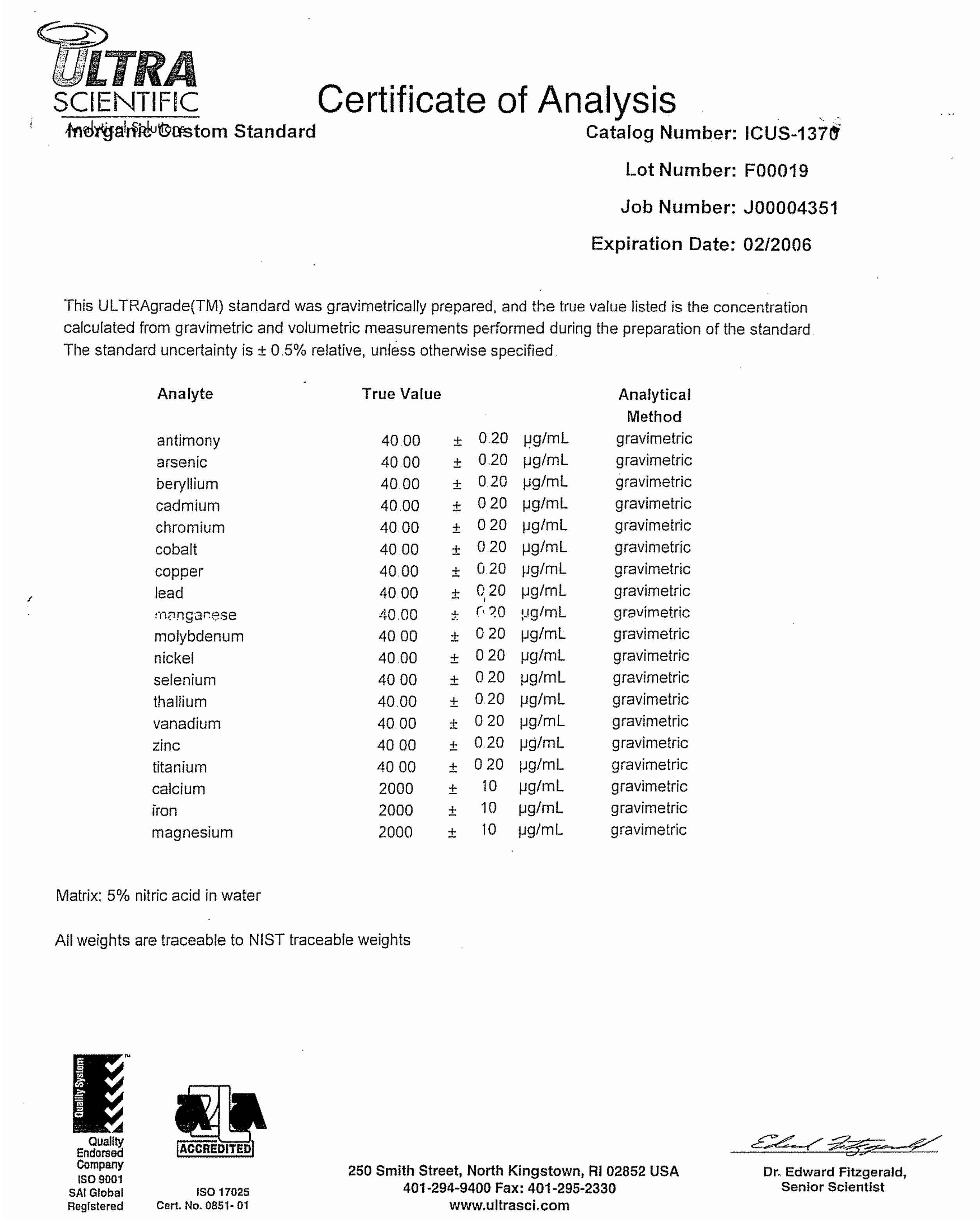
This solution is valid for a period of one year from the shipping date provided the solution is kept tightly capped and stored under normal laboratory conditions.

Theodore C. Rains

Exp. Date JUL 0 8

MSDS ATTACHED

Theodore C. Rains, Ph.D.
President



ULTRA SCIENTIFIC
Analytical Solutions

Certificate of Analysis

Inorganic Custom Standard

Catalog Number: ICUS-1370
Lot Number: F00019
Job Number: J00004351
Expiration Date: 02/2006

This ULTRAgrade(TM) standard was gravimetrically prepared, and the true value listed is the concentration calculated from gravimetric and volumetric measurements performed during the preparation of the standard. The standard uncertainty is ± 0.5% relative, unless otherwise specified.

Analyte	True Value				Analytical Method
antimony	40.00	±	0.20	µg/mL	gravimetric
arsenic	40.00	±	0.20	µg/mL	gravimetric
beryllium	40.00	±	0.20	µg/mL	gravimetric
cadmium	40.00	±	0.20	µg/mL	gravimetric
chromium	40.00	±	0.20	µg/mL	gravimetric
cobalt	40.00	±	0.20	µg/mL	gravimetric
copper	40.00	±	0.20	µg/mL	gravimetric
lead	40.00	±	0.20	µg/mL	gravimetric
manganese	40.00	±	0.20	µg/mL	gravimetric
molybdenum	40.00	±	0.20	µg/mL	gravimetric
nickel	40.00	±	0.20	µg/mL	gravimetric
selenium	40.00	±	0.20	µg/mL	gravimetric
thallium	40.00	±	0.20	µg/mL	gravimetric
vanadium	40.00	±	0.20	µg/mL	gravimetric
zinc	40.00	±	0.20	µg/mL	gravimetric
titanium	40.00	±	0.20	µg/mL	gravimetric
calcium	2000	±	10	µg/mL	gravimetric
iron	2000	±	10	µg/mL	gravimetric
magnesium	2000	±	10	µg/mL	gravimetric

Matrix: 5% nitric acid in water

All weights are traceable to NIST traceable weights

Quality System
Quality Endorsed Company
ISO 9001
SAI Global Registered

A2LA
ACCREDITED
ISO 17025
Cert. No. 0851-01

250 Smith Street, North Kingstown, RI 02852 USA
401-294-9400 Fax: 401-295-2330
www.ultrasci.com

Edward Fitzgerald
Dr. Edward Fitzgerald,
Senior Scientist

TestAmerica
THE LEADER IN ENVIRONMENTAL TESTING

Title: Total Organic Carbon
Method: 415.1, 9060 and SM5310

Once printed, this is considered an uncontrolled document

Approvals (Signature/Date):

Lisa R Matecki 12/28/09
Lisa Matecki Date
Department Manager

Kenneth E. Kasperek 12/28/09
Kenneth Kasperek Date
Health & Safety Manager / Coordinator

Peggy Gray-Erdmann 12/28/09
Peggy Gray-Erdmann Date
Quality Assurance Manager

12/28/09
Christopher Spencer Date
Laboratory Director

Facility Distribution No. ___________ **Distributed To:**________________________

1.0 Scope and Application

1.1 Analytes, Matrix(s), and Reporting Limits

- This SOP was derived from EPA Method 415.1, Standard Methods 5310 and SW846 Third Edition Method 9060.
- Applicable matrices are all aqueous samples or solid wastes.
- The standard reporting limit is 1.0 mg/l.

On occasion clients may request modifications to this SOP. These modifications are handled following the procedures outlined in Section 10 in the Quality Assurance Manual.

2.0 Summary of Method

2.1 This method is applicable to all aqueous samples including ground, drinking, surface, and saline waters, as well as domestic and industrial wastes.

2.2 Solid waste may be analyzed for leachable Total Organic Carbon after first generating a leachate using the ASTM Shake Extraction Procedure.

2.3 Total Carbon (TC), Total Inorganic Carbon (TIC) and Total Organic Carbon (TOC) are all determined by wet oxidation. Each form of carbon is ultimately measured as carbon dioxide (CO_2) by a nondispersive infrared detector (NDIR) that has been calibrated to directly display the mass of CO_2 detected.

2.4 TIC is determined by measuring the carbon dioxide released by sample acidification. As the pH of the sample is lowered, carbonate and bicarbonate ions are converted to dissolved carbon dioxide. The dissolved carbon dioxide is carried into a NDIR calibrated to directly display the mass of carbon dioxide detected.

2.5 TOC is determined by measuring the carbon dioxide released by chemical oxidation of the organic carbon in the sample. After the sample has been acidified and purged of TIC, sodium persulfate, a strong oxidizer is added. This oxidant quickly reacts with organic carbon in the sample at 100°C to form carbon dioxide. When the oxidation reaction is complete, the carbon dioxide is purged from the solution and detected as described for TIC.

2.6 TOC is determined by measuring the carbon dioxide released by complete oxidation of all carbon present in the sample (inorganic and organic). For this analysis, first add acid and persulfate to the sample and allow a specific reaction time to convert all carbon present to carbon dioxide. When the reaction is complete, the resulting carbon dioxide is purged from the solution and detected as described for TIC.

2.7 Glassware for this analysis must be HCl washed.

3.0 Definitions

All definitions are consistent with those described by TestAmerica's Corporate Quality Assurance Management Plan.

3.1 Organic carbon: carbon present in the form of organic carbon-based compounds.

3.2 Inorganic carbon: carbon present in the form of inorganic compounds; usually carbon dioxide.

4.0 Interferences

Inorganic halides in samples compete with the organics for persulfate. The Model 1010 is able to analyze samples with up to 30 mg of chlorine without any modification. When samples contain over 30 mg of chlorine, additional persulfate reagent, increased TOC reaction time and a halide scrubber option are necessary.

5.0 Safety

Employees must abide by the policies and procedures in the Corporate Safety Manual, Radiation Safety Manual and this document.

This procedure may involve hazardous material, operations and equipment. This SOP does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of the method to follow appropriate safety, waste disposal and health practices under the assumption that all samples and reagents are potentially hazardous. Safety glasses, gloves, lab coats and closed-toe, nonabsorbent shoes are a minimum.

5.1 Specific Safety Concerns or Requirements

Sodium Persulfate is a strong oxidizer. Avoid contact with combustible materials, organic materials, strong reducing agents, and excess heat.

5.2 Primary Materials Used

The following is a list of the materials used in this method, which have a serious or significant hazard rating. **Note: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the MSDS for each of the materials listed in the table.** A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the MSDS for each material before using it for the first time or when there are major changes to the MSDS.

Material [1]	Hazards	Exposure Limit[2]	Signs and symptoms of exposure
Sulfuric Acid	Corrosive Oxidizer Dehydra-dator	1 mg/m^3	This material will cause burns if comes into contact with the skin or eyes. Inhalation of vapors will cause irritation of the nasal and respiratory system.
Phosphoric Acid	Corrosive	1 Mg/M3 TWA	Inhalation is not an expected hazard unless misted or heated to high temperatures. May cause redness, pain, and severe skin burns. May cause redness, pain, blurred vision, eye burns, and permanent eye damage.

Sodium Persulfate	Oxidizer Corrosive	0.1 Mg/M3-TWA as Persulfate	Causes irritation to the respiratory tract. Symptoms may include sore throat, shortness of breath, inflammation of nasal passages, coughing, and wheezing. Causes severe irritation or burns to the skin and eyes. Symptoms include redness, itching, pain and burns. May cause allergic skin reactions. Can cause eye damage.
[1] – Always add acid to water to prevent violent reactions.			
[2] – Exposure limit refers to the OSHA regulatory exposure limit.			

6.0 Equipment and Supplies

- Sample vials (40-mL vials with caps and septa).
- 100 mL and 1000 mL volumetric flasks
- Class A pipettes.
- Eppendorfs with a range of 100ul to 1000ul and 500ul to 5000ul.
- A 100 mL and a 50 mL graduated cylinder

6.1 Instrumentation

- O·I·Analytical Carbon Analyzer (Model 1010 and 1030) with corresponding autosampler

7.0 Reagents and Standards

7.1 Carbon-free mili-Q water used for initial calibration blanks (ICB), initial calibration verification (ICV), laboratory control samples (LCS), continuing calibration verifications (CCV), method blanks (MBLK) and the ERA to make up all standards or with any dilution a sample may need.

7.2 1000ppm Stock Standard KHP standard purchased from two separate vendors. ERA standard purchased from Environmental Resource Associates may be substituted for 60 PPM KHP standard and used for ICV when calibrating a new curve.

7.3 A 60.0PPM and a 30.0PPM initial calibration verification (ICV) and Laboratory Control Standard (LCS):

- Dilute 60.0-ml stock 1000PPM KHP STD to 1000 ml of mili-Q water in a volumetric flask
- Dilute 30.0-ml stock 1000PPM KHP STD to 1000 ml of mili-Q water in a volumetric flask

7.4 Matrix Spike Solution (MS): 880 ul of 1000 mg/l KHP STD is added to 44 mls of sample. The final expected concentration of spike in the sample is 20 mg/l. If a dilution is required, only 800 ul of 1000mg/l KHP STD is added to 40mls of sample.

7.5 Sodium Persulfate (20%) reagent- Pre-purchased from OI Analytical.

- If needed to make: add 400g of $Na_2S_2O_8$ to carbon free mili-Q water to create a total final reagent volume of 2 liter. Stirring may be necessary; do not heat. The shelf life for this solution is approximately three weeks. Solution made according to OI Operations Manual.

7.6 Phosphoric Acid (5%) reagent- Pre-purchased from OI Analytical.

- If needed to make: prepare a (5%) phosphoric acid solution by adding 118.0 ml of reagent grade (85%) H_3PO_4 to carbon free mili-Q water to create a total final volume of 2 liters. (caution; exothermic). Solution made according to OI Operations Manual.

7.7 UHP Nitrogen tanks (carrier gas).

8.0 Sample Collection, Preservation, Shipment and Storage

Sample container, preservation techniques and holding times may vary and are dependent on sample matrix, method of choice, regulatory compliance, and/or specific contract or client requests. Listed below are the holding times and the references that include preservation requirements.

Matrix	Sample Container	Min. Sample Size	Preservation	Holding Time	Reference
Water	Vials with caps and septa	40 mLs	HCL with a pH <2 and zero headspace; Cool 4 $\pm$ 2°C	28 Days	40 CFR 136.3 SW846 3rd edition Standard Methods 5310
Soil	Glass wide jar	4oz	Cool 4 $\pm$ 2°C		N/A

9.0 Quality Control

9.1 **Sample QC** - The following quality control samples are prepared with each batch of samples.

Quality Controls	Frequency	Control Limit
Method Blank (MBLK)	1 in 10 or fewer samples	< Rpt. Limit
Laboratory Control Sample (LCS)	1 in 10 or fewer samples	Statistical Limits [3]
Matrix Spike (MS)[1]	1 in 10 for method 415.1 1 in 20 for method 9060	Statistical Limits [3]
MS Duplicate (MSD)[1]	1 in 10 for method 415.1 1 in 20 for method 9060	Statistical Limits [3]

[1] The samples selected for MS/MSD are random, unless specifically requested by a client.
[2] Analytical and QC samples (MBLK, LCS, MS/MSD)
[3] Statistical control limits are updated annually and are updated into LIMS.

9.2 Instrument QC

9.2.1 Initial Calibration Verification (ICV) - Initial Calibration Verification: must be analyzed immediately after the calibration curve using a second source standard (separate from the curve) to verify that the calibration curve is acceptable.

9.2.2 Initial Calibration Blank (ICB) - Calibration blanks must be analyzed after every calibration curve.

9.2.3 Laboratory Control Sample (LCS) - must be analyzed at the beginning of every sequence, and for every 10 or fewer samples analyzed.

9.2.4 Method Blank (MBLK) - Method blanks must be analyzed at the beginning of every sequence, and for every 10 or fewer samples analyzed.

9.2.5 Matrix Duplicate (MD) and a sample Matrix Spike (MS) must be analyzed every 20 samples or fewer for method 415.1 and every 10 samples of fewer for method 9060.

- 3rd Edition and 40 CFR protocols require MD and MS every 20 samples.
- New York State ASP protocol requires MD and MS for every 10 samples.

Include a line for each appropriate parameter you used above.

Step	***Standards***	***Type***	***Control Limit***	***Frequency***
Initial Cal	0-50PPM for instrument 1010; and 0-100PPM for instrument 1030	LINEAR	R= <0.995	Minimum every 3 months
ICV	ERA	LINEAR	90-110%	EVERY 3 MONTHS
ICB	Milli-Q water	LINEAR	<1.0 mg/l	EVERY 3 MONTHS
LCS	60PPM	LINEAR	90-110%	EVERY 3 MONTHS
MBLK	Milli-Q water	LINEAR	<1.0 mg/l	Before a run and every 10 samples
MD	Sample	LINEAR	Statistically derived and stored in LIMS	9060: every 10 samples; 415.1/SM5310: every 20 samples
MS	20PPM	LINEAR	Statistically derived and stored in LIMS	9060: every 10 samples; 415.1/SM5310: every 20 samples

A calibration curve may be fitted to the calibration solution concentration/response data using the computer. Acceptance or control limits should be established using the difference between the measured values of the calibration solution and the “true value” concentration.

10.0 Procedure

- The autosampler carousel is capable of holding up to 51 sample vials for instrument 1010 and 87 samples for instrument 1030. Place LCS's, Blanks, MS, MD and samples in appropriate slots in the carousel. It is recommended that 5 reagent blanks be run at the beginning of each analytical run as an instrument clean up.

- The autosampler carousel is secured to the autosampler deck and covered with the carousel cover. (Note: the carousel cover must be in place to operate the autosampler.)

- Set up the analytical sequence by entering the number of samples and number of injections per sample (consult the instrument software manual for details).

  - Method 415.1 requires two injections with an MD and MS every 20 samples.

  -Method 9060 requires four injections with an MD and MS every 10 samples.

- Activate "Start" from the software menu (consult the instrument software manual for details). A printout of results will follow. (Consult O·I's Technical manuals for software specific functionality).

- Dilute any samples that have an excessive amount of particles with mili-Q water, as to not clog the needle and contaminate the tubing within the system. (Note dilution on run log).

- Leachable TOC samples are prepared using ASTM Leaching procedure. The extract is transferred into a pre-preserved HCL vial with a septa and analyzed on the instrument.
- Dry weights must be performed on all soil samples to adjust the final reported concentration.
- The pH must be checked after the analytical sequence has been completed on each sample that has been analyzed past 48 hours from sampling. If a sample is analyzed within 48 hours of being sampled, it is unnecessary to do a pH check on it. This is documented on the analytical run sequence sheets for every batch.

10.1 Calibration

Before any instrument is used as a measurement device, the instrument response to known reference materials must be determined. The manner in which various instruments are calibrated depends on the particular type of instrument and its intended use. All sample measurements must be made within the calibration range of the instrument. Preparation of all reference materials used for calibration must be documented.

- Prepare calibration standards by diluting the following volumes of 1,000 mg/l KHP STD with 100 ml carbon free mili-Q water in a 100ml volumetric flask. Prepare standards as indicated in the following table. The Control Limit for all area counts on a curve need to be $\leq$ 20 RSD

Instrument 1030 Calibration:

Concentration	Volume
100.0 mg/l	10.0 ml 1,000mg/l KHP STD dilute up to 100 ml with carbon free mili-Q water
50.0 mg/l	5.0 ml 1,000mg/l KHP STD dilute up to 100 ml with carbon free mili-Q water.
10.0 mg/l	1.0 ml 1,000mg/l KHP STD dilutes up to 100 ml with carbon free mili-Q water.
5.0 mg/l	0.5 ml 1,000mg/l KHP STD dilute up to 100 ml with carbon free mili-Q water.
1.0 mg/l	0.1 ml 1,000mg/l KHP STD dilute up to 100 ml with carbon free mili-Q water.
0 mg/l	100ml Carbon free mili-Q water

Instrument 1010 Calibration:

Concentration	Volume
50.0 mg/l	5.0 ml 1,000mg/l KHP STD dilute up to 100 ml with carbon free mili-Q water.
25.0 mg/l	2.5 ml 1,000mg/l KHP STD dilutes up to 100 ml with carbon free mili-Q water.
5.0 mg/l	0.5 ml 1,000mg/l KHP STD dilute up to 100 ml with carbon free mili-Q water.
1.0 mg/l	0.1 ml 1,000mg/l KHP STD dilute up to 100 ml with carbon free mili-Q water.
0 mg/l	100ml Carbon free mili-Q water

11.0 Calculations / Data Reduction

If the result of any injection exceeds the linear range of the calibration, the sample must be re-analyzed using a dilution.

11.1 Leachable TOC samples and soil QC are calculated in AIMS:

$$\frac{\text{TOC result (mg/l)}}{\text{Weight of the sample (g)}} \times \text{final volume (soil and de ionized water) (ml)}$$

Then dry weight correct this calculated result. Result is divided by Dry Weight.

11.2 ICV / CCV, LCS % Recovery $= \dfrac{\text{observed concentration}}{\text{known concentration}} \times 100$

11.3 MS % Recovery $= \dfrac{\text{(spiked sample) - (unspiked sample)}}{\text{spiked concentration}} \times 100$

11.4 Precision (RPD) $= \dfrac{|x_1 - x_2|}{\left(\dfrac{x_1 + x_2}{2}\right)} \times 100$

11.5 Matrix Duplicate (MD) $= \dfrac{|\text{orig. sample value - dup. sample value}|}{[(\text{orig. sample value + dup. sample value})/2]} \times 100$

11.6 Concentration = mg/kg or L $= \dfrac{C \times V \times D}{W}$

Where:
C = sample concentration in extract (ppm)
V = Volume of extract (mL)
D = Dilution Factor
W = Weight/Volume of sample aliquot extracted (grams or mLs)

NOTE: All dry weight corrections are made in LIMS at the time the final report is prepared.

12.0 Method Performance

12.1 Method Detection Limit Study (MDL)

Method Detection Limit: A valid method detection limit for each analyte of interest must be generated. The MDL must be below the reporting limit for each analyte. The procedure for determination of the method detection limit is given in 40 CFR Part 136, Appendix B. See TestAmerica's, "Method Detection Limit Studies," current revision, for further guidance. Current TA Buffalo MDLs are maintained the QA department and are easily viewed in the laboratory LIMs system.

12.2 Demonstration of Capabilities

A one–time initial demonstration of performance for each individual method for both soils and water matrices must be generated.

12.2.1 This requires quadruplicate analysis of a mid–level check standard containing all of the standard analytes for the method using the same procedures used to analyze samples, including sample preparation.

12.2.2 Calculate the average recovery and standard deviation of the recovery for each analyte of interest.

12.2.3 Compare these results with the acceptance criteria given in the Method or to laboratory historical limits (if available).

12.2.4 Repeat the test for any analyte that does not meet the acceptance criteria. Repeated failure for any analyte indicates the need for the laboratory to evaluate the analytical procedure and take corrective action.

12.3 Training Requirements

- The supervisor has the responsibility to ensure that an analyst who has been properly trained in its use and has the required experience performs this procedure.
- The following analyst validation information is maintained for this method in the laboratory QA files.
- The analyst must complete the laboratory safety orientation training that includes, but is not limited to, chemicals, PPE requirements, and electrical safety.
- The analyst must read and understand this SOP.
- The analyst must read and understand the Method used as reference for this SOP.
- The analyst must complete a DOC or successfully analyze PT samples annually.
- The analyst must complete the TA Quality Assurance Training.

13.0 Pollution Control

It is TestAmerica's policy to evaluate each method and look for opportunities to minimize waste generated (i.e., examine recycling options, ordering chemicals based on quantity needed, preparation of reagents based on anticipated usage and reagent stability).

14.0 Waste Management

All waste will be disposed of in accordance with Federal, State and Local regulations. Where reasonably feasible, technological changes have been implemented to minimize the potential for pollution of the environment. Employees will abide by this method and the policies in section 13 of the Corporate Safety Manual for "Waste Management and Pollution Prevention."

The following waste streams are produced when this method is carried out.

-Acidic waste from the auto-analyzer must be disposed of in the "A" waste container.

15.0 References / Cross-References

15.1 Standard Methods for the Examination of Water and Wastewater, 18th Edition, American Public Health Association/ American Water Works/ Water Environment Federation, Washington, DC.

15.2 Method 415.1, "Methods for Chemical Analysis of Water and Wastes", U.S. Environmental Protection Agency, Environmental Monitoring and Support Laboratory, Revised March 1983

15.3 Method 9060, "Test Methods for Evaluating Solid Waste"; SW-846, Third Edition, 12/96.

16.0 Method Modifications:

Item	Method	Modification
1	*9060* *SM5310* *415.1*	*Reagents in section 7.5 and 7.6 made according to instrument(OI analytical) operational manual*

17.0 Attachments

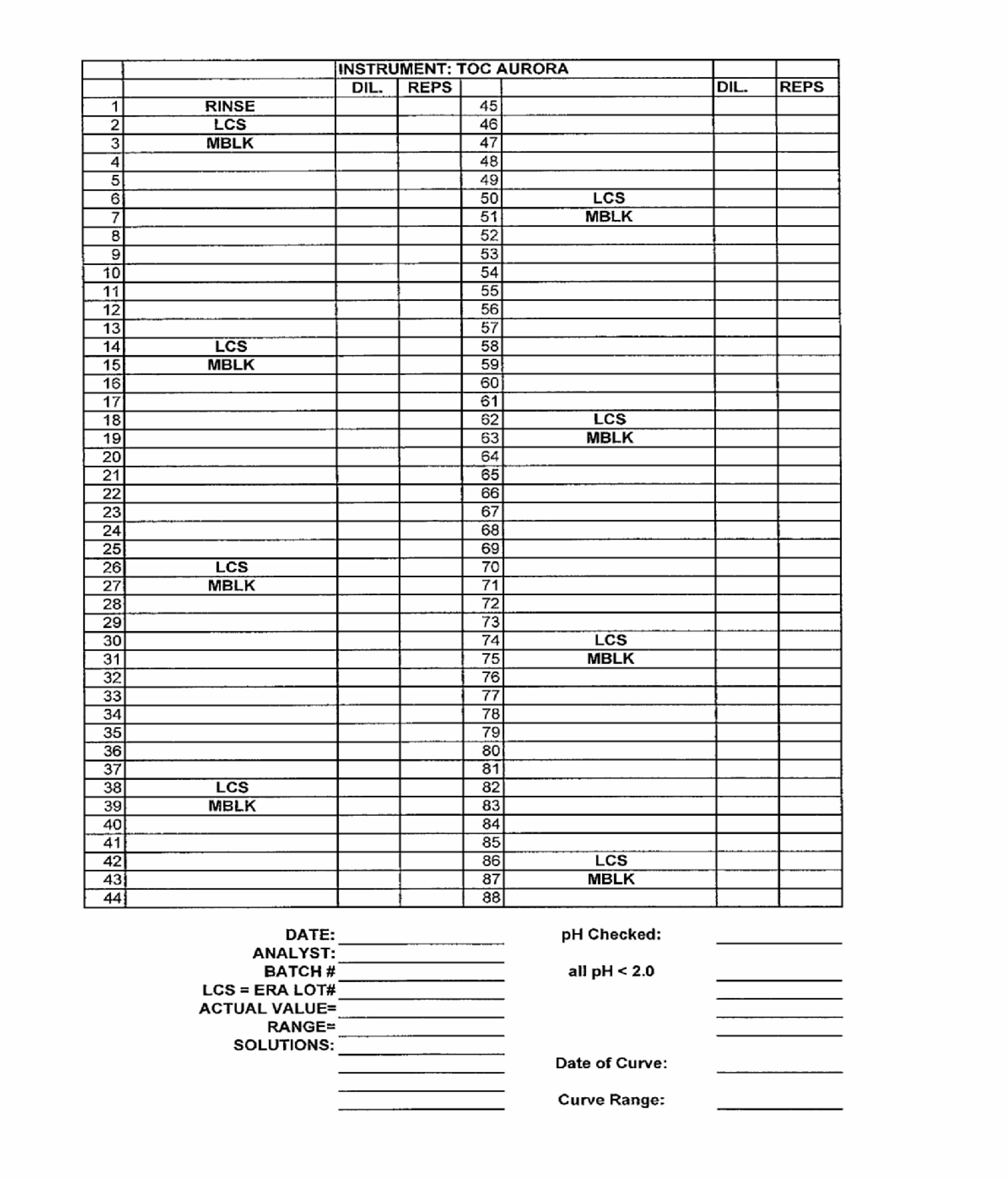
17.1 Analytical Run Sequence for Instrument 1030
17.2 Analytical Run Sequence for Instrument 1010
17.3 Analytical Batch for Instrument 1030
17.4 Analytical Batch for Instrument 1010
17.5 Wet Chemistry Batch Summary and Data Review Checklist

18.0 Revision History

- Revision 1, dated 31 December 2009
  - Updated Sodium Persulfate reagent, 15% to 20%
  - Updated attachments
- Revision 0, dated 19 December, 2007
  - Integration for TestAmerica and STL operations.

- Updated attachments
- Added pH check and documentation to section 10.0

Attachment 1
Analytical Run Sequence for the 1030



		INSTRUMENT: TOC AURORA					
		DIL.	REPS			DIL.	REPS
1	RINSE			45			
2	LCS			46			
3	MBLK			47			
4				48			
5				49			
6				50	LCS		
7				51	MBLK		
8				52			
9				53			
10				54			
11				55			
12				56			
13				57			
14	LCS			58			
15	MBLK			59			
16				60			
17				61			
18				62	LCS		
19				63	MBLK		
20				64			
21				65			
22				66			
23				67			
24				68			
25				69			
26	LCS			70			
27	MBLK			71			
28				72			
29				73			
30				74	LCS		
31				75	MBLK		
32				76			
33				77			
34				78			
35				79			
36				80			
37				81			
38	LCS			82			
39	MBLK			83			
40				84			
41				85			
42				86	LCS		
43				87	MBLK		
44				88			

DATE: ____________________

ANALYST: ____________________

BATCH # ____________________

LCS = ERA LOT# ____________________

ACTUAL VALUE= ____________________

RANGE= ____________________

SOLUTIONS: ____________________

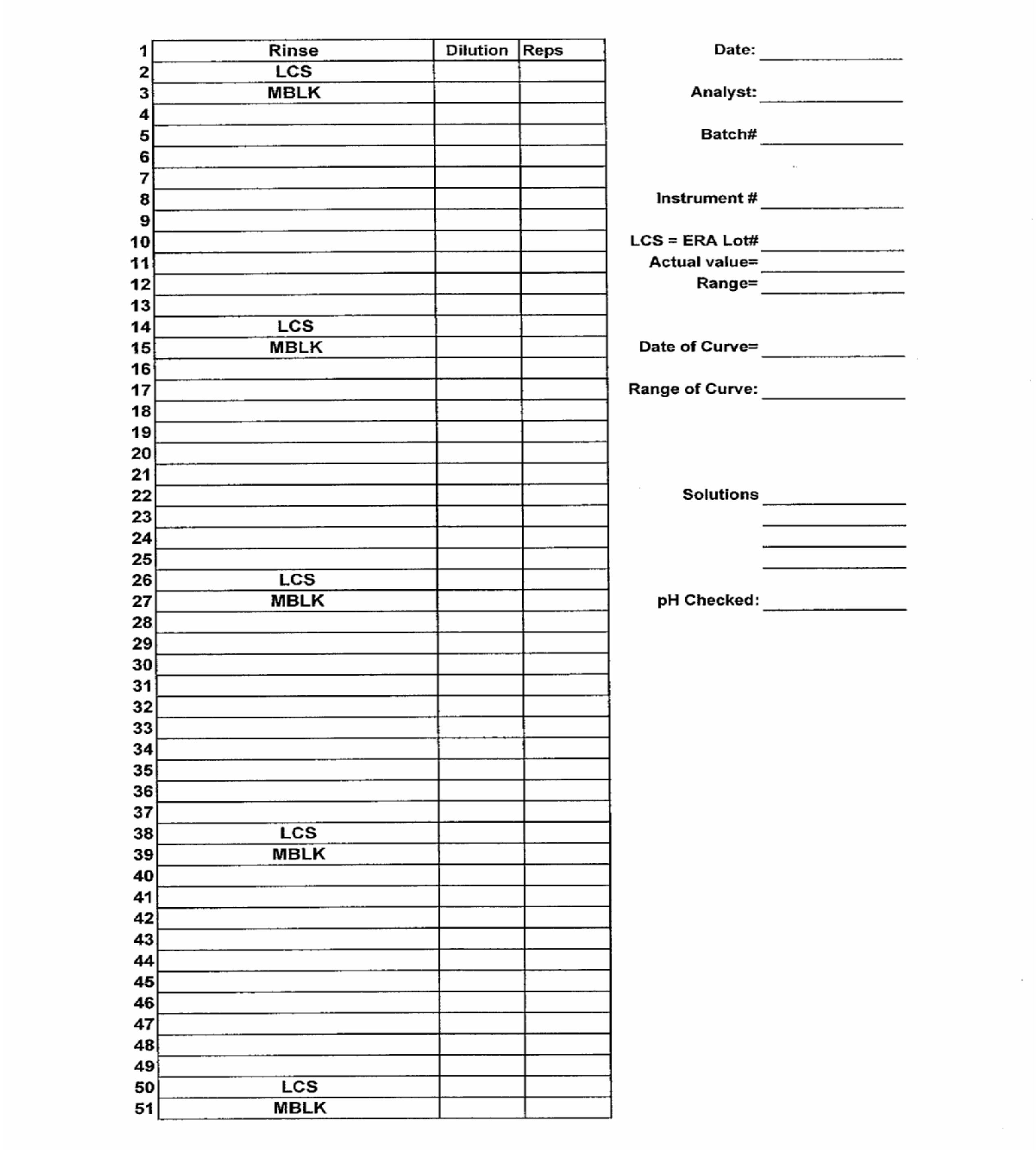
pH Checked: ______________

all pH < 2.0 ______________

Date of Curve: ______________

Curve Range: ______________

Attachment 2
Analytical Run Sequence for Instrument 1010



1	Rinse	Dilution	Reps
2	LCS		
3	MBLK		
4			
5			
6			
7			
8			
9			
10			
11			
12			
13			
14	LCS		
15	MBLK		
16			
17			
18			
19			
20			
21			
22			
23			
24			
25			
26	LCS		
27	MBLK		
28			
29			
30			
31			
32			
33			
34			
35			
36			
37			
38	LCS		
39	MBLK		
40			
41			
42			
43			
44			
45			
46			
47			
48			
49			
50	LCS		
51	MBLK		

Date: ________________

Analyst: ________________

Batch# ________________

Instrument # ________________

LCS = ERA Lot# ________________
Actual value= ________________
Range= ________________

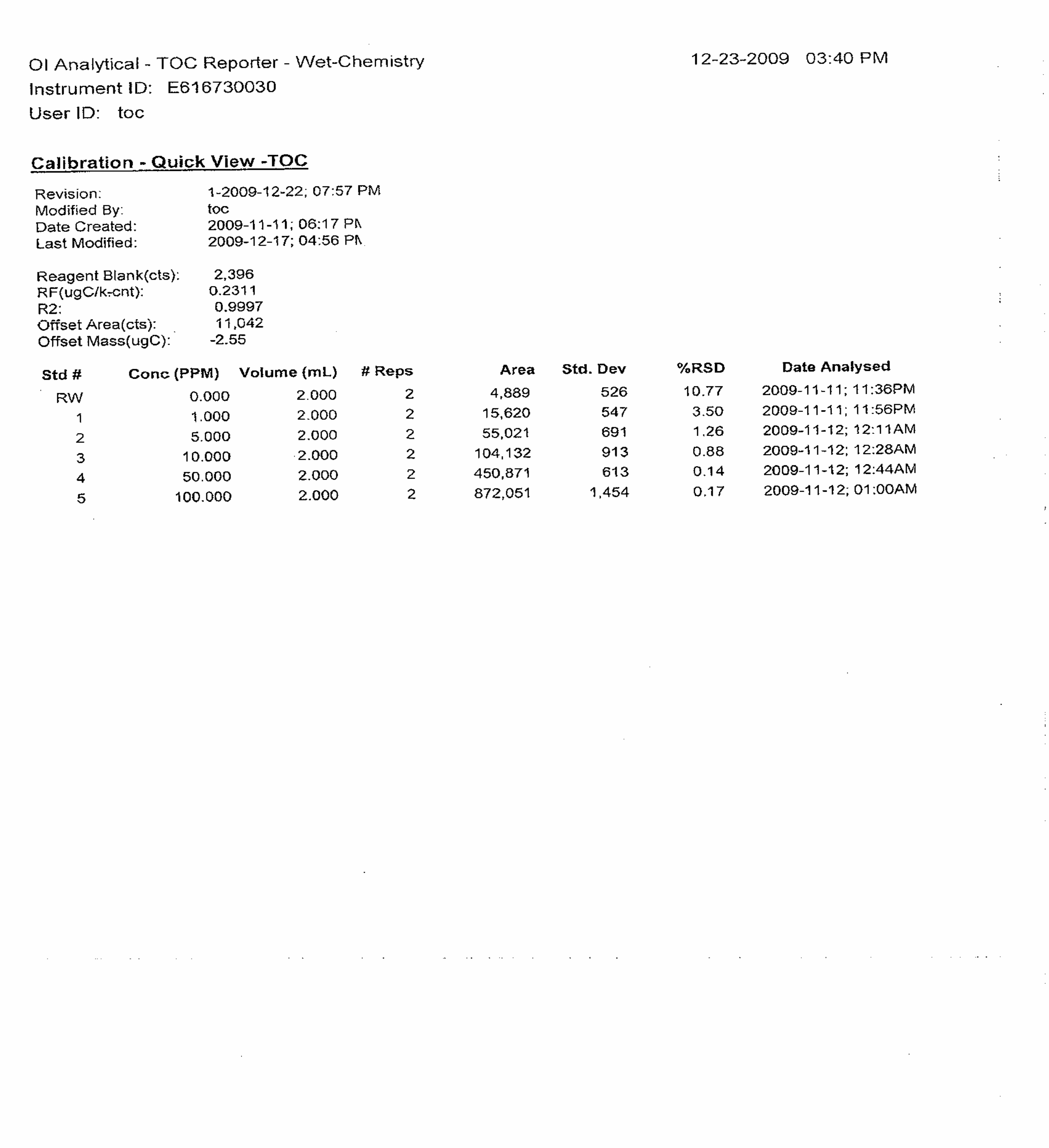
Date of Curve= ________________

Range of Curve: ________________

Solutions ________________
____
____
____

pH Checked: ________________

Attachment 3
Analytical Batch for instrument 1030



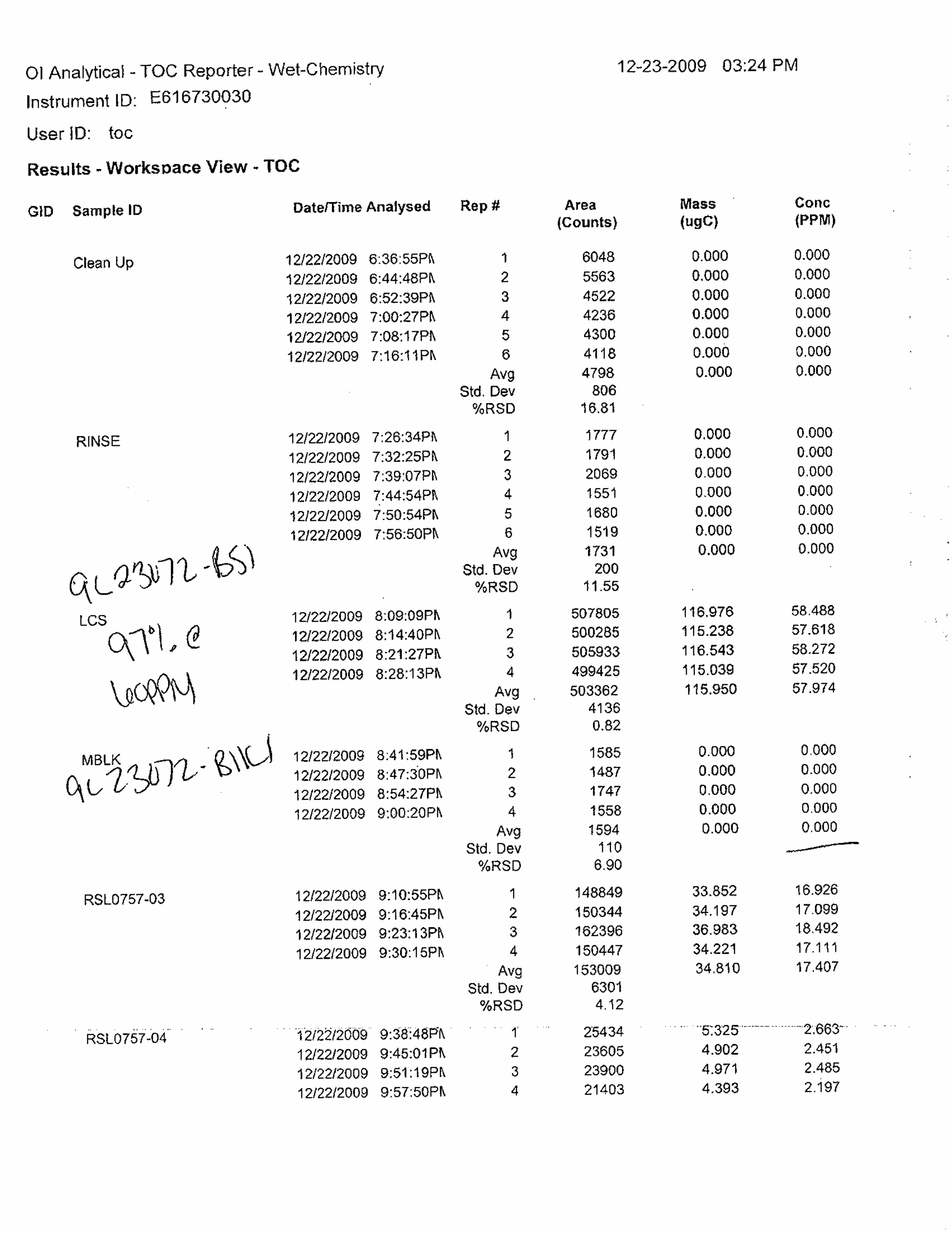
OI Analytical - TOC Reporter - Wet-Chemistry 12-23-2009 03:40 PM
Instrument ID: E616730030
User ID: toc

Calibration - Quick View -TOC

Revision:	1-2009-12-22; 07:57 PM
Modified By:	toc
Date Created:	2009-11-11; 06:17 PM
Last Modified:	2009-12-17; 04:56 PM

Reagent Blank(cts):	2,396
RF(ugC/k-cnt):	0.2311
R2:	0.9997
Offset Area(cts):	11,042
Offset Mass(ugC):	-2.55

Std #	Conc (PPM)	Volume (mL)	# Reps	Area	Std. Dev	%RSD	Date Analysed
RW	0.000	2.000	2	4,889	526	10.77	2009-11-11; 11:36PM
1	1.000	2.000	2	15,620	547	3.50	2009-11-11; 11:56PM
2	5.000	2.000	2	55,021	691	1.26	2009-11-12; 12:11AM
3	10.000	2.000	2	104,132	913	0.88	2009-11-12; 12:28AM
4	50.000	2.000	2	450,871	613	0.14	2009-11-12; 12:44AM
5	100.000	2.000	2	872,051	1,454	0.17	2009-11-12; 01:00AM

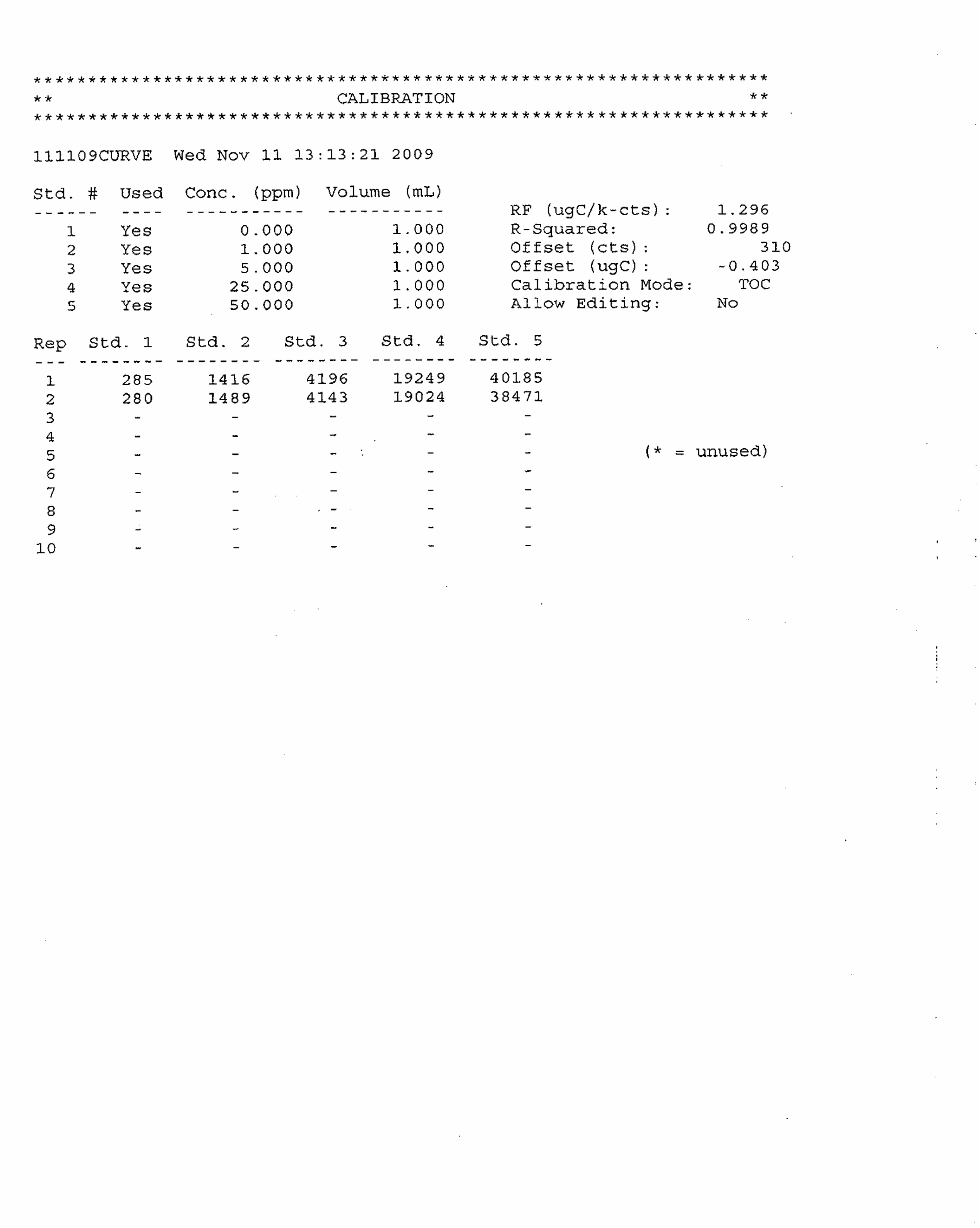


OI Analytical - TOC Reporter - Wet-Chemistry 12-23-2009 03:24 PM
Instrument ID: E616730030
User ID: toc

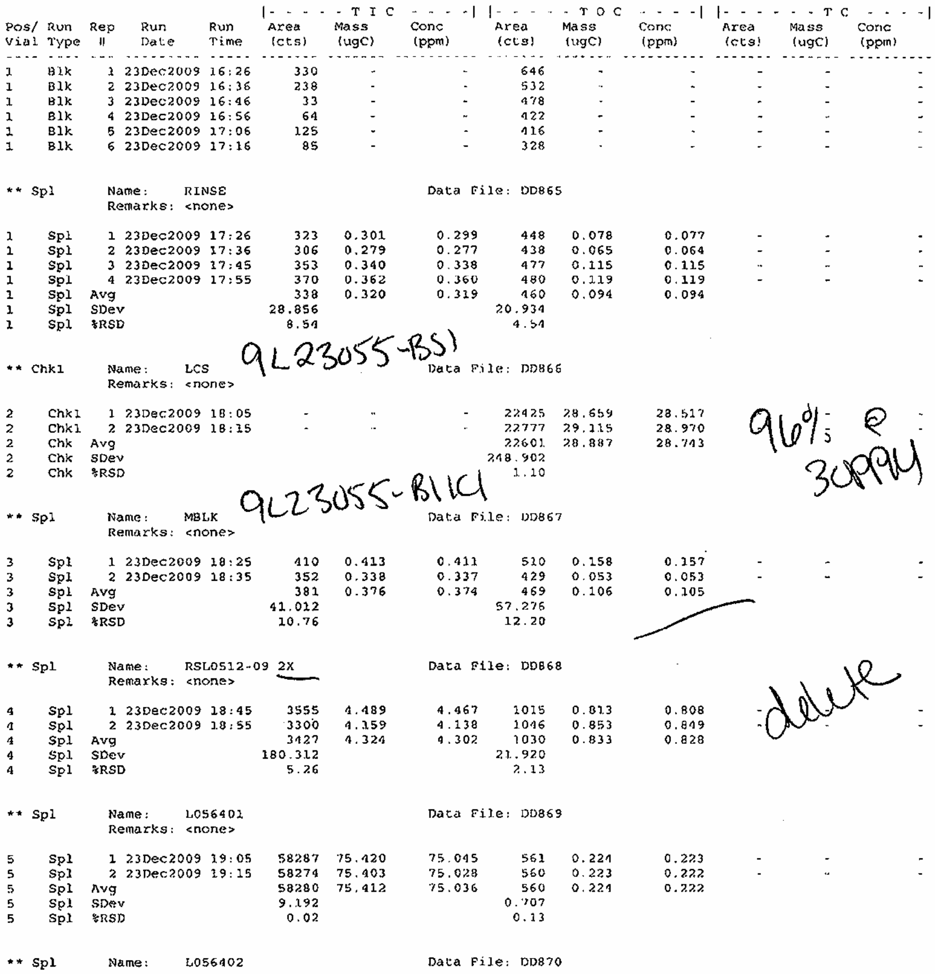
Results - Workspace View - TOC

GID	Sample ID	Date/Time Analysed	Rep #	Area (Counts)	Mass (ugC)	Conc (PPM)
	Clean Up	12/22/2009 6:36:55PN	1	6048	0.000	0.000
		12/22/2009 6:44:48PN	2	5563	0.000	0.000
		12/22/2009 6:52:39PN	3	4522	0.000	0.000
		12/22/2009 7:00:27PN	4	4236	0.000	0.000
		12/22/2009 7:08:17PN	5	4300	0.000	0.000
		12/22/2009 7:16:11PN	6	4118	0.000	0.000
			Avg	4798	0.000	0.000
			Std. Dev	806		
			%RSD	16.81		
	RINSE	12/22/2009 7:26:34PN	1	1777	0.000	0.000
		12/22/2009 7:32:25PN	2	1791	0.000	0.000
		12/22/2009 7:39:07PN	3	2069	0.000	0.000
		12/22/2009 7:44:54PN	4	1551	0.000	0.000
		12/22/2009 7:50:54PN	5	1680	0.000	0.000
		12/22/2009 7:56:50PN	6	1519	0.000	0.000
			Avg	1731	0.000	0.000
	9C23072-BS1		Std. Dev	200		
			%RSD	11.55		
	LCS 97% @ 60PPM	12/22/2009 8:09:09PN	1	507805	116.976	58.488
		12/22/2009 8:14:40PN	2	500285	115.238	57.618
		12/22/2009 8:21:27PN	3	505933	116.543	58.272
		12/22/2009 8:28:13PN	4	499425	115.039	57.520
			Avg	503362	115.950	57.974
			Std. Dev	4136		
			%RSD	0.82		
	MBLK 9C23072-BLK1	12/22/2009 8:41:59PN	1	1585	0.000	0.000
		12/22/2009 8:47:30PN	2	1487	0.000	0.000
		12/22/2009 8:54:27PN	3	1747	0.000	0.000
		12/22/2009 9:00:20PN	4	1558	0.000	0.000
			Avg	1594	0.000	0.000
			Std. Dev	110		
			%RSD	6.90		
	RSL0757-03	12/22/2009 9:10:55PN	1	148849	33.852	16.926
		12/22/2009 9:16:45PN	2	150344	34.197	17.099
		12/22/2009 9:23:13PN	3	162396	36.983	18.492
		12/22/2009 9:30:15PN	4	150447	34.221	17.111
			Avg	153009	34.810	17.407
			Std. Dev	6301		
			%RSD	4.12		
	RSL0757-04	12/22/2009 9:38:48PN	1	25434	5.325	2.663
		12/22/2009 9:45:01PN	2	23605	4.902	2.451
		12/22/2009 9:51:19PN	3	23900	4.971	2.485
		12/22/2009 9:57:50PN	4	21403	4.393	2.197

Attachment 4:
Analytical Batch for Instrument 1010



```
*******************************************************************
**                         CALIBRATION                           **
*******************************************************************

111109CURVE  Wed Nov 11 13:13:21 2009

Std. #  Used  Conc. (ppm)  Volume (mL)
------  ----  -----------  -----------     RF (ugC/k-cts):      1.296
   1    Yes         0.000        1.000     R-Squared:          0.9989
   2    Yes         1.000        1.000     Offset (cts):          310
   3    Yes         5.000        1.000     Offset (ugC):       -0.403
   4    Yes        25.000        1.000     Calibration Mode:     TOC
   5    Yes        50.000        1.000     Allow Editing:       No

Rep  Std. 1   Std. 2   Std. 3   Std. 4   Std. 5
--- -------- -------- -------- -------- --------
 1      285     1416     4196    19249    40185
 2      280     1489     4143    19024    38471
 3        -        -        -        -        -
 4        -        -        -        -        -
 5        -        -        -        -        -          (* = unused)
 6        -        -        -        -        -
 7        -        -        -        -        -
 8        -        -        -        -        -
 9        -        -        -        -        -
10        -        -        -        -        -
```



```
                                      |- - - - - T I C  - - - -| |- - - - - T O C  - - - -| |- - - - - - T C  - - - -|
Pos/ Run  Rep    Run      Run     Area    Mass      Conc      Area    Mass      Conc      Area    Mass     Conc
Vial Type  #     Date     Time    (cts)   (ugC)     (ppm)     (cts)   (ugC)     (ppm)     (cts)   (ugC)    (ppm)
---- ---- --- --------- ----- ------- ------- ---------- ------- ------- ---------- ------- ------- ---------
1    Blk    1 23Dec2009 16:26     330      -          -        646      -          -        -       -          -
1    Blk    2 23Dec2009 16:36     238      -          -        532      -          -        -       -          -
1    Blk    3 23Dec2009 16:46      33      -          -        478      -          -        -       -          -
1    Blk    4 23Dec2009 16:56      64      -          -        422      -          -        -       -          -
1    Blk    5 23Dec2009 17:06     125      -          -        416      -          -        -       -          -
1    Blk    6 23Dec2009 17:16      85      -          -        328      -          -        -       -          -


** Spl       Name:     RINSE                      Data File: DD865
             Remarks: <none>

1    Spl    1 23Dec2009 17:26     323   0.301      0.299       448   0.078      0.077      -       -          -
1    Spl    2 23Dec2009 17:36     306   0.279      0.277       438   0.065      0.064      -       -          -
1    Spl    3 23Dec2009 17:45     353   0.340      0.338       477   0.115      0.115      -       -          -
1    Spl    4 23Dec2009 17:55     370   0.362      0.360       480   0.119      0.119      -       -          -
1    Spl   Avg                    338   0.320      0.319       460   0.094      0.094
1    Spl   SDev                28.856                       20.934
1    Spl   %RSD                  8.54                         4.54

** Chk1      Name:     LCS                        Data File: DD866
             Remarks: <none>

2    Chk1   1 23Dec2009 18:05       -      -          -      22425  28.659     28.517      -       -          -
2    Chk1   2 23Dec2009 18:15       -      -          -      22777  29.115     28.970      -       -          -
2    Chk   Avg                                               22601  28.887     28.743
2    Chk   SDev                                            248.902
2    Chk   %RSD                                               1.10

** Spl       Name:     MBLK                       Data File: DD867
             Remarks: <none>

3    Spl    1 23Dec2009 18:25     410   0.413      0.411       510   0.158      0.157      -       -          -
3    Spl    2 23Dec2009 18:35     352   0.338      0.337       429   0.053      0.053      -       -          -
3    Spl   Avg                    381   0.376      0.374       469   0.106      0.105
3    Spl   SDev                41.012                       57.276
3    Spl   %RSD                 10.76                        12.20

** Spl       Name:     RSL0512-09 2X              Data File: DD868
             Remarks: <none>

4    Spl    1 23Dec2009 18:45    3555   4.489      4.467      1015   0.813      0.808      -       -          -
4    Spl    2 23Dec2009 18:55    3300   4.159      4.138      1046   0.853      0.849      -       -          -
4    Spl   Avg                   3427   4.324      4.302      1030   0.833      0.828
4    Spl   SDev               180.312                       21.920
4    Spl   %RSD                  5.26                         2.13

** Spl       Name:     L056401                    Data File: DD869
             Remarks: <none>

5    Spl    1 23Dec2009 19:05   58287  75.420     75.045       561   0.224      0.223      -       -          -
5    Spl    2 23Dec2009 19:15   58274  75.403     75.028       560   0.223      0.222      -       -          -
5    Spl   Avg                  58280  75.412     75.036       560   0.224      0.222
5    Spl   SDev                 9.192                        0.707
5    Spl   %RSD                  0.02                         0.13

** Spl       Name:     L056402                    Data File: DD870
```

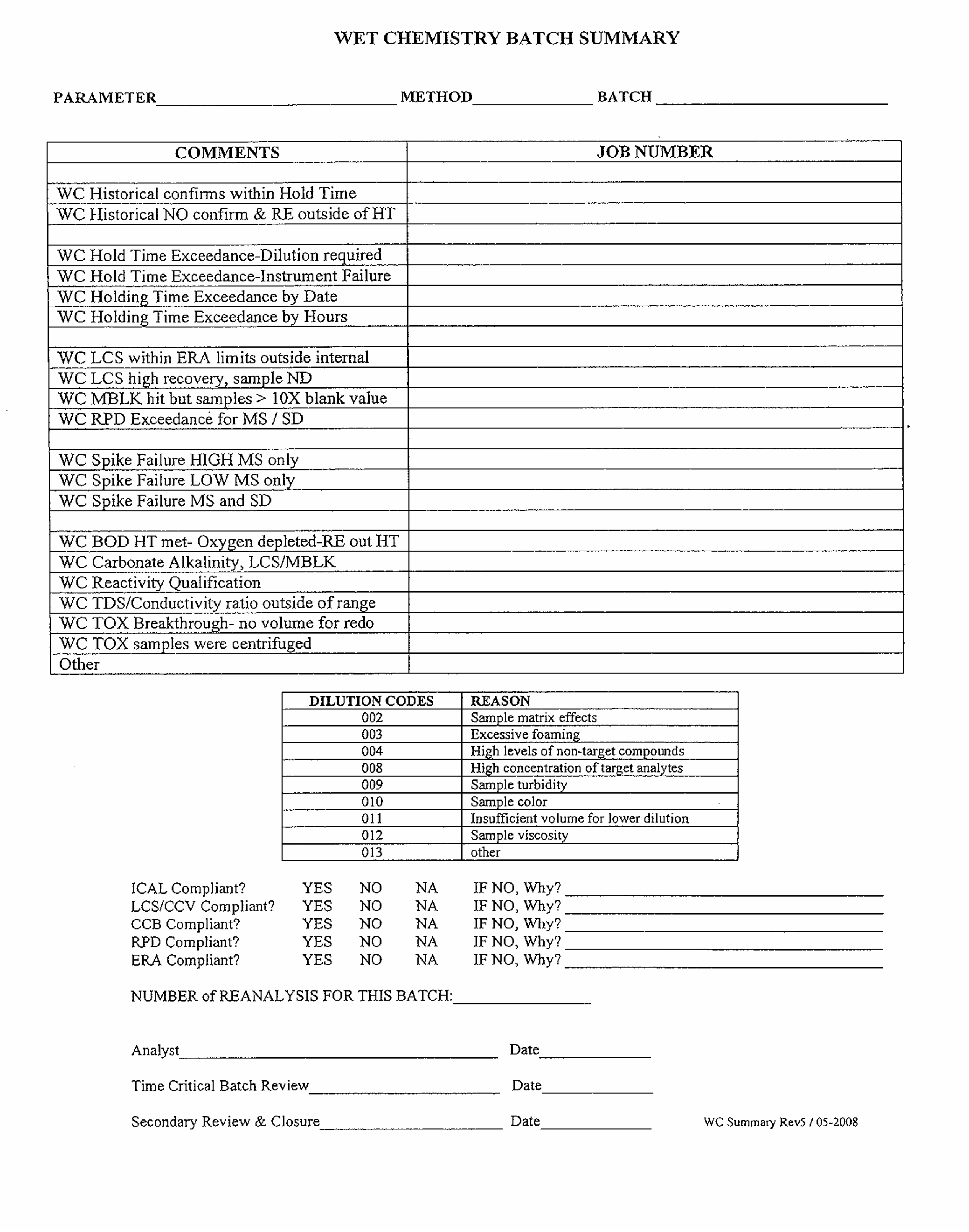
9L23055-BS1

96% @ 30ppm

9L23055-B1K1

dilute

Attachment 5
Wet Chemistry Batch Summary and Data Review Checklist



WET CHEMISTRY BATCH SUMMARY

PARAMETER______________________________ METHOD_______________ BATCH _____________________________

COMMENTS	JOB NUMBER
WC Historical confirms within Hold Time	
WC Historical NO confirm & RE outside of HT	
WC Hold Time Exceedance-Dilution required	
WC Hold Time Exceedance-Instrument Failure	
WC Holding Time Exceedance by Date	
WC Holding Time Exceedance by Hours	
WC LCS within ERA limits outside internal	
WC LCS high recovery, sample ND	
WC MBLK hit but samples > 10X blank value	
WC RPD Exceedance for MS / SD	
WC Spike Failure HIGH MS only	
WC Spike Failure LOW MS only	
WC Spike Failure MS and SD	
WC BOD HT met- Oxygen depleted-RE out HT	
WC Carbonate Alkalinity, LCS/MBLK	
WC Reactivity Qualification	
WC TDS/Conductivity ratio outside of range	
WC TOX Breakthrough- no volume for redo	
WC TOX samples were centrifuged	
Other	

DILUTION CODES	REASON
002	Sample matrix effects
003	Excessive foaming
004	High levels of non-target compounds
008	High concentration of target analytes
009	Sample turbidity
010	Sample color
011	Insufficient volume for lower dilution
012	Sample viscosity
013	other

ICAL Compliant? YES NO NA IF NO, Why? ____
LCS/CCV Compliant? YES NO NA IF NO, Why? ____
CCB Compliant? YES NO NA IF NO, Why? ____
RPD Compliant? YES NO NA IF NO, Why? ____
ERA Compliant? YES NO NA IF NO, Why? ____

NUMBER of REANALYSIS FOR THIS BATCH:_________________

Analyst____ Date______________

Time Critical Batch Review________________________ Date______________

Secondary Review & Closure_______________________ Date______________

WC Summary Rev5 / 05-2008

TestAmerica
THE LEADER IN ENVIRONMENTAL TESTING

SOPInterimChange
Rev.1
11/6/07

TestAmerica Buffalo
SOP Interim Change Form

Once printed, this is considered an uncontrolled document.

OP Number: BF-OP-001	**IC Number:**

OP Title: Toxicity Characteristic Leaching Procedure (TCLP) EPA Method 1311

OP Sections Affected by Change: 10.1.3

eason for Addition or Change:

f samples are not 100% dry the multi-phase spreadsheet must be used.

se the following procedure in conjunction with tab 2 of the excel spreadsheet.

he samples are filtered under pressure through glass Microfiber filters.
rior to assembly of the filtration apparatus, clean all parts by washing with soapy water followed by rinsing with eionized water.

ollow all steps listed in the excel spreadsheet. Cells in green need to be filled in. Cells in orange are calculations or onstants. Cells in yellow are informational.

ecord weight of empty container used to collect the liquid phase from sample. Label container with job number, and iquid phase. This volume is saved for recombination after tumbling.

ou will be filtering the entire sample volume, so check to make sure there are no other analysis assigned.

ecord the weight of the sample added to the pressure filtration unit.

erify that the empty container is in place to collect the liquid that is being filtered out. Transfer sample to filtration unit, aking care to spread the sample evenly over the filter paper.

radually apply pressure until liquid flow has ceased and pressurizing gas moves through the filter. Do not exceed 50 si.

ecord the weight of the liquid filtered and the container in the spreadsheet. Record the pH reading of the liquid filtered

he spreadsheet automatically calculates the % Solids. The Weight of Dry sample after pressure filtration gives an pproximate weight of the amount of soil remaining on the filter paper. Use this information to determine if there is nough solid material to set the sample (100 gram minimum for Metals and Extractable, 25 gram minimum for Volatile).

Continue with procedure as outlined in sections 10.1.4-10.1.9.

After 18 +/- 2 hours of tumbling record tumbler end time, and min/max temperature readings. Remove samples from the umbler. Test a small aliquot of the liquid portion filtered before tumbling and the liquid from the tumbled portion for niscibility.
f samples are incompatible, they can not be recombined, and must be relinquished as individual samples.

f samples are compatible, filter the tumbled portion with regards to the analysis assigned. (Metals samples are filtered hrough nitric rinsed filter paper, Extractable samples are filtered through unpreserved filter paper.)

Record the volume of filtered sample. The spreadsheet automatically calculates how much liquid filtered using pressure filtration to add to the liquid filtered from the tumbled portion.

Submitted By: Shanna Snider Date: 11-20-09

APPROVED BY:

Department Supervisor: Shanna Snider Date: 11-20-09

QA Manager: B Gray-Edwards Date: 12/15/09

Laboratory Manager: Jennifer Pierce Date: 12/15/09

Laboratory Director: Date: 12/15/09

TestAmerica

THE LEADER IN ENVIRONMENTAL TESTING

TestAmerica Buffalo
SOP Interim Change Form

SOP Number: BF-OP-001

IC Number:

SOP Title: Toxicity Characteristic Leaching Procedure (TCLP) EPA Method 1311

SOP Sections Affected by Change: 10.1.10, 10.1.11

Reason for Addition or Change:

If samples are mixed phase, use Multi-phase tab on TCLP spreadsheets.

For TCLP Metals/Extractables: Record weight of empty filtrate container (record in spreadsheet). Add 100 grams of mixed phase sample to filtration unit (weight of Wet + Dry sample in spreadsheet). Gradually increase pressure until all liquid is filtered from sample. Weigh Liquid + Container and record in spreadsheet. The spreadsheet will automatically calculate the % solids, weight of the dry sample, and the appropriate amount of extraction fluid to add. Tumble the solid portion for 18 +/- 2 hours, store the liquid portion for evaluation after the solid portion has tumbled. After tumbling test a small portion of both liquids for compatibility. If compatible, combine filtered liquid portion with tumbled portion. Filter entire volume following filtration procedures outlined in section 10.1.9. If incompatible, relinquish both portions to analytical departments for analysis.

For VOA TCLP: All filtration is to be done in ZHE unit used to set the sample in.

Record weight of empty filtrate container (use a VOA vial to collect) in spreadsheet. Add 25 grams of mixed phase sample to filtration unit (weight of Wet + Dry sample in spreadsheet). Gradually increase pressure until all liquid is filtered from sample. Weigh Liquid + Container and record in spreadsheet. The spreadsheet will automatically calculate the % solids, weight of the dry sample, and the appropriate amount of extraction fluid to add. Tumble the solid portion for 18 +/- 2 hours, store the liquid portion for evaluation after the solid portion has tumbled. After tumbling test a small portion of both liquids for compatibility. Take care to minimize exposure of either portion to air to reduce loss of target analytes. If compatible, combine filtered liquid portion with tumbled portion. If incompatible, relinquish both portions to analytical departments for analysis.

TestAmerica

THE LEADER IN ENVIRONMENTAL TESTING

Submitted By:_Shanna Snider

Date: 5-8-09

APPROVED BY:

Department Supervisor: Shanna Snider Date: 5/8/09

QA Manager: Date: 6/28/09

Laboratory Manager: Date: 6/29/09

Laboratory Director: Date: 6/29/09

Toxicity Characteristic Leaching Procedure (TCLP)
EPA Method 1311

Once printed, this is considered an uncontrolled document

Approvals (Signature/Date):

Shanna Snider 04/01/09
Shanna Snider Date
Department Manager

Kenneth E. Kasperek 04/01/09
Kenneth E Kasperek Date
Technical Director

Peggy Gray-Erdmann 04/01/09
Peggy Gray-Erdmann Date
Quality Assurance Manager

04/01/09
Christopher A Spencer Date
Laboratory Director

Facility Distribution No. ___________ **Distributed To:**___________________

1.0 Scope and Application

If a total analysis of any of the waste demonstrates that individual analytes are not present in the waste, or that they are present but at such low concentrations that the appropriate regulatory levels could not possibly be exceeded, the TCLP need not be run.

If an analysis of any one of the liquid fractions of the TCLP extract indicates that a regulated compound is present at such high concentrations that, even after accounting for dilution from the other fractions of the extract, the concentration would be above the regulatory level for that compound, then the waste is hazardous and it is not necessary to analyze the remaining fractions of the extract.

1.1 Analytes, Matrix(s), and Reporting Limits

This method is applicable to liquid, solid and multiphasic wastes.

Reporting Limit: N/A

On occasion clients may request modifications to this SOP. These modifications are handled following the procedures outlined in the Quality Assurance Manual.

2.0 Summary of Method

The wastes are initially characterized and defined by matrix (liquid, solid or mixed phase) and by pH. This preliminary characterization determines the type of TCLP extraction procedure to be applied. Wastes containing less than 0.5 percent dry solid material are classified as liquid wastes and after filtration, are defined as the final TCLP extract. If the wastes contain greater than or equal to 0.5 percent solids, the liquid, if any, is separated from the solid phase and stored for later analysis. The solid phase is extracted with an amount of extraction fluid equal to 20 times the weight of the solid phase. Samples for volatiles analysis are extracted in a special pressurized extraction vessel. Extractions are conducted for a period of 18 ± 2 hours, followed by analysis of the extracts by approved EPA methodologies.

3.0 Definitions

TCLP : Toxicity Characteristic Leaching Procedure
Standard definitions can be found in section 5.1 of the laboratory quality manual

Percent Solids: That fraction of a waste sample from which no liquid may be forced out by an applied pressure.

4.0 Interferences

Potential interferences that may be encountered during analysis are discussed in individual analytical methods.

Glassware and equipment contamination may result in analyte degradation. Soap residue on glassware and equipment may contribute to this. All glassware and equipment should be rinsed very carefully to avoid this problem. All glassware and equipment used for TCLPs destined for metals analysis must be rinsed with 12.5 percent nitric acid prior to washing to minimize metals contamination.

Phthalates may be eliminated by proper glassware cleanup and by avoiding plastics. Only glass, Teflon or stainless steel tumblers may be used for leachates to be analyzed for organics. Plastic tumblers may be used for leachates to be analyzed for metals.

5.0 Safety

Employees must abide by the policies and procedures in the Corporate Environmental Health and Safety Manual (CW-E-M-001), Radiation Safety Manual and this document. This procedure may involve hazardous material, operations and equipment. This SOP does not address all of the safety problems associated with its use. It is the responsibility of the user of the method to follow appropriate safety, waste disposal and health practices under the assumption that all samples and reagents are potentially hazardous. Safety glasses, gloves, lab coats and closed-toe, nonabsorbent shoes are a minimum.

5.1 Specific Safety Concerns or Requirements

The use of safety glasses and protective clothing are required throughout the entire procedure.

The solvents and reagents used in this extraction procedure are hazardous if improperly handled. Care must be taken during preparation and use of acetic acid, hydrochloric acid, nitric acid and sodium hydroxide solutions. Additional health and safety information is available and must be read from the Material Safety Data Sheets (MSDS) maintained in the laboratory.

The acetic acid extraction fluid in the nonvolatile extraction vessels may react with carbamates in the sample to form CO2 gas. Pressure buildup could potentially cause the vessels to explode. The vessels should be periodically vented during extraction, and once again prior to removal from the rotation apparatus to prevent this occurrence.

Proper precautions must be taken when using pressurized nitrogen during the filtration and pressurized procedures. All steps of procedure should be done under a fume hood.

5.2 Primary Materials Used

The following is a list of the materials used in this method, which have a serious or significant hazard rating. Note: **This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the MSDS for each of the materials listed in the table.** A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the MSDS for each material before using it for the first time or when there are major changes to the MSDS.

Material (1)	**Hazards**	**Exposure Limit (2)**	**Signs and symptoms of exposure**
Acetic Acid	Corrosive Poison Flammable	10 ppm-TWA	Contact with concentrated solution may cause serious damage to the skin and eyes. Inhalation of concentrated vapors may cause serious damage to the lining of the nose, throat, and lungs. Breathing difficulties may occur.
Hydrochloric Acid	Corrosive Poison	5 ppm-Ceiling	Inhalation of vapors can cause coughing, choking, inflammation of the nose, throat, and upper respiratory tract, and in severe cases, pulmonary edema, circulatory failure, and death. Can cause redness, pain, and severe skin burns. Vapors are irritating and may cause damage to the eyes. Contact may cause severe burns and permanent eye damage.
Nitric Acid	Corrosive Oxidizer Poison	2 ppm-TWA 4 ppm-STEL	Nitric acid is extremely hazardous; it is corrosive, reactive, an oxidizer, and a poison. Inhalation of vapors can cause breathing difficulties and lead to pneumonia and pulmonary edema, which may be fatal. Other symptoms may include coughing, choking, and irritation of the nose, throat, and respiratory tract. Can cause redness, pain, and severe skin burns. Concentrated solutions cause deep ulcers and stain skin a yellow or yellow-brown color. Vapors are irritating and may cause damage to the eyes. Contact may cause severe burns and permanent eye damage.
Sodium Hydroxide	Corrosive	2 Mg/M3-Ceiling	Severe irritant. Effects from inhalation of dust or mist vary from mild irritation to serious damage of the upper respiratory tract, depending on severity of exposure. Symptoms may include sneezing, sore throat or runny nose. Contact with skin can cause irritation or severe burns and scarring with greater exposures. Causes irritation of eyes, and with greater exposures it can cause burns that may result in permanent impairment of vision, even blindness.
1 – Always add acid to water to prevent violent reactions.			
2 – Exposure limit refers to the OSHA regulatory exposure limit.			

6.0 Equipment and Supplies

6.1 Instrumentation

Agitation apparatus: The rotation apparatus must be capable of rotating the extraction vessel in an end-over-end fashion at 30 ± 2 rpm.

PH meter: calibrated daily

6.2 Supplies

Extraction Vessels:

Zero-Headspace Extraction Vessel (ZHE)-This device is for use only when the waste is being tested for the mobility of volatile analytes. The ZHE allows for liquid/solid separation within the device and effectively precludes headspace. The vessels shall have an internal volume of 500-600 mL and be equipped to accommodate a 90-110mm filter. The device contains O-rings, which should be replaced frequently.

Bottle Extraction Vessel- Borosilicate medium walled glass, Teflon screw cap, for semi-volatile and pesticides.

2 Liter plastic extraction bottles with lids, for metal only extractions.

TCLP-ZHE Filtration Apparatus.

ZHE Extraction Fluid Transfer Device

A pH Meter and pH probe-accurate to ± 0.05 units at 25° C. Prior to TCLP pH measurements, calibrate the pH meter and electrode in accordance with the manufacturer's recommendations. Calibrate the pH meter using buffers, which bracket the pH of the samples and extraction fluid.

Laboratory Balance-balance must be accurate to within ± 0.01 grams. All weight measurements are to be within ± 0.1 grams.

Magnetic stirrer

Glassware:
  Beakers, glass 250mL
  Graduated cylinders, 2000mL.
  Erlenmeyer flasks, glass, 1000-mL.
  Whatman Glass Microfiber Filters – grade GF/F.
  Pre-washed Nitric filters purchased by ESS.
  Pre-preserved 40-mL vials.

Mortar and Pestle.

Standard Sieve 9.5mm.
Compressed Nitrogen

7.0 Reagents and Standards

Water for nonvolatile extractions is ASTM Type II from the deionized water system in the Organic Prep Lab.

Reagent Water for Volatile extractions is generated from the Volatile-Free purification system located in Organic Prep area.

Glacial Acetic Acid, ACS reagent grade.

Hydrochloric Acid (1N), ACS reagent grade.

Sodium hydroxide (1N), ACS reagent grade.

Nitric acid (1N), ACS reagent grade.

Extraction Fluid #1, pH=4.93 (Section 10).

Extraction Fluid #2, pH=2.88 (Section 10).

8.0 Sample Collection, Preservation, Shipment and Storage

A minimum of 130g of sample is required for Organics/Metals. A minimum of 30g is required for VOA.

Preservatives shall not be added to samples prior to extraction.

When the waste is to be evaluated for volatiles analytes, care shall be taken to minimize the loss of volatiles. Samples shall be collected and stored in order to prevent the loss of volatiles analytes.

TCLP extractions and the analysis of the extracts must be conducted within the time period specified in Section 9. Extracts to be analyzed for metals must be preserved with 1N ACS reagent grade nitric acid to a pH of less than 2, unless precipitation occurs. Extracts should be preserved for other analytes according to the guidance given in the individual analysis methods. Extracts for volatiles shall not be allowed to come into contact with the atmosphere (no headspace) to prevent losses.

9.0 Quality Control

9.1 Sample QC

Extraction Vessel Blanks

The nonvolatile extraction vessels are 2-L borosilicate glass bottles with screw caps. Each extraction vessel must be demonstrated to be free of contamination by performing a blank extraction in each vessel. The extraction blanks are set up in vessels, which are rotated as samples . One blank must be extracted per extraction fluid per rotation. The vessel selected for EBLK tumbling is rotated each time. A separate blank is required for ZHE extractions. A separate blank is required for metal extractions done in plastic containers.

Using a 2000mL graduated cylinder add extraction fluid #1 (or extraction fluid #2) depending on preliminary sample evaluation, to the vessel. Place the container in the tumbler and secure the lid. Rotate the vessel at 30 ± 2 rpm for 18 ± 2 hours.

A minimum of one blank (using the same extraction fluid as used for the samples) must be analyzed for every 20 extractions that have been conducted in an extraction vessel. This applies to both volatile and nonvolatile extractor vessels.

TCLP extractions, preparations and analyses must be conducted within the following time periods:

From Field Collection to TCLP Extraction (Filtration)

Volatiles	14 days
Semivolatiles	14 days
Mercury	28 days
Metals, except mercury	180 days

From TCLP Extraction to Preparation Extraction

Volatiles	Not Applicable
Semivolatiles	7 days
Mercury	Not Applicable
Metals, except mercury	Not Applicable

From Preparation Extraction to Analysis

Volatiles	14 days
Semivolatiles	40 days
Mercury	28 days
Metals, except mercury	180 days

Total Elapsed Time

Volatiles	28 days
Semivolatiles	61 days
Mercury	56 days
Metals, except mercury	360 days

9.2 Instrument QC

N/A

10.0 Procedure

10.1 Sample Preparation

The TCLP preparation procedure can be broken down into four sections:

- Preliminary Sample Evaluation
- Preparation of Extraction Fluids
- TCLP Extraction Procedure for Nonvolatile Analytes
- TCLP Extraction Procedure for Volatile Compounds

10.1.1 Preliminary Sample Evaluation

A preliminary evaluation of the samples is performed prior to TCLP extraction. The results of the evaluation determine how the extraction is conducted and how the results are reported. The preliminary evaluation includes the following:

- Verification that sample contains no obvious liquid phase. Press down on sample with tongue depressor. If sample contains no obvious liquid, proceed to particle size reduction. If sample does yield liquid, determine percent solids

- Determination of percent solids using page 2 tab of spreadsheet.

- Determination if the waste contains insignificant amount of solid material, and is therefore the TCLP extract after filtration.

- Determination if the solid part of the waste needs particle size reduction.

- Determination of the extraction fluid to be used for the nonvolatile extractions, based on the pH of the waste.

10.1.2 Liquid Samples Extraction (Filter Only samples):

If the samples have been found to contain less than 0.5% dry solids, the filtrate is the TCLP extract.

Using a GF/F filter and filter apparatus collect the filtrate into the appropriate container. If metals are to be analyzed, acid treated filter papers are used.

Once filtered, samples are relinquished to the appropriate department.

If the filtrate is to be analyzed for metals, the pH is adjusted by the Metals Department to less than 2 with 1N metals grade nitric acid. Check an aliquot for precipitation before acidifying the entire extract. If a precipitate does form, do not adjust the pH of the extract. Analyze the extract for metals as soon as possible.

The Extractable portion is stored at 4° C in main sample cooler.

10.1.3 Percent Solids Determination:

If samples are not 100% dry, the amount of solid material in the sample must be determined.

NOTE: If the sample is a mixed phase sample, contact the PM to determine which phase the client is interested in.

Use the following procedure in conjunction with tab 2 of the excel spreadsheet.

The samples are filtered under pressure through glass Microfiber filters.

Prior to assembly of the filtration apparatus, clean all parts by washing with soapy water followed by rinsing with deionized water and reagent grade water.

Weigh a Whatman glass microfiber filter (grade GF/F) and record the weight in the TCLP spreadsheet.

Set up filtration apparatus.

Rinse the glass filter paper and the metal filter screen with deionized water.

Place the filter paper on the screen and in the extractor such that the glass fiber filter will be facing the sample.

Transfer 100g (to the nearest 0.1 g) of a representative aliquot of the sample into a tared 250 mL beaker, record the total weight in the spreadsheet.

Carefully pour or spread the sample onto the filter paper in the cylinder. Material may stick to the sides of the beaker. Quantitatively determine the amount transferred to the filtration apparatus by weighing the filter container and record the weight in the spreadsheet.

Gradually apply vacuum or gentle pressure of 10-50 psi, until air or pressurizing gas moves through the filter. If this point is not reached under 50 psi, and if no additional liquid has passed through the filter in any 2 minute interval, slowly increase the pressure in 10 psi increments to a maximum of 50 psi. After each incremental increase of 10 psi, if the pressurizing gas has not moved through the filter, and if no additional liquid has passed through the filter in any 2 minute interval, proceed to the next 10 psi increment. When the pressurizing gas begins to move through the filter, or when liquid flow has ceased at 50 psi, filtration does not result in any additional filtrate within any 2 minute period, stop the filtration.

NOTE: Some wastes, such as oily wastes and some paint wastes, will obviously contain some material that appears to be a llquid. Even after applying vacuum or pressure filtration, this material may not filter. If this is the case, the material within the filtration device is defined as a solid. Do not replace the original filter with a fresh filter under any circumstances. Use only one filter.

$$\text{Percent Dry Solids} = \frac{(\text{Wt of dry waste + Filter}) - \text{Wt of filter}}{\text{Initial Wt of Waste}} \times 100$$

$$\text{Percent solids} = \frac{\text{Weight of solids}}{\text{Total weight of waste}} \times 100$$

If the percent solids exceeds 0.5%, the liquid. If any, is saved for either future combination with the TCLP extract or for separate analysis.

10.1.4 Particle Size Reduction:

If the solid material in the sample can pass through a 9.5 mm sieve (less than 1 cm in diameter), particle size reduction is unnecessary.

If the samples need particle size reduction, crush or grind the sample with a mortar and pestle or whatever means necessary to reduce the sample's particle size. Record in spreadsheet.

10.1.5 Fluid Determination for Non volatile Analyses:

An aliquot of sample is initially tested for pH. The results determine which fluid is used for nonvolatile TCLP Extraction. Volatile TCLP extraction uses only extraction fluid #1 , prepared with volatile free reagent water.

Weigh out 5.0 g of representative sample. Record weight in spreadsheet.

Using a 100-mL graduated cylinder, add 96.5 ml of DiH2O and stir the sample for 5 minutes using a magnetic stirrer.

Measure the pH of the sample using a calibrated pH meter and record in the spreadsheet. Calibrate the pH meter using buffers, which bracket the pH of the samples and extraction fluid.

If pH is less than 5, use extraction fluid #1 for the TCLP sample extraction.

If pH is greater than 5, using a disposable 10mL pipette, add 3.5 ml of 1N HCl and swirl. Warm the sample on a hotplate to 50°C and hold at that temperature for 10 minutes.

Allow the solution to cool to room temperature, measure the pH, record in the logbook. If the pH is now less than 5, use extraction fluid #1. If the pH is still greater than 5, use extraction fluid #2.

10.1.6 Preparing Extraction Fluid:

If the extraction fluid is made more than 24 hours before use, the pH must be checked prior to extraction.

Care must be taken to ensure adequate mixing of large volumes to make certain that a stable pH has been reached prior to recording the result

Preparation of Extraction **Fluid #1** (pH 4.93 + 0.05)
To make a carboy (24 L): add 136.8ml glacial acetic acid, 154.3 1N NaOH and dilute to the mark with DiH_2O. Invert repeatedly to mix well. Using the pH meter, monitor the pH of the solution. Stir with a magnetic stirrer until the pH stabilizes. The pH must be in the range of 4.93 ± 0.05. If it is not, it must be remade. Record the pH, lot number of the acids and volumes into Element. Record the tracking number Element assigns in spreadsheet. Pre-made fluid concentrate, purchased from Environmental Express can also be used.

Preparation of Extraction **Fluid #2** (pH 2.88 + 0.05)
To make a 20L carboy add 130mL of acetic acid into 16L DiH20 and invert repeatedly to mix well. The pH of this fluid must be 2.88 + 0.05. If it is not, it must be remade. Record the pH, lot number of the acids and volumes into Element. Record the tracking number Element assigns in spreadsheet. Pre-made fluid concentrate, purchased from Environmental Express can also be used.

Preparation of the **Volatile Free Extraction Fluid** for ZHE (pH 4.93 + 0.05)
To make a carboy (24 L): add 136.8ml glacial acetic acid, 154.3 1N NaOH and dilute to the 16L mark with volatile free water. Invert repeatedly to mix well. Using the pH meter, monitor the pH of the solution. Stir with a magnetic stirrer until the pH stabilizes. The pH must be in the range of 4.93 + 0.05. If it is not, it must be remade. Record the pH, lot number of the acids and volumes into Element. Record the tracking number Element assigns in spreadsheet.

10.1.7 TCLP Extraction Procedure- Nonvolatile Samples Metals/Extractables

This procedure describes the TCLP extraction of samples for Extractables (semivolatile, pesticide, herbicide) and metal analysis.

10.1.8 Glassware Preparation:

All extraction vessels, glassware and utensils used for the extraction procedure must be washed with soapy water, rinsed with dilute nitric acid, rinsed with tap water and then followed by a rinse with reagent grade water.

10.1.9 Setting Solid Phase Samples (TCLP Organics, TCLP Metals):

If the sample is found to be 100% solids, and requires no particle size reduction (or has undergone reduction).

Weigh out 100g sample and transfer to the extraction vessel. Record the weight and the vessel number in the spreadsheet.

The volume of the extraction fluid used is 20 times the sample weight (100g sample aliquot to 2000mL of extraction fluid). If there is insufficient volume the client must be notified before proceeding

After the sample and appropriate extraction fluid has been added to the vessel record the initial pH of extract in the spreadsheet. Cap the vessel and clamp the vessels to the rotator.

Rotate the vessel at 30 ± 2 rpm for 18 ± 2 hours. Record the analyst, date, time and temperature at the beginning and end of the extraction. The temperature in the room should be maintained at 23± 2° C and checked using a min/max thermometer.

When the extractions are complete, remove the extractors from the rotator and separate the liquid and solid phases be filtering through a new glass fiber filter (GF/F).

Carefully decant the extraction fluid into the filtration apparatus. Collect the fluid in appropriate containers; glass for extractables and plastic for the metals. Discard the solid material left in the vessel.

Measure and record the final pH of the final extract.

If metals are to be analyzed, the filter must be acid washed and the pH of the extract corrected to < 2 with 1N metals reagent grade nitric acid.

Store the extractable portion in the afcee sample cooler. Release the metals portion to the metals digestion analyst. Update status of the samples in the LIMS System.

10.1.10 Mixed Phase Samples:

If the samples are mixed phase, decant and filter the liquid part first, using sufficient sample to perform the required analysis.

After the liquid part of the aliquot has been filtered, pour or spread the solid material onto the same filter. Complete the filtration procedure, and record the weights of the filtrate and the solid material.

Hold the liquid portion for future analyses, or for combination with the TCLP extract.

Evaluate the solid portion of the sample and transfer to the extraction vessel.

When transferring the solid material in the filtration apparatus to the extraction vessel, include the filter. Record all weights in the spreadsheet.

Conduct the extraction of the solid material.

If the filtered liquid phase of the mixed phase sample is compatible with the liquid extract from the solid phase extraction, combine the phases. This combination is the final mixed phase TCLP extract.
Analyze the TCLP extracts according to the appropriate analytical methods.

10.1.11 Setting Solid Phase Samples (TCLP ZHE):

This method is used for the TCLP extraction of samples for volatile analysis. Care must be taken to minimize the loss of volatiles by limiting the exposure of the samples, the filtrate, and the extracts to the atmosphere. Headspace should not be allowed in any of the extraction or collection containers.

NOTE: **If the sample consists of pure oil, paint or solvent, consult the supervisor before proceeding.** If the sample matrix is deemed unacceptable for the ZHE extractor, the supervisor is required to contact the Project Manager to notify the client. The project manager and client would then evaluate if a total Volatile analysis of the sample will meet the needs of the project. The analytical test would be updated to reflect a total analysis. The results of this 'Total Analysis' should be considered approximately 20 times higher than a TCLP leaching procedure.

Assemble the ZHEs:
All pieces of ZHE apparatus are labeled and assembled as a single unit. Assuming the vessel is unassembled, first place the Model #3745-ZHE body (3) with either end up and install the air side flange O-ring (12) in the gland on top of the body.

Replace the piston O-rings (12) on the piston (5) by stretching them over the piston and into the gland. NOTE: Be careful not to "roll" the O-rings into the gland. Wet the piston with extraction fluid.

Align the piston (5) carefully with the top of the body (3) and gently press it into the body. Continue pressing the piston into the body until it is completely inside the cylinder. NOTE: Care is necessary not to damage the O-rings. It may be helpful to moisten the O-rings with extraction fluid.

Locate the air side flange (1) and place it on top of the ZHE. Visually align the holes in the flange and the ZHE body and secure the flange with three knobs (8). Uniformly tighten the knobs.

Invert the partially assembly ZHE such that the air side flange (1) is now down.

Prepare a new Filter-Pak by placing a glass filter element between the two Stainless steel screens. Set the assembled filter-Pak to the side, until sample is added.

Locate the waste side flange (2) and install a Filter-Pak O-ring (12) in the inner gland of the flange. Install the body O-ring (12) in the outer gland of the waste side flange. NOTE: Be careful not to "roll" the O-rings into the glands.

Invert the waste side flange (2) and place it on top of the ZHE, aligning the holes at the same time.

Place the assembled ZHE with liquid inlet/outlet valve (14) on the top.

Verify that both the liquid inlet/outlet valve (13) and the quick-exhaust/relief valve (16) are open. NOTE: The quick exhaust/relief valve is open when the handle is up and the liquid inlet/outlet valve is open when the arrow points up.

Transfer of Sample to the ZHE

If the sample matrix is 100% solid material, and particle size reduction is unnecessary, weigh 25g of sample to the nearest 0.1g. Record weight in the spreadsheet.

If particle size reduction is needed, reduce size, then transfer 25g (+/- 0.1) to ZHE.

If the sample matrix is a mixed phase, use the percent solids information and the following calculation to determine the correct sample size to use:

$$\text{Wt of Waste to Charge (grams) ZHE} = \frac{25\text{ grams}}{\text{Percent Solids}} \times 100$$

Pour the appropriate weight of the mixed waste slurry into a tared beaker and transfer to the ZHE, quickly attaching the top flange. Reweigh the beaker and record in the weight ion the spreadsheet.

Introduce the sample to be extracted into the open top of the ZHE making sure that the piston (5) is far enough into the body (3) to provide sufficient free volume. Weigh out a representative sample of a maximum of 25.00 ± 0.1 g. Record the weight in the spreadsheet and the number of the vessel being used.

Use the following formula to determine how much extraction fluid to add to the ZHE:

$$\text{Wt of the extraction fluid} = \frac{20\text{X percent solids X Wt of waste filtered}}{100}$$

For example, if the sample has been classified as 100% solid, 500 mL of extraction fluid will have to be injected.

Check that the body O-ring (12) is properly seated in its gland and reinstall the Filter-Pak in the top of the ZHE. NOTE: Center the Filter-Pak carefully in its recess.

Verify that the Filter-Pak O-ring (12) and the waste side flange O-ring (12) are properly seated in their respective glands. Place the waste side flange (2) back on the unit and uniformly tighten the three knobs to close the vessel.

Close the quick-exhaust/ relief valve (16) on the air side flange (1). Do not close the liquid inlet/outlet valve. NOTE: The quick-exhaust/relief valve is closed when the handle is horizontal.

Introduce the proper amount of extraction fluid into the vessel using a peristaltic pump. The object is to put the 500 ml of extraction fluid into the vessel without opening it as to not expose the sample inside to the atmosphere. A metering pump is now set up next to the vessel.

First, with the tubing off of the top of the vessel, open the 2-way stainless steel valve and the vent relief valve on the bottom of the vessel. Now, start the metering pump (containing 500 ml of extraction fluid). When the fluid is almost to the end of the tubing, connect the tubing to the Luer fitting on top of the vessel securely. The fluid should start flowing into the vessel, pushing the piston back down to the bottom of the cylinder. The process may be slow. When all the fluid has been pushed into the vessel, shut the pump off, close the 2-way stainless steel valve, and close the vent relief valve on the bottom of the vessel. Disconnect the tubing and set the pump apparatus aside.

Close the liquid inlet/outlet valve (13) and the quick-exhaust/ relief valve (16). Close the valve on the syringe and disconnect it from the ZHE. Physically rotate the vessel end over end 2 or 3 times and place it back on the chair (6) with the liquid inlet/outlet valve on top.

Connect the pressure source to the air side flange and set the line pressure at 5-10 psi. Slowly open the liquid inlet/outlet valve (13) to bleed out any headspace that may have been introduced during the addition of extraction fluid and close the liquid inlet/outlet valve at the first sign of liquid. Allow the pressure gage on the ZHE to stabilize and remove the pressure source. NOTE: 10 psi is recommended.

Place the ZHE in the rotary agitator (tumbler) and tumble for 18 ± 2 hours at 30 ± 2 RPM. The temperature in the room should be maintained at 23 ± 2° C. Record the analyst, date, time and temperature at the beginning and end of the extraction.

When rotation is complete, check that the pressure gauges still read 10 psi and record in spreadsheet. If the vessel is no longer pressurized, repeat the extraction with a new sample. Pressure readings are recorded in the excel spreadsheet.

Remove the ZHEs from the rotation apparatus and let stand for two hours to settle.

10.1.12 Filtering TCLP ZHE

Once the contents of the ZHE have settled, attach a pressure regulated source (set to 0 psi) of filtered compressed air or dry nitrogen to the gas inlet quick disconnect (14) found on the airside flange (1)). Note: Compressed air must be prefiltered (5micron rating) to prevent particulate matter from scoring the walls of the ZHE.

Increase the supply pressure to 5-10 psi and note the pressure on the vessel gage (17) is approximately 2-5 psi. This indicates that the piston (5) is in motion and removing the sample headspace. At the first sign of liquid release from the liquid inlet/outlet valve (13) immediately close the valve (13). Disconnect the pressure source and open the quick-exhaust/relief valve (16) to depressurize the ZHE. NOTE: This will take a short period of time depending on the volume of free air remaining on the sample side of the vessel. NOTE: Pressure greater than 10 psi may be necessary.

Attach an appropriate filtrate collection container to the liquid inlet/outlet valve and firmly hand tighten the Speed-Nut (10). Close the quick-exhaust/Teflon syringes.

Reattach the pressure source to the vessel and set the line pressure at 5-10 psi. Slowly open the liquid inlet/outlet valve (13) on the ZHE. Gradually increase the supply pressure in 10 psi increments (up to the maximum of 50 psi) until no more initial liquid phase is expelled in a two minute interval or until the 40mL vial is full and free of any visible air bubbles. NOTE: The pressure gage (17) on the vessel should be allowed to stabilize at the line pressure setting before beginning the two-minute period.

Close the liquid inlet/outlet valve(s), disconnect the pressure source and remove the collection device. Depressurize the ZHE by slowly opening the quick-exhaust/relief valve (16). NOTE: Leave the quick-exhaust/relief valve open.

10.1.13 Leak-testing the ZHE

Pressurize the ZHE to 50 psi and place in a large container of water. If bubbles escape from the vessel, the seals are leaking.

De-pressurize the ZHE and open the side that is leaking and re-wet the O-rings. Recheck for leaks in the seal. The leak check must be performed before every extraction and documented in the logbook.

10.1.14 Cleaning and Maintaining the ZHEs

Before the ZHEs are assembled, wash all the parts with hot, soapy water followed by rinsing with tap and deionized water. A sonic bath can be used to clean the more difficult parts. After cleaning, the cylinder, piston, stainless steel screens, and any rubber O-rings can be placed in the 103°C oven for a few hours. Only these parts can be heat treated as the heat damages any part containing a valve. Make sure the parts are room temperature before setting up samples. Make sure the pistons do not get interchanged. ZHE O-rings must be free of cuts or cracks or the extractors may leak. Before assembly, examine the O-rings for damage.

10.1.15 Aqueous Samples

If the samples have been found to contain less than 0.5 percent dry solids, the filtrate is the TCLP extract.

Collect the filtrate directly into a 40 mL VOA vial pre-preserved with HCL, allowing for no headspace to form.

Store the vials in the GC/MS sample cooler. Release samples in the LIMS System.

10.2 Calibration

The pH meter is calibrated every day prior to use. The calibration is performed with a 10.00 pH buffer solution and a 4.01 pH buffer solution, and then checked against a 7.01 pH buffer solution. The check must be accurate within ±0.05. If the check fails, repeat the calibration until a passing check has been achieved.

Analytical balances are calibrated every 6 months and checked daily to ensure calibration is maintained.

11.0 Calculation/ Data Reduction

See calculations noted in Procedure.

12.0 Method Performance

12.1 Method Detection Limit Study (MDL)

The method detection limit (MDL) is the lowest concentration that can be detected for a given analytical method and sample matrix with 99% confidence that the analyte is present. The MDL is determined according to the laboratory's MDL procedure in the QA Manual. MDLs reflect a calculated (statistical) value determined under ideal laboratory conditions in a clean matrix, and may not be achievable in all environmental matrices. The laboratory maintains MDL studies for analyses performed; these are verified at least annually unless method requirements require a greater frequency.

12.2 Demonstration of Capabilities

Refer to the laboratory QA manual for specific demonstration of capabilities procedures.

12.3 Training Requirements

Refer to the laboratory QA manual for specific training requirements.

13.0 Pollution Control

It is TestAmerica's policy to evaluate each method and look for opportunities to minimize waste generated (i.e., examine recycling options, ordering chemicals based on quantity needed, preparation of reagents based on anticipated usage and reagent stability). Employees must abide by the policies in Section 13 of the Corporate Environmental Health and Safety Manual (CW-E-M-001) for "Waste Management and Pollution Prevention."

14.0 Waste Management

Waste management practices are conducted consistent with all applicable rules and regulations. Excess reagents, samples and method process wastes are disposed of in an accepted manner. Waste description rules and land disposal restrictions are followed. . All waste will be disposed of in accordance with Federal, State and Local regulations. Waste disposal procedures are incorporated by referencing section 13 of the Corporate Safety Manual for "Waste Management and Pollution Prevention."

The following waste streams are produced when this method is carried out:

Acidic waste from sample extract: All acid waste is disposed of in "A" waste satellite containers (except nitric acid waste). When full, the satellite container is transferred to the secure waste storage area and disposed of by appropriately trained laboratory technicians in accordance to all state and federal regulations.

Solid waste from sample extract: The solid waste from the sample extract is dried and disposed of in a "BE" satellite container. When full, the satellite container is transferred to the secure waste storage area and disposed of by appropriately trained laboratory technicians in accordance to all state and federal regulations.

Remaining TCLP extracts: The remaining TCLP extracts are considered "A" waste and are disposed of directly to a 55-gallon "A" waste drum by appropriately trained laboratory technicians in accordance to all state and federal regulations.

15.0 References / Cross-References

Method 1311, "Test Methods for Evaluating Solid Waste", EPA SW846 Third Edition, 12/96

16.0 Method Modifications:

N/A

17.0 Attachments

Figure 1: Tumbler- Rotary Agitation Apparatus
Figure 2: Zero Headspace Extractor (ZHE)
Figure 3: ZHE Specifications

Figure 4: ZHE and Fluid Metering Pump
Figure 5: Flow chart
Figure 6: Regulated analytes for Toxicity Characteristic with Regulatory Levels.
Attachment 1: Spreadsheet – TCLP Metals/Extractables
Attachment 2: Mixed Phase spreadsheet (Page 2 tab)
Attachment 3: Filter Only Spreadsheets for Metals/Organics/VOA
Attachment 4: Spreadsheet – TCLP VOA

18.0 <u>Revision History</u>

Revision 1, dated 13 March 2009

- Integration for TestAmerica operations
- Changed SOP references from logbook to spreadsheet
- Updated attachments to include spreadsheets

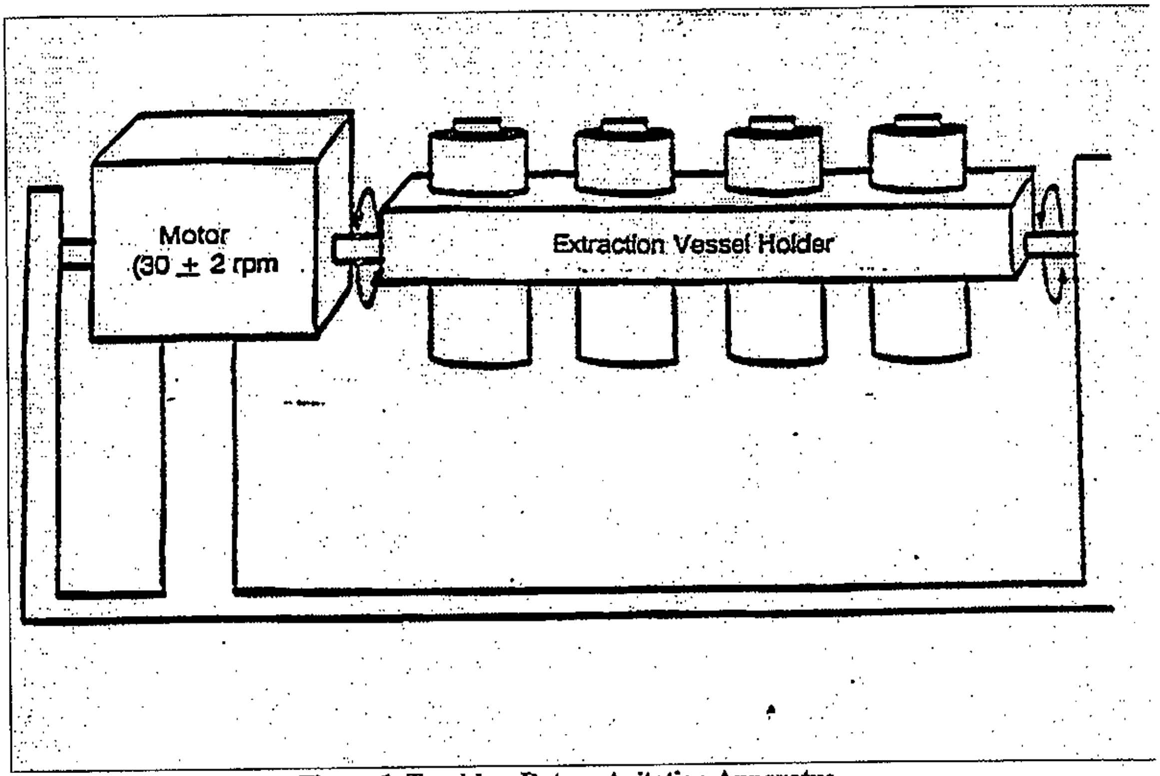


Figure 1. Tumbler- Rotary Agitation Apparatus

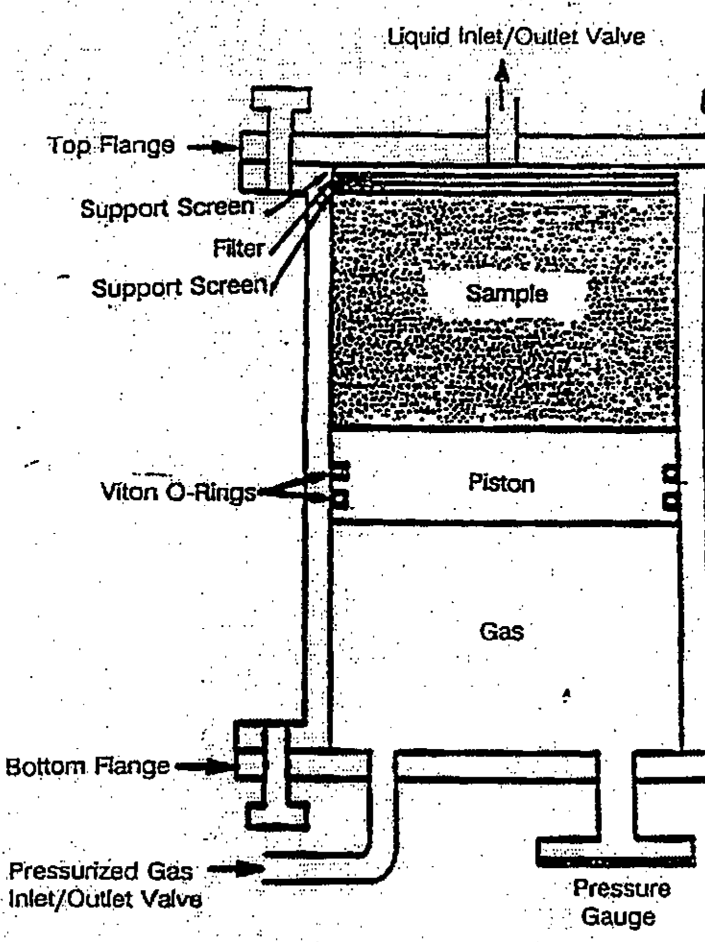
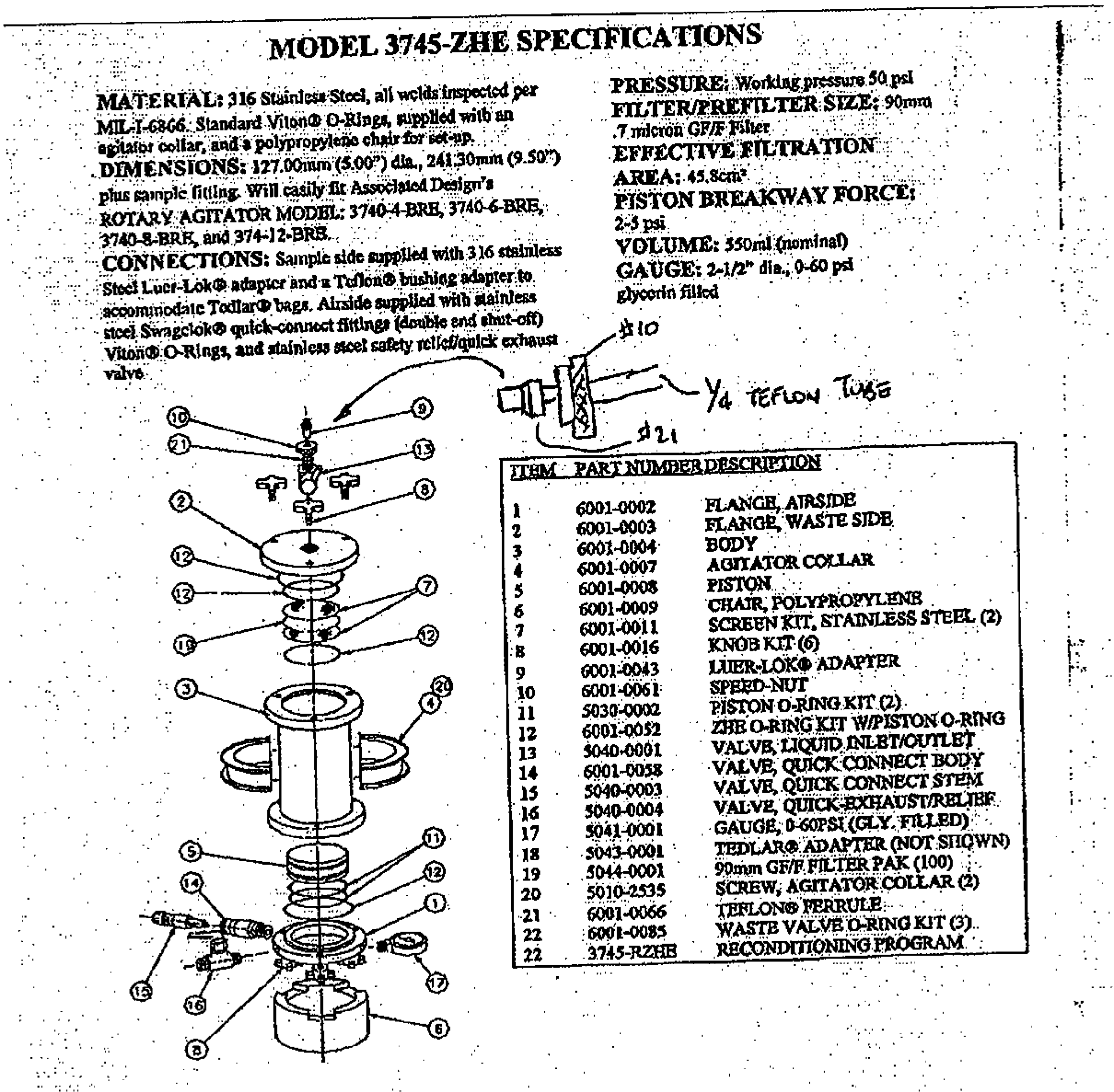


Figure 2. Zero Headspace Extractor (ZHE)



MODEL 3745-ZHE SPECIFICATIONS

MATERIAL: 316 Stainless Steel, all welds inspected per MIL-I-6866. Standard Viton® O-Rings, supplied with an agitator collar, and a polypropylene chair for set-up.
DIMENSIONS: 127.00mm (5.00") dia., 241.30mm (9.50") plus sample fitting. Will easily fit Associated Design's ROTARY AGITATOR MODEL: 3740-4-BRE, 3740-6-BRE, 3740-8-BRE, and 374-12-BRE.
CONNECTIONS: Sample side supplied with 316 stainless Steel Luer-Lok® adapter and a Teflon® bushing adapter to accommodate Tedlar® bags. Airside supplied with stainless steel Swagelok® quick-connect fittings (double end shut-off) Viton® O-Rings, and stainless steel safety relief/quick exhaust valve.

PRESSURE: Working pressure 50 psi
FILTER/PREFILTER SIZE: 90mm .7 micron GF/F Filter
EFFECTIVE FILTRATION AREA: 45.8cm²
PISTON BREAKWAY FORCE: 2-5 psi
VOLUME: 350ml (nominal)
GAUGE: 2-1/2" dia., 0-60 psi glycerin filled

ITEM	PART NUMBER	DESCRIPTION
1	6001-0002	FLANGE, AIRSIDE
2	6001-0003	FLANGE, WASTE SIDE
3	6001-0004	BODY
4	6001-0007	AGITATOR COLLAR
5	6001-0008	PISTON
6	6001-0009	CHAIR, POLYPROPYLENE
7	6001-0011	SCREEN KIT, STAINLESS STEEL (2)
8	6001-0016	KNOB KIT (6)
9	6001-0043	LUER-LOK® ADAPTER
10	6001-0061	SPEED-NUT
11	5030-0002	PISTON O-RING KIT (2)
12	6001-0052	ZHE O-RING KIT W/PISTON O-RING
13	5040-0001	VALVE, LIQUID INLET/OUTLET
14	6001-0058	VALVE, QUICK CONNECT BODY
15	5040-0003	VALVE, QUICK CONNECT STEM
16	5040-0004	VALVE, QUICK-EXHAUST/RELIEF
17	5041-0001	GAUGE, 0-60PSI (GLY. FILLED)
18	5043-0001	TEDLAR® ADAPTER (NOT SHOWN)
19	5044-0001	90mm GF/F FILTER PAK (100)
20	5010-2535	SCREW, AGITATOR COLLAR (2)
21	6001-0066	TEFLON® FERRULE
22	6001-0085	WASTE VALVE O-RING KIT (3)
22	3745-RZHE	RECONDITIONING PROGRAM

Figure 3:ZHE Specifications

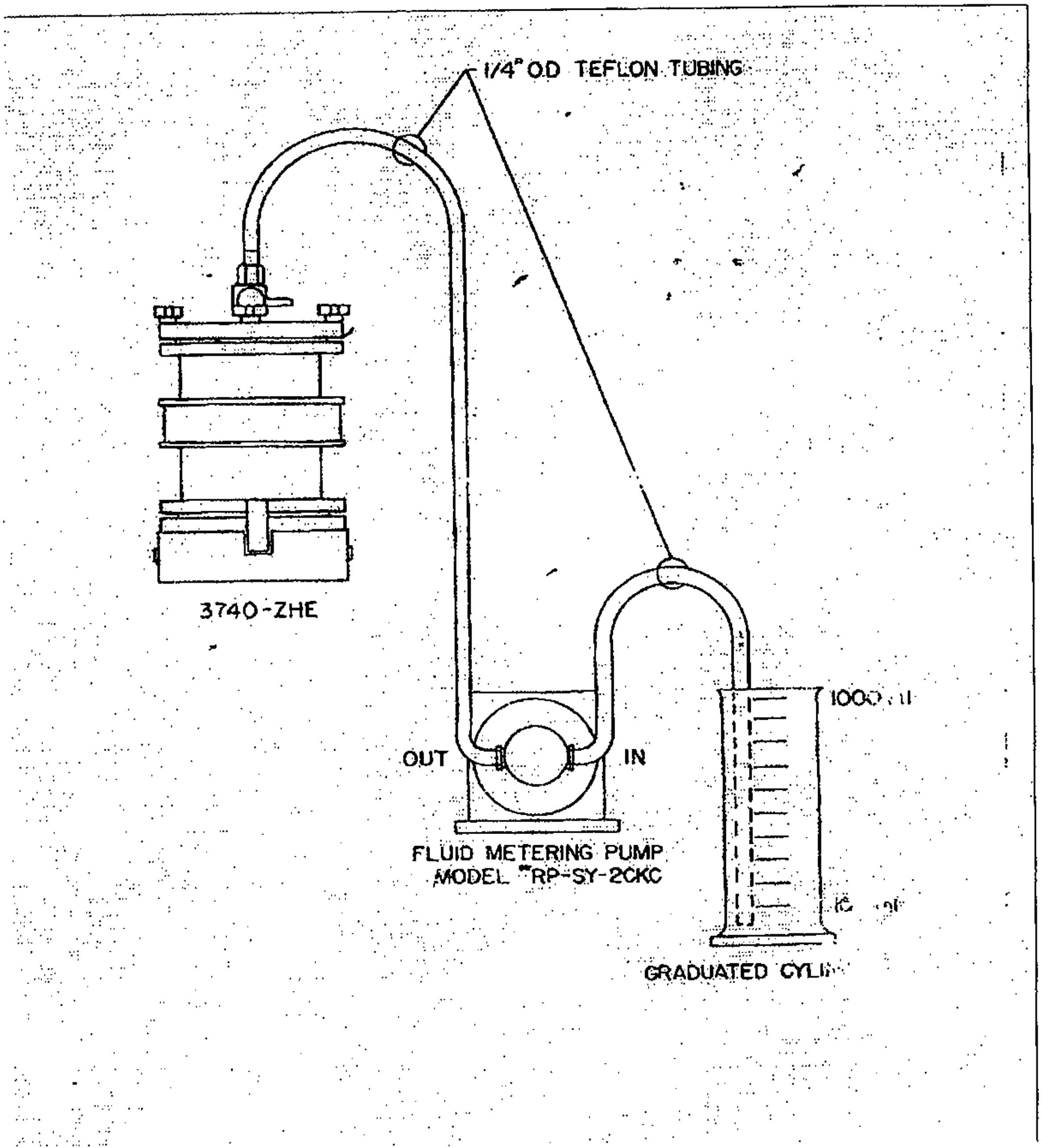


Figure 4. ZHE and Fluid Metering Pump

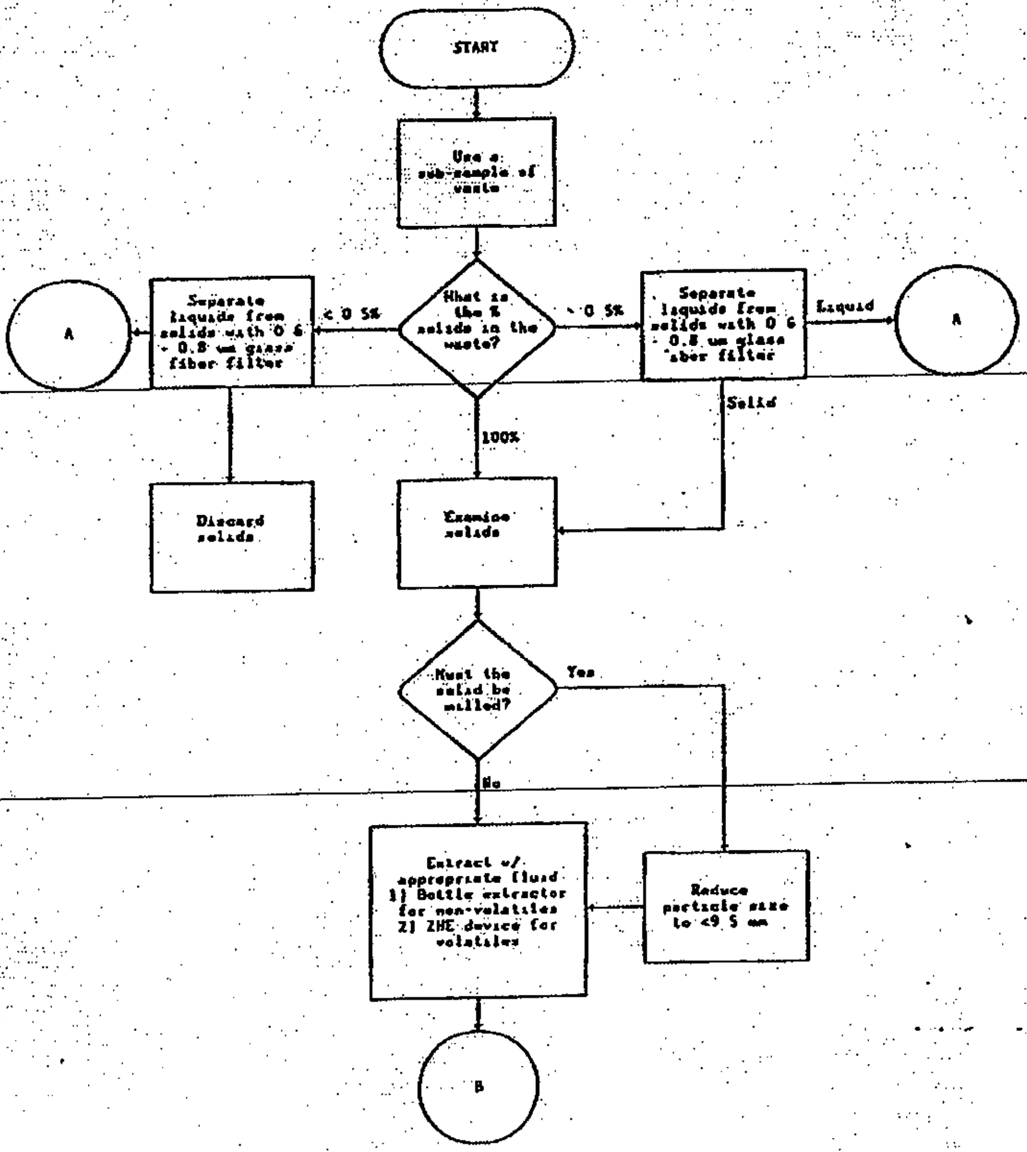


Figure 5. Flow Chart (page 1 of 2)

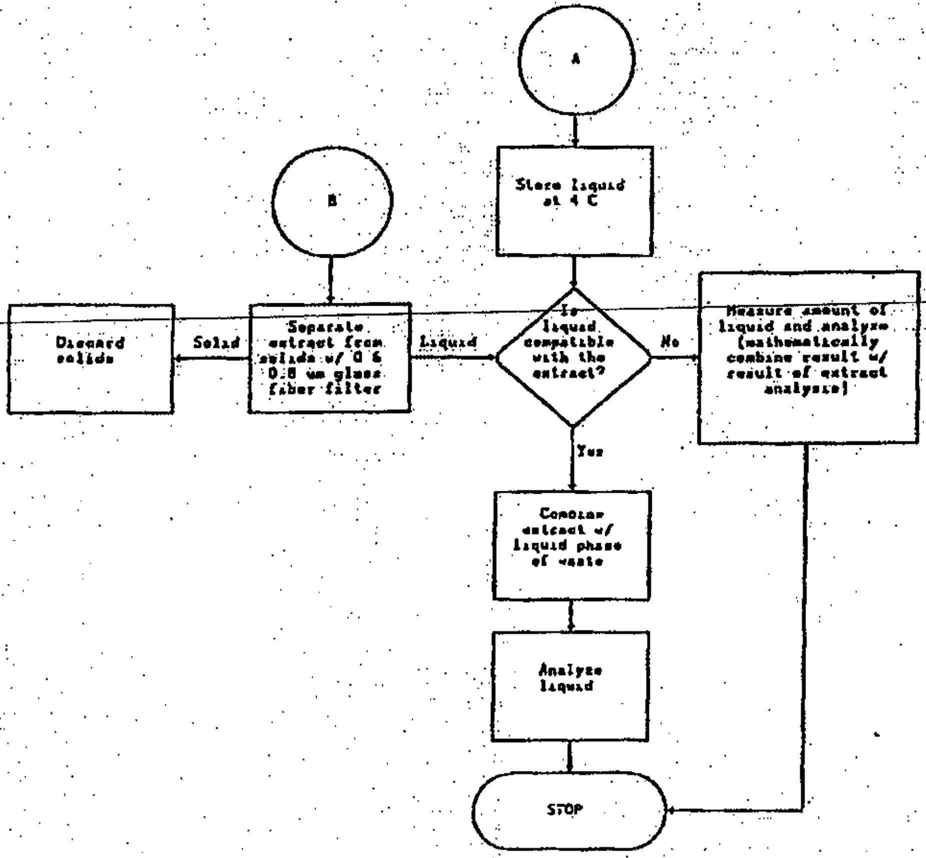
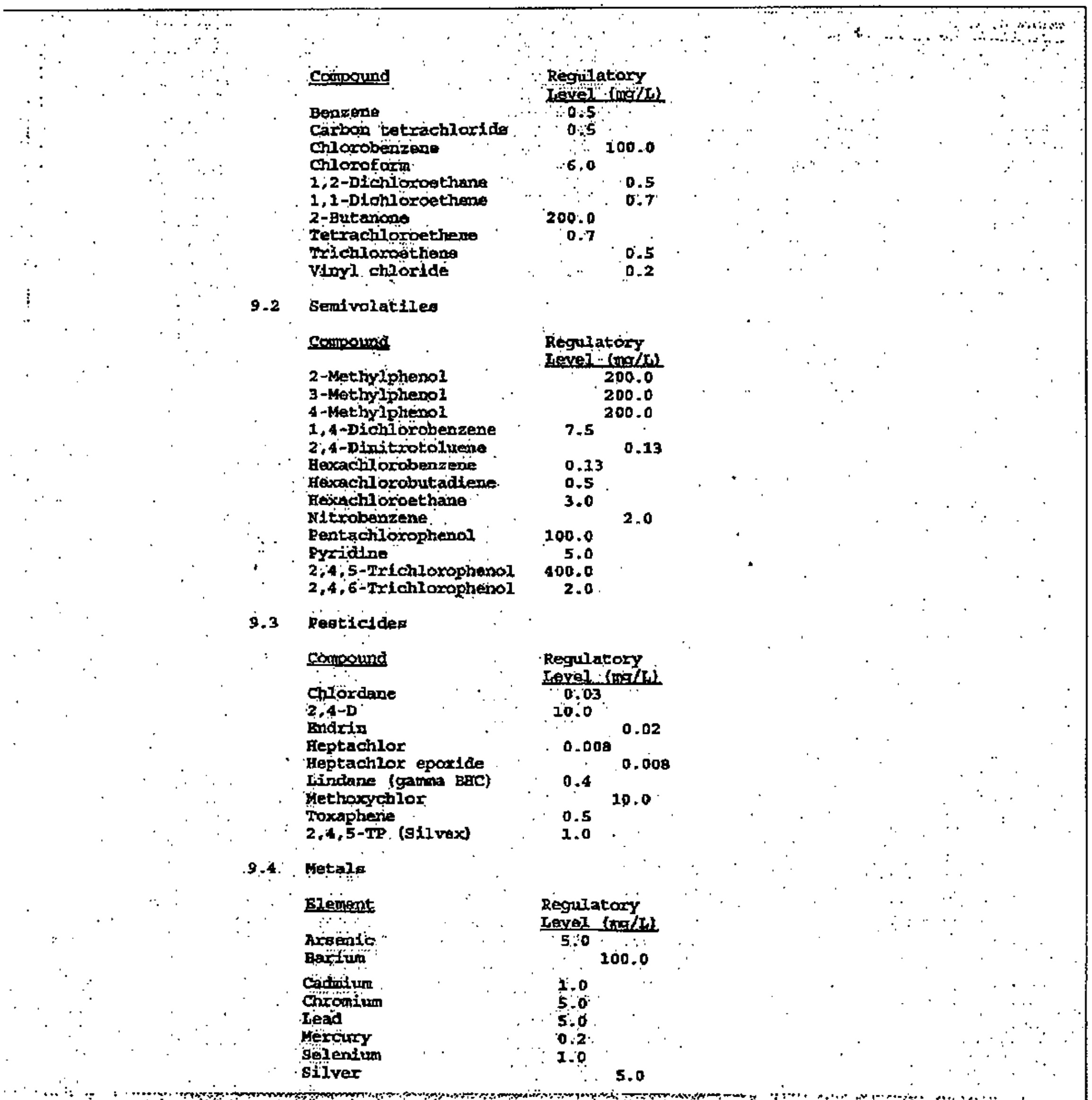


Figure 5. Flow Chart (page 2 of 2)



Compound	Regulatory Level (mg/L)
Benzene	0.5
Carbon tetrachloride	0.5
Chlorobenzene	100.0
Chloroform	6.0
1,2-Dichloroethane	0.5
1,1-Dichloroethene	0.7
2-Butanone	200.0
Tetrachloroethene	0.7
Trichloroethene	0.5
Vinyl chloride	0.2

9.2 Semivolatiles

Compound	Regulatory Level (mg/L)
2-Methylphenol	200.0
3-Methylphenol	200.0
4-Methylphenol	200.0
1,4-Dichlorobenzene	7.5
2,4-Dinitrotoluene	0.13
Hexachlorobenzene	0.13
Hexachlorobutadiene	0.5
Hexachloroethane	3.0
Nitrobenzene	2.0
Pentachlorophenol	100.0
Pyridine	5.0
2,4,5-Trichlorophenol	400.0
2,4,6-Trichlorophenol	2.0

9.3 Pesticides

Compound	Regulatory Level (mg/L)
Chlordane	0.03
2,4-D	10.0
Endrin	0.02
Heptachlor	0.008
Heptachlor epoxide	0.008
Lindane (gamma BHC)	0.4
Methoxychlor	10.0
Toxaphene	0.5
2,4,5-TP (Silvex)	1.0

9.4 Metals

Element	Regulatory Level (mg/L)
Arsenic	5.0
Barium	100.0
Cadmium	1.0
Chromium	5.0
Lead	5.0
Mercury	0.2
Selenium	1.0
Silver	5.0

Figure 6. Regulated Analytes for Toxicity Characteristic with Regulatory Levels

Attachment 1 **Spreadsheet – TCLP Metals/Extractables**

FLUID 1 BATCH#	
FLUID 2 BATCH#	

SOLUTIONS:					
	1.68	4.00	7.00	10.00	12.54
Thermo Orion					
Eutech					

Analyst Set	
Date Set	
Tumbler Start	
Analyst Filter	
Tumbler Stop Time	
Tumbler Min. Temp.	
Tumbler Max. Temp.	
Date/ Time Filtration	
Tumbler Calibration Date (30 +/- 2 rpm)	

Extraction Fluid Preparation				
Element ID	Extraction Fluid	Analyst	Date	pH

				Extraction Fluid Deterimination					Sample Prep. For Extraction			Final TCLP Extract	
Job #	M or M/E	Vessel (Only Applies to Extractable)	Reduced Y or N	Weight of Test Sample	DiH2O added (mLs)	Initial pH after 5 min.	Final pH after acid	Extraction Fluid Used	Weight of Sample	Volume of Fluid (mLs)	Initial pH of extract	Volume Filtered	Final pH of Extract
			N		96			1		0			
			N		96			1		0			
			N		96			1		0			
			N		96			1		0			
			N		96			1		0			
			N		96			1		0			
			N		96			1		0			
			N		96			1		0			
			N		96			1		0			
			N		96			1		0			
			N		96			1		0			
			N		96			1		0			
			N		96			1		0			
			N		96			1		0			
			N		96			1		0			
			N		96			1		0			
			N		96			1		0			
			N		96			1		0			
			N		96			1		0			
			N		96			1		0			

Comments ______________________________

Data Entry:		Date:	
2nd Review:		Date:	

Attachment 2 **Mixed Phase spreadsheet (Page 2 tab)**

Job Number	M or M/E	Weigt of Empty Container	Weight of Wet+Dry Sample (g)	Weight of Liquid + Container (g)	% Solids	Weight of sample to be Tumbled	Volume of Extraction Fluid Added		pH of Liquid Extract	Are Liquid Volumes Compatible ? (Y/N)		pH of Combined Volume	Volume Filtered (mLs)
					#DIV/0!	0	0	Tumble			If Yes, Combine Filtered Liquid Portion to Tumbed TCLP before filtering. If No, Treat as 2 separate Extracts		
					#DIV/0!	0	0						
					#DIV/0!	0	0						
					#DIV/0!	0	0						
					#DIV/0!	0	0						
					#DIV/0!	0	0						
					#DIV/0!	0	0						
					#DIV/0!	0	0						
					#DIV/0!	0	0						
					#DIV/0!	0	0						
					#DIV/0!	0	0						
					#DIV/0!	0	0						
					#DIV/0!	0	0						
					#DIV/0!	0	0						
					#DIV/0!	0	0						
					#DIV/0!	0	0						
					#DIV/0!	0	0						
					#DIV/0!	0	0						
					#DIV/0!	0	0						
					#DIV/0!	0	0						
					#DIV/0!	0	0						
					#DIV/0!	0	0						

Attachment 3 **Filter Only Spreadsheets for Metals/Organics/VOA**

Filter Only (metals/organics)

Analyst	
Date	
Time	
Batch #	

Job #	Sample I.D.	M or M/E

Filter Only (VOA)

Analyst	
Date	
Time	
Batch #	

Job #	Sample I.D.

Attachment 4: TCLP ZHE Spreadsheet (VOAS)

FLUID 1 BATCH#	

SOLUTIONS:	OP-	OP-	OP-
	4.00	7.00	10.00

Analyst Filter	
Tumbler Stop Time	
Tumbler Min. Temp.	
Tumbler Max. Temp.	
Date/Time Filtration	
Analyst Set	
Date Set	
Tumbler Start	
Tumbler Calibration Date (30+/-2 rpm)	

VOA Extration Fluid Preparation			
Element ID	Analyst	Date	pH

Comments:

	Sample Prep. For Extraction		Final TCLP Extract				
Job #	ZHE	Initial ZHE Pressure	Final ZHE Pressure	Pressure Test	Weight of Sample	Volume of Fluid (mLs)	Volume Filtered
	1			PASS		0	
	2			PASS		0	
	3			PASS		0	
	4			PASS		0	
	5			PASS		0	
	6			PASS		0	
	7			PASS		0	
	8			PASS		0	
	9			PASS		0	
	10			PASS		0	
	11			PASS		0	
	12			PASS		0	
	13			PASS		0	
	14			PASS		0	
	15			PASS		0	
	16			PASS		0	
	17			PASS		0	
	18			PASS		0	
	22			PASS		0	
	23			PASS		0	
	24			PASS		0	
	25			PASS		0	
	26			PASS		0	

Data Entry By:

Date:

2nd Review By:

Date:

Total Dissolved Solids
Method 160.1 and SM 2540C

Once printed, this is considered an uncontrolled document

Approvals (Signature/Date):

Lisa R Matecki 01/27/10
Lisa Matecki Date
Department Manager

Kenneth E. Kasperek 01/27/10
Kenneth E. Kasperek Date
Health & Safety Manager / Coordinator

Peggy Gray-Erdmann 01/27/10
Peggy Gray-Erdmann Date
Quality Assurance Manager

01/27/10
Christopher A. Spencer Date
Laboratory Director

Copyright Information:
This documentation has been prepared by TestAmerica Analytical Testing Corp. and its affiliates ("TestAmerica"), solely for their own use and the use of their customers in evaluating their qualifications and capabilities in connection with a particular project. The user of this document agrees by its acceptance to return it to TestAmerica upon request and not to reproduce, copy, lend, or otherwise disclose its contents, directly or indirectly, and not to use if for any other purpose other than that for which it was specifically provided. The user also agrees that where consultants or other outside parties are involved in the evaluation process, access to these documents shall not be given to said parties unless those parties also specifically agree to these conditions.

THIS DOCUMENT CONTAINS VALUABLE CONFIDENTIAL AND PROPRIETARY INFORMATION. DISCLOSURE, USE OR REPRODUCTION OF THESE MATERIALS WITHOUT THE WRITTEN AUTHORIZATION OF TESTAMERICA IS STRICTLY PROHIBITED. THIS UNPUBLISHED WORK BY TESTAMERICA IS PROTECTED BY STATE AND FEDERAL LAW OF THE UNITED STATES. IF PUBLICATION OF THIS WORK SHOULD OCCUR THE FOLLOWING NOTICE SHALL APPLY:

©COPYRIGHT 2007 TESTAMERICA ANALYTICAL TESTING CORP. ALL RIGHTS RESERVED

Facility Distribution No. ___________ **Distributed To:**________________________

1.0 Scope and Application

1.1 Analytes, Matrix(s), and Reporting Limits

1.1.1 This method is applicable to drinking, surface, and saline waters, domestic and industrial wastes.

1.1.2 The practical range of determination is from 10 mg/L to 20,000 mg/L.

On occasion clients may request modifications to this SOP. These modifications are handled following the procedures outlined in the Quality Assurance Manual.

2.0 Summary of Method

Testing for total dissolved solids involves weighing a clean 250 ml beaker to the nearest milligram, filtering a known volume of sample through a glass fiber filter and evaporating the filtrate. Weigh the dish again with the resulting residue, and then subtract the two results to determine the amount of milligram of residue per liter of water.

3.0 Definitions

3.1 Filterable residue is defined as those solids capable of passing through a glass fiber filter and dried to constant weight of < 0.5 mg.

3.2 Standard definitions are found in section 3.0 of the Laboratory Quality Manual.

4.0 Interferences

4.1 Highly mineralized waters containing significant concentrations of calcium, magnesium, chloride and/or sulfate may be hydroscopic and will require prolonged drying, desiccation and rapid weighing.

4.2 Samples containing high concentrations of bicarbonate will require careful and possibly prolonged drying at 180 degrees C to insure all the bicarbonate is converted to carbonate.

4.3 Too much residue in the beaker will crust over and entrap water that will not be driven off during drying. Total residue should be limited to about 200 mg

5.0 Safety

Employees must abide by the policies and procedures in the Corporate Safety Manual, Radiation Safety Manual and this document.

This procedure may involve hazardous material, operations and equipment. This SOP does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of the method to follow appropriate safety, waste disposal and health practices under the assumption that all samples and reagents are potentially hazardous. Safety glasses, gloves, lab coats and closed-toe, nonabsorbent shoes are a minimum.

5.1 Specific Safety Concerns or Requirements

None

6.0 Equipment and Supplies

6.1 Supplies

- Glass fiber filter discs, 4.7 cm or 2.1 cm, without organic binder, Reeve Angel type 934-AH, Gelman type A/E, or equivalent.
- Filter holder, membrane filter funnel or Beaker adapter.
- Suction flask, 1000mL
- Beakers, 250mLs
- Drying oven, 180 degrees C ± 2 degrees.
- Desiccator
- Graduated cylinder, 100mLs

7.0 Reagents and Standards

7.1 LCS: 500 mg/L NaCl Std: Dissolve 0.5 g of sodium chloride into 1 liter of Di water.

8.0 Sample Collection, Preservation, Shipment and Storage

Preservation of the sample is not practical; analysis should be performed within 7 days after sample collection. Refrigeration to 4 degrees C to minimize microbiological decomposition of solids is recommended.

9.0 Quality Control

9.1 Sample QC -

9.1.1 Laboratory Control Sample (LCS): Analyze one LCS at the beginning and end of the analytical procedure and another after every 20 samples or less. Obtained values must be ± 15% of the true value.

9.1.2 Method Blank: To determine freedom from contamination, prepare one blank at the beginning and end of the analytical procedure and after every 20 samples or less. The blank consists of 100mLs of Deionized water that goes through all the steps of the analytical procedure as the samples and standards. The MBLK must exhibit values less than the quantitation limit (10 mg/L).

9.1.3 A duplicate sample must be run every 20 samples.

10.0 Procedure

10.1 Heat a clean 250mL beaker to 180 +/- 2 degrees C for 1 hour. Cool in desiccator and store until ready to use. Weigh immediately before use.

10.2 Preparation of glass fiber filter disc: Place the disc on the membrane filter. While vacuum is applied, wash the disc with three successive 20mL volumes of distilled water. Remove all traces of the water by continuing to apply the vacuum after water has passed through. Discard washings.

10.3 Use a maximum of 100mLs of sample volume. After the drying cycle, if 100mLs of sample yields greater then 200mg of dried residue in the evaporating dish the sample must be reanalyzed using less sample volume. If necessary perform Specific Conductance on the sample to determine the proper volume to use. The conductivity result will determine the volume of sample based on the following relationship:

Specific Conductance (*u*S)	Sample Volume to use for TDS
0-3000	100mL
3000-6000	50mL
6000-12000	25mL
12000+	15mL

Excessive residue in a beaker may cause water-trapping crust, therefore limit the sample to no more than 200mg for final residue. Using the historical data of a sample will indicate the amount of sample needed for the analysis. The following table is a guide to determine the sample volume based upon historical results:

Historical Result of TDS Values (mg/l)	Sample Volume to use for TDS
0-1500	100mL
1500-3000	50mL
3000-6000	25mL
6000-12000	15mL
12000-18000	10mL
18000-30000	5mL
30000 +	1mL

10.4 Assemble the filtering apparatus and begin suction. Shake the sample vigorously and rapidly transfer volume to the funnel.

10.5 Filter the sample through the glass fiber filter, rinse with three 10mL portions of distilled water and continue to apply vacuum for about 3 minutes after filtration is complete to remove as much water as possible.

10.6 Transfer the filtrate including the washings to a weighed glass beaker and evaporate to dryness.

10.7 Dry the sample over night at $180^o \pm 2^o$. Cool in a desiccator and weigh. Repeat the desiccation cycle until a constant weight is obtained or until weight change is less than 0.5mg.

11.0 Calculations / Data Reduction

11.1 Total Dissolved Solids mg/L= $\frac{(A-B) \times 1000}{C}$

A= weight of sample and dish in grams
B= weight of dish in grams
C= volume of sample used in liters

11.2 Relative Percent Difference (RPD):

$$\mathrm{RPD} = \frac{|x_1 - x_2|}{\left(\frac{x_1 + x_2}{2}\right)} \times 100$$

where:

x_1 = analytical % recovery
x_2 = replicate % recovery

11.3 Percent Recovery for LCS:

$$\%\,\mathrm{Recovery\,(LCS)} = 100\left(\frac{E}{C}\right)$$

where:

E = obtained (experimental) value C = true value

12.0 Method Performance

12.1 Demonstration of Capabilities

12.1.1 A one–time initial demonstration of performance for each individual method for water matrices must be generated.

12.1.1.1 This requires quadruplicate analysis of an LCS check standard and a Method Blank containing all of the standard analytes for the method using the same procedures used to analyze samples, including sample preparation.

12.1.1.2 Compare these results with the acceptance criteria given in the Method or to laboratory historical limits (if available).

12.1.1.3 Repeat the test for any analyte that does not meet the acceptance criteria. Repeated failure for any analyte indicates the need for the laboratory to evaluate the analytical procedure and take corrective action.

12.2 Training Requirements

12.2.1 The supervisor has the responsibility to ensure that this procedure is performed by an analyst who has been properly trained in its use and has the required experience.

12.2.2 The following analyst validation information is maintained for this method in the laboratory QA files.

12.2.3 The analyst must complete the laboratory safety orientation training that includes, but is not limited to, chemicals, PPE requirements, and electrical safety.

12.2.4 The analyst must read and understand this SOP.

12.2.5 The analyst must read and understand the Method used as reference for this SOP.

12.2.6 The analyst must complete a DOC or successfully analyze PT samples annually.

12.2.7 The analyst must complete the Test America Quality Assurance Training.

13.0 Pollution Control

It is TestAmerica's policy to evaluate each method and look for opportunities to minimize waste generated (i.e., examine recycling options, ordering chemicals based on quantity needed, preparation of reagents based on anticipated usage and reagent stability). Employees must abide by the policies in Section 13 of the Corporate Environmental Health and Safety Manual (CW-E-M-001) for "Waste Management and Pollution Prevention."

14.0 Waste Management

14.1 All waste will be disposed of in accordance with Federal, State and Local regulations. Where reasonably feasible, technological changes have been implemented to minimize the potential for pollution of the environment. Employees will abide by this method and the policies in section 13 of the Corporate Safety Manual for "Waste Management and Pollution Prevention."

14.2 The following waste streams are produced when this method is carried out:

Any acidic sample waste generated by the analysis must be disposed of in "A" waste containers.

15.0 References / Cross-References

15.1 EPA Method for Chemical Analyses of Water and Wastes, 1971, method 160.1

15.2 Standard Methods 19th Edition, method 2540C.

16.0 Method Modifications:

Item	Method	Modification
1	160.1	Samples are homogenized by vigorously shaking; a mechanical

		stirrer is not needed
2	160.1	Samples are not evaporated in the steam bath

17.0 Attachments

17.1 Analytical Sequence
17.2 Analytical Batch
17.3 Wet Chemistry Batch Summary Sheet

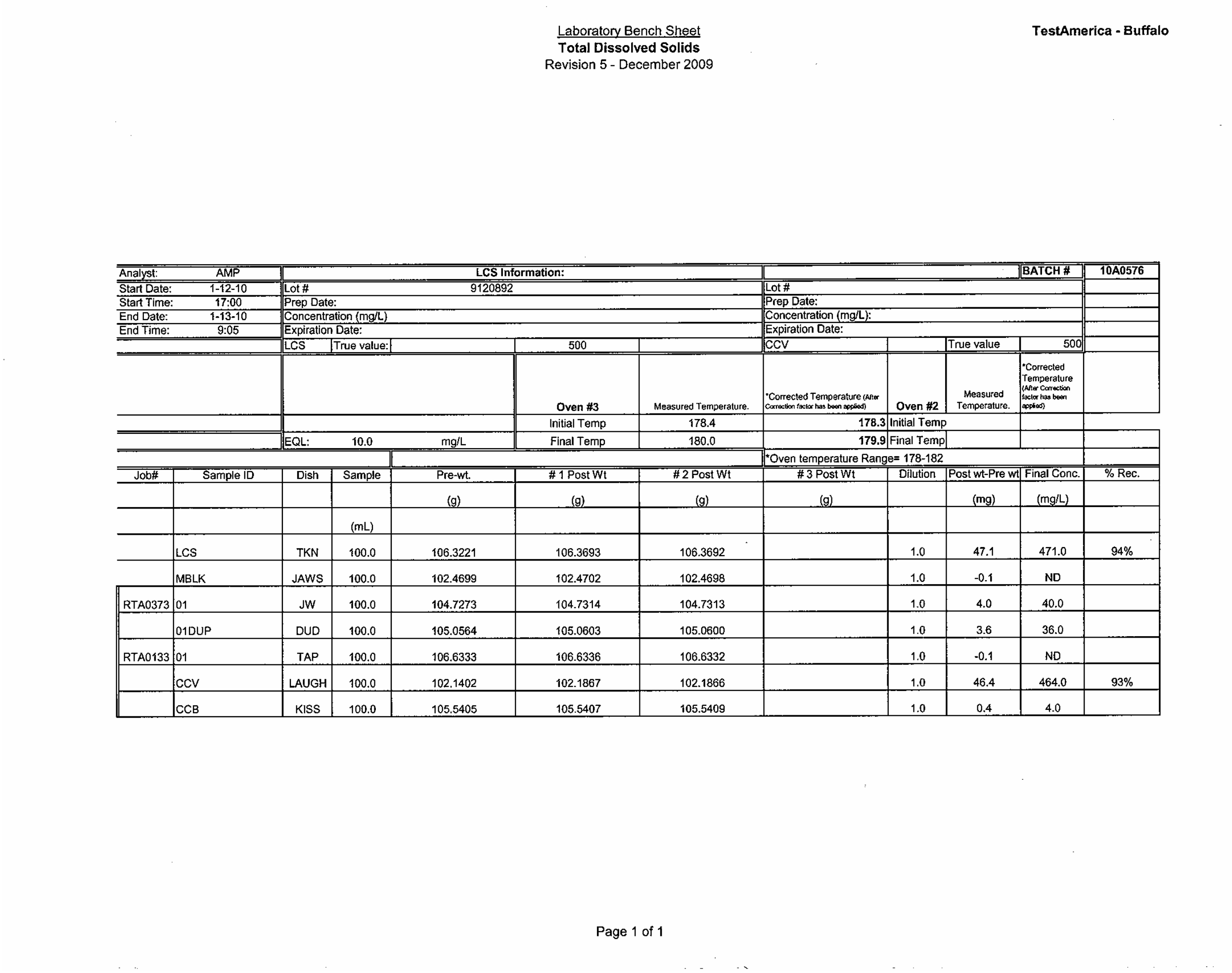
18.0 Revision History

- Revision 1, 03/06/2009
  - Updated attachments 17.2, 17.3
- Revision 2, 01/27/2010
  - Updated attachments

Attachment 17.1
Analytical Sequence

LCS
MBLK
SAMPLE
SAMPLE
SAMPLE
SAMPLE
SAMPLE
SAMPLE
SAMPLE
SAMPLE
SAMPLE
SAMPLE
SAMPLE
SAMPLE
SAMPLE
SAMPLE
SAMPLE
SAMPLE
SAMPLE
SAMPLE
SAMPLE
SAMPLE DUPLICATE (MD)
LCS
MBLK

Attachment 17.2
Analytical Batch



Laboratory Bench Sheet
Total Dissolved Solids
Revision 5 - December 2009

TestAmerica - Buffalo

Analyst: AMP
Start Date: 1-12-10
Start Time: 17:00
End Date: 1-13-10
End Time: 9:05

LCS Information:
Lot # 9120892
Prep Date:
Concentration (mg/L)
Expiration Date:
LCS True value: 500

BATCH # 10A0576
Lot #
Prep Date:
Concentration (mg/L):
Expiration Date:
CCV True value 500

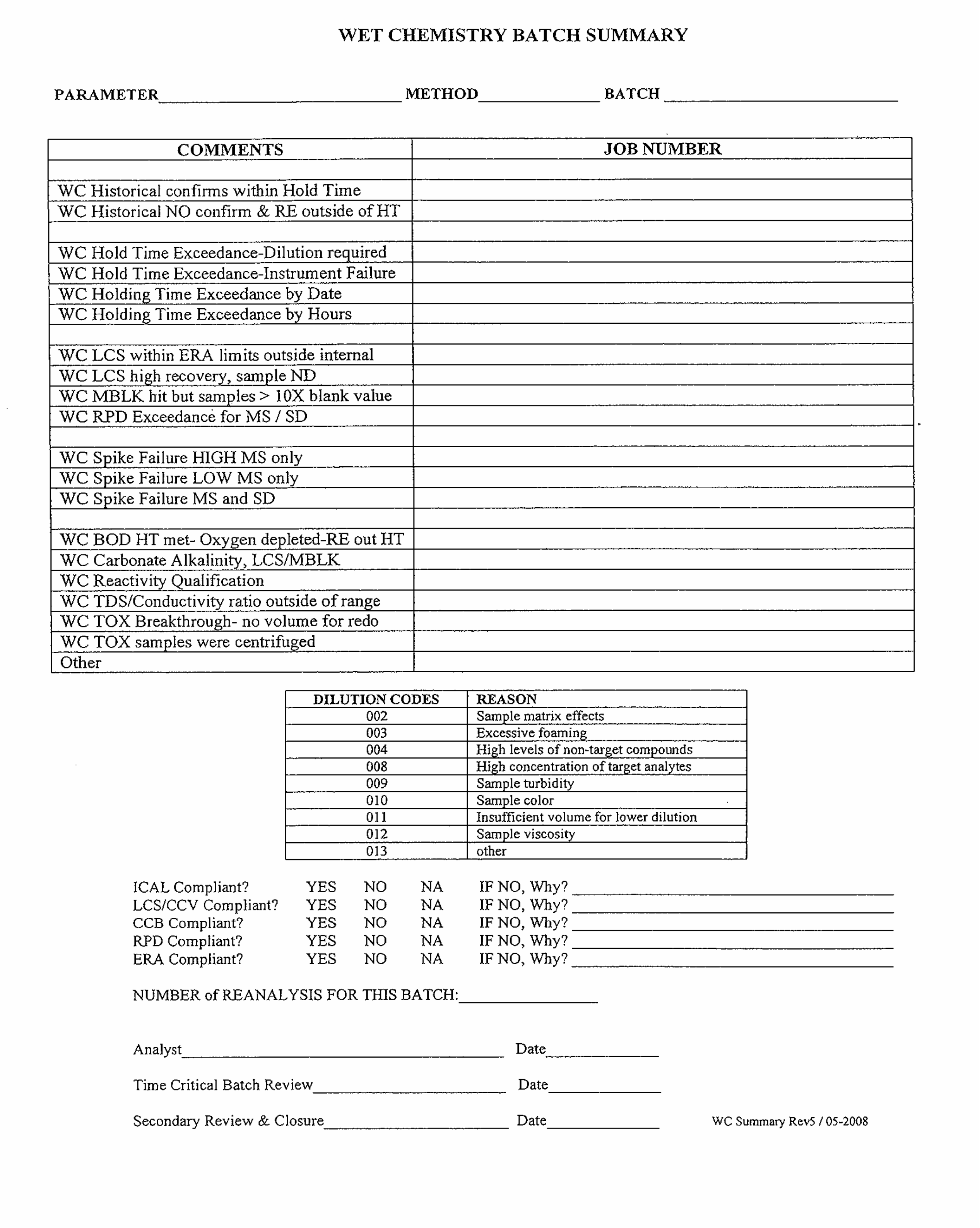
	Oven #3	Measured Temperature.	*Corrected Temperature (After Correction factor has been applied)	Oven #2	Measured Temperature.	*Corrected Temperature (After Correction factor has been applied)
	Initial Temp	178.4	178.3	Initial Temp		
EQL: 10.0 mg/L	Final Temp	180.0	179.9	Final Temp		

*Oven temperature Range= 178-182

Job#	Sample ID	Dish	Sample (mL)	Pre-wt. (g)	# 1 Post Wt (g)	# 2 Post Wt (g)	# 3 Post Wt (g)	Dilution	Post wt-Pre wt (mg)	Final Conc. (mg/L)	% Rec.
	LCS	TKN	100.0	106.3221	106.3693	106.3692		1.0	47.1	471.0	94%
	MBLK	JAWS	100.0	102.4699	102.4702	102.4698		1.0	-0.1	ND	
RTA0373	01	JW	100.0	104.7273	104.7314	104.7313		1.0	4.0	40.0	
	01DUP	DUD	100.0	105.0564	105.0603	105.0600		1.0	3.6	36.0	
RTA0133	01	TAP	100.0	106.6333	106.6336	106.6332		1.0	-0.1	ND	
	CCV	LAUGH	100.0	102.1402	102.1867	102.1866		1.0	46.4	464.0	93%
	CCB	KISS	100.0	105.5405	105.5407	105.5409		1.0	0.4	4.0	

Page 1 of 1

Attachment 17.3
Wet Chemistry Batch Summary Sheet



WET CHEMISTRY BATCH SUMMARY

PARAMETER______________________________ METHOD_______________ BATCH _____________________________

COMMENTS	JOB NUMBER
WC Historical confirms within Hold Time	
WC Historical NO confirm & RE outside of HT	
WC Hold Time Exceedance-Dilution required	
WC Hold Time Exceedance-Instrument Failure	
WC Holding Time Exceedance by Date	
WC Holding Time Exceedance by Hours	
WC LCS within ERA limits outside internal	
WC LCS high recovery, sample ND	
WC MBLK hit but samples > 10X blank value	
WC RPD Exceedance for MS / SD	
WC Spike Failure HIGH MS only	
WC Spike Failure LOW MS only	
WC Spike Failure MS and SD	
WC BOD HT met- Oxygen depleted-RE out HT	
WC Carbonate Alkalinity, LCS/MBLK	
WC Reactivity Qualification	
WC TDS/Conductivity ratio outside of range	
WC TOX Breakthrough- no volume for redo	
WC TOX samples were centrifuged	
Other	

DILUTION CODES	REASON
002	Sample matrix effects
003	Excessive foaming
004	High levels of non-target compounds
008	High concentration of target analytes
009	Sample turbidity
010	Sample color
011	Insufficient volume for lower dilution
012	Sample viscosity
013	other

ICAL Compliant?	YES	NO	NA	IF NO, Why? ______________________________
LCS/CCV Compliant?	YES	NO	NA	IF NO, Why? ______________________________
CCB Compliant?	YES	NO	NA	IF NO, Why? ______________________________
RPD Compliant?	YES	NO	NA	IF NO, Why? ______________________________
ERA Compliant?	YES	NO	NA	IF NO, Why? ______________________________

NUMBER of REANALYSIS FOR THIS BATCH:_________________

Analyst_______________________________________ Date______________

Time Critical Batch Review_______________________ Date______________

Secondary Review & Closure______________________ Date______________

WC Summary Rev5 / 05-2008

Title: FLASH POINT
Method 1010

Once printed, this is considered an uncontrolled document

Approvals (Signature/Date):

Lisa R Matecki 12/28/09
Lisa Matecki Date
Department Manager

Kenneth E. Kasperek 12/28/09
Kenneth Kasperek Date
Health & Safety Manager / Coordinator

Peggy Gray-Erdmann 12/28/09
Peggy Gray-Erdmann Date
Quality Assurance Manager

12/28/09
Christopher Spencer Date
Laboratory Director

Facility Distribution No. ___________ **Distributed To:**________________________

1.0 Scope and Application

1.1. These test methods cover the determination of flash point by closed cup tester to determine the flash point of all types of liquid and soil samples.

1.2. Flash point measures tendency of the sample to form a flammable mixture with aie under controlled laboratory conditions. It is only one of a number of properties, which must be considered in assessing the overall flammability hazard of a material

1.3. Flash point is used in shipping and safety regulations to define flammable and combustible materials. One should consult the particular regulation involved for precise definitions of classes.

1.4. Flash point can indicate the possible presence of highly volatile and flammable materials in a relatively nonvolatile or nonflammable material.

1.5. Analytes, Matrix(s), and Reporting Limits

1.5.1. These methods determine the flash point of fuels, oils, suspensions of solids and liquids including those that tend to form a surface film under test conditions and other liquids of similar viscosity.

1.5.2. If no flash occurs, record the result as ">176°F".

On occasion clients may request modifications to this SOP. These modifications are handled following the procedures outlined in the Quality Assurance Manual.

2.0 Summary of Method

2.1. Method 1010 uses a closed-cup flash point tester technique to determine the flash point of liquids according to ASTM D-93, method A or B. Liquids containing non-filterable, suspended solids shall also be tested using this method. Crushed solid materials or soils can be analyzed in the closed-cup instrument by method B with the stirrer removed or immobilized.

2.2. Sample is heated in an automated closed-cup system at a slow, constant rate with continual stirring if the sample is aqueous. An electrical igniter is automatically dipped into the cup at regular intervals with simultaneous interruption of stirring. The lowest temperature at which application of the coil causes vapors above the sample to ignite is recorded as the flash point. The instrument detects the flash point by the sudden increase in temperature. Flash point results are automatically corrected for the barometric pressure by a sensor and software in the instrument.

3.0 Definitions

3.1. Flash point is the lowest temperature corrected to a barometric pressure, at which application of a test flame causes the vapor of a specimen to ignite under specified conditions of the test.

3.2. Standard definitions are found in the Laboratory Quality Manual.

4.0 Interferences

4.1. Low boiling oils or neat liquids may boil over at temperatures below the 176 degree Fahrenheit limit. If such a sample fails to flash below its boiling point, a result of ">(b.p. Temperature)" may be assigned to the sample.

4.2. Drafts, incorrect starting temperature, incorrect heating rate and incorrect stirring rate will affect the observed flash point. The automated closed-cup instrument is programmed to operate within specifications of the method chosen (A or B) on the keyboard at the beginning of the test.

5.0 Safety

Employees must abide by the policies and procedures in the Corporate Safety Manual, Radiation Safety Manual and this document.

This procedure may involve hazardous material, operations and equipment. This SOP does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of the method to follow appropriate safety, waste disposal and health practices under the assumption that all samples and reagents are potentially hazardous. Safety glasses, gloves, lab coats and closed-toe, nonabsorbent shoes are a minimum.

Specific Safety Concerns or Requirements

5.1. In the event a sample ignites in the test apparatus, do not attempt to remove the sample. Turn off the apparatus and flame. The flame should go out when the cup is closed. If this does not happen the flame may be extinguished by covering the sample with on-flammable material. After the apparatus has cooled the sample may be removed.

5.2. Be sure that this test is not conducted in an area where there may be vapors present from highly flammable substances, such as ether.

5.3. **Absolutely no flammable materials should be in the hood with the flash point apparatus.**

5.4. Fire extinguisher will be located near the flash point apparatus.

5.5. Be cautious with samples that exhibit flammable characteristics (e.g., smells like solvent) when conducting this test.

5.6. Take appropriate safety precautions during the initial application of the test flame, since samples containing low-flash material can give an abnormally strong flash when the test flame is first applied.

5.2 Primary Materials Used

The following is a list of the materials used in this method, which have a serious or significant hazard rating. **Note: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the MSDS for each of the materials listed in the table.** A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the MSDS for each material before using it for the first time or when there are major changes to the MSDS.

Material	**Hazards**	**Exposure Limit**[1]	**Signs and symptoms of exposure**
P-Xylene	Flammable Irritant	100 ppm-TWA	Inhalation of vapors may be irritating to the nose and throat. Inhalation of high concentrations may result in nausea, vomiting, headache, ringing in the ears and severe breathing difficulties, which may be delayed in onset. High vapor concentrations are anesthetic and central nervous system depressants. Skin contact results in loss of natural oils and often results in a characteristic dermatitis. May be absorbed through the skin. Vapors cause eye irritation. Splashes cause severe irritation, possible corneal burns and eye damage.
[1] – Exposure limit refers to the OSHA regulatory exposure limit.			
*Always add acid to water to prevent violent reactions.			

6.0 Equipment and Supplies

6.1. Instrumentation

6.1.1. Herzog Model HFP 339, Automated Closed-Cup flash point tester. The apparatus is set up on a level table in a draft-free area.
6.1.2. The thermometer is specific to the instrument and must be purchased from the vendor. It is calibrated annually against a NIST-certified thermometer. The tester is equipped with a barometric pressure sensor and automatically corrects the observed flash point.
6.1.3. The instrument also requires a flash point sensor, an electrical igniter, a sample cup and a lid that seals the cup. The stir paddle is attached to the cup lid.

7.0 Reagents and Standards

7.1. P-Xylene; Flash point = 81 $\pm$ 2°F (27.2 + 1.1^{o}C)

8.0 Sample Collection, Preservation, Shipment and Storage

Matrix	Sample Container	Min. Sample Size	Preservation	Holding Time	Reference
Waters	Glass	100g	None Cool 4 ± 2°C	150 days	ASTM D93-80, SW-846 3rd edition
Soils	Glass	100g	None Cool 4 ± 2°C	150 days	ASTM D93-80

8.1. Samples are to be preserved by cooling to 4 ±2°C and stored in glass containers. Do not store samples in plastic container, since volatile material may diffuse through the walls of the enclosure.

8.2. Erroneously high flash points may be obtained if precautions are not taken to avoid the loss of volatile material. Do not open containers unnecessarily and make a transfer unless the sample temperature is at least the equivalent of 18°F below the expected flash point. Do not use samples from leaky containers for these test methods.

9.0 Quality Control

9.1. Begin and end each run of 20 samples or fewer with analysis of p-Xylene LCS. The obtained value for p-Xylene should be within 79-83°F.

9.2. Run at least one duplicate every 20 samples or fewer. The relative percent difference between duplicate analyses should be <10%.

9.3. All samples exhibiting a Flash point must be run in duplicate.

9.4. **Sample QC** - The following quality control samples are prepared with each batch of samples.

Quality Controls	Frequency	Control Limit
Laboratory Control Sample (LCS)	1 in 20 or fewer samples	81 ± 2 degrees
Sample Duplicate [1]	1 in 20 or fewer samples	RPD <10%

[1] The sample selection for duplicates is random, unless specifically requested by a client or if the sample flashes below 176°F.

9.5. Instrument QC

Not Applicable

10.0 Procedure

10.1. Thoroughly clean and dry all parts of the cup and its accessories before starting the test, being sure to remove any solvent. It is imperative to examine the Herzog electrical igniter prior to operation for signs of mechanical or physical damage. A damaged igniter could result in a high flash point. Do not clean the Herzog keyboard with any kind of solvent.

10.2. Fill the cup to the level indicated by the filling mark inside of the cup, approximately 70 mLs.

10.3. Mix the sample well. Viscous samples may be warmed but only enough to mix and dispense the sample. Cool the sample to at least 20°F below the expected flash point for the closed cup testing.

10.4. Fill the cup to the mark.

10.5. Place and lock the lid on the cup and insert the thermometer. Insert the thermometer directly into any solid sample. Also insert the thermal detector and attach the drive rod.

10.6. If a liquid sample is being analyzed, attach the stirring mechanism. The instrument will automatically stir the sample at the appropriate rate if the stirring mechanism is assembled. **Do not attempt to stir soil samples.**

Note: Refer to the instrument manual for more detailed instructions, if needed.

10.7. The display should read “Ready”. If not, press the “Menu” key until “Ready” appears on the screen. Press “Start”. Enter the sample number when prompted by the instrument.

10.8. Press “Start” again. You will be asked to enter an expected flash point. For the P-Xylene standard, enter the lowest possible temperature as the expected flash point. If no flash is expected, enter “140°F” as the expected flash point. If a sample apparently contains a solvent, a lower temperature may be chosen. The igniter is applied beginning 41°F below the expected flash point and continues for 36°F beyond the set point, unless a flash is detected. If 140°F is used as the expected flash point, the test will begin at 99°F and end at 176°F or when a flash is detected, whichever is lower.

Note: 140°F is the SW-846 ignitability hazard point.

10.9. After entering the expected flash point, press “Start” once again. You will be asked whether or not to start the test. Enter “Yes”. After a brief self-check, the instrument will automatically apply the ignition coil to the sample every 2 °F. The rate of temperature rise and the stirring speed are also pre-programmed.

10.10. A buzzer will sound if a flash point is detected and the flash point will be displayed on the screen. If no flash point is detected up to the temperature of 176°F, the screen will read “error”. The cooling cycle will automatically begin when the test is over.

10.11. Sample Preparation

See section 10.0

10.12. Calibration

Not applicable

11.0 Calculations / Data Reduction

11.1. Record the sample number, the temperature at which the test was begun and the observed flash point. The observed flash point is already corrected for barometric pressure.

11.2. Accuracy

The LCS must fall within 2 degrees of the expected Flash point for the standard

11.3. Precision (RPD)

Matrix Duplicate (MD)

$$\mathrm{RPD} = \frac{|x_1 - x_2|}{\left(\frac{x_1 + x_2}{2}\right)} \text{ x } 100$$

where:
x_1 = analytical % recovery
x_2 = replicate % recovery

12.0 Method Performance

12.1. Method Detection Limit Study (MDL)

Not Applicable

12.2. Demonstration of Capabilities

12.2.1. A one–time initial demonstration of performance must be generated.

12.3. Training Requirements

12.3.1. The supervisor has the responsibility to ensure that an analyst who has been properly trained in its use and has the required experience performs this procedure

12.3.2. The following analyst validation information is maintained for this method in the laboratory QA files.

12.3.2.1. The analyst must read and understand this SOP.

12.3.2.2. The analyst must complete a DOC or successfully analyze PT samples annually.

12.3.2.3. The analyst must complete the TestAmerica Quality Assurance Training.

13.0 Pollution Control

It is TestAmerica’s policy to evaluate each method and look for opportunities to minimize waste generated (i.e., examine recycling options, ordering chemicals based on quantity needed, preparation of reagents based on anticipated usage and reagent stability).

14.0 Waste Management

Waste management practices are conducted consistent with all applicable rules and regulations. Excess reagents, samples and method process wastes are disposed of in an accepted manner. Waste description rules and land disposal restrictions are followed. Waste disposal procedures are incorporated by reference to section 13 of the Corporate Safety Manual for "Waste Management and Pollution Prevention".

15.0 References / Cross-References

15.1. D93-80 Test Methods for Flash points by Pensky- Martens closed tester, American Society for Testing and Materials. 1916 Race St., Philadelphia, PA 19103, 04.99, 1986.

15.2. Method 1010, "Pensky-Martens Closed Cup Method for Determining Ignitability", Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, Final Update II, September 1994.

16.0 Method Modifications

Item	**Method**	**Modification**
NA	**NA**	**NA**

17.0 Attachments

17.1 Analytical Run sequence
17.2 Analytical Batch
17.3 Wet Chemistry Batch Summary & Data Review Checklist

18.0 Revision History

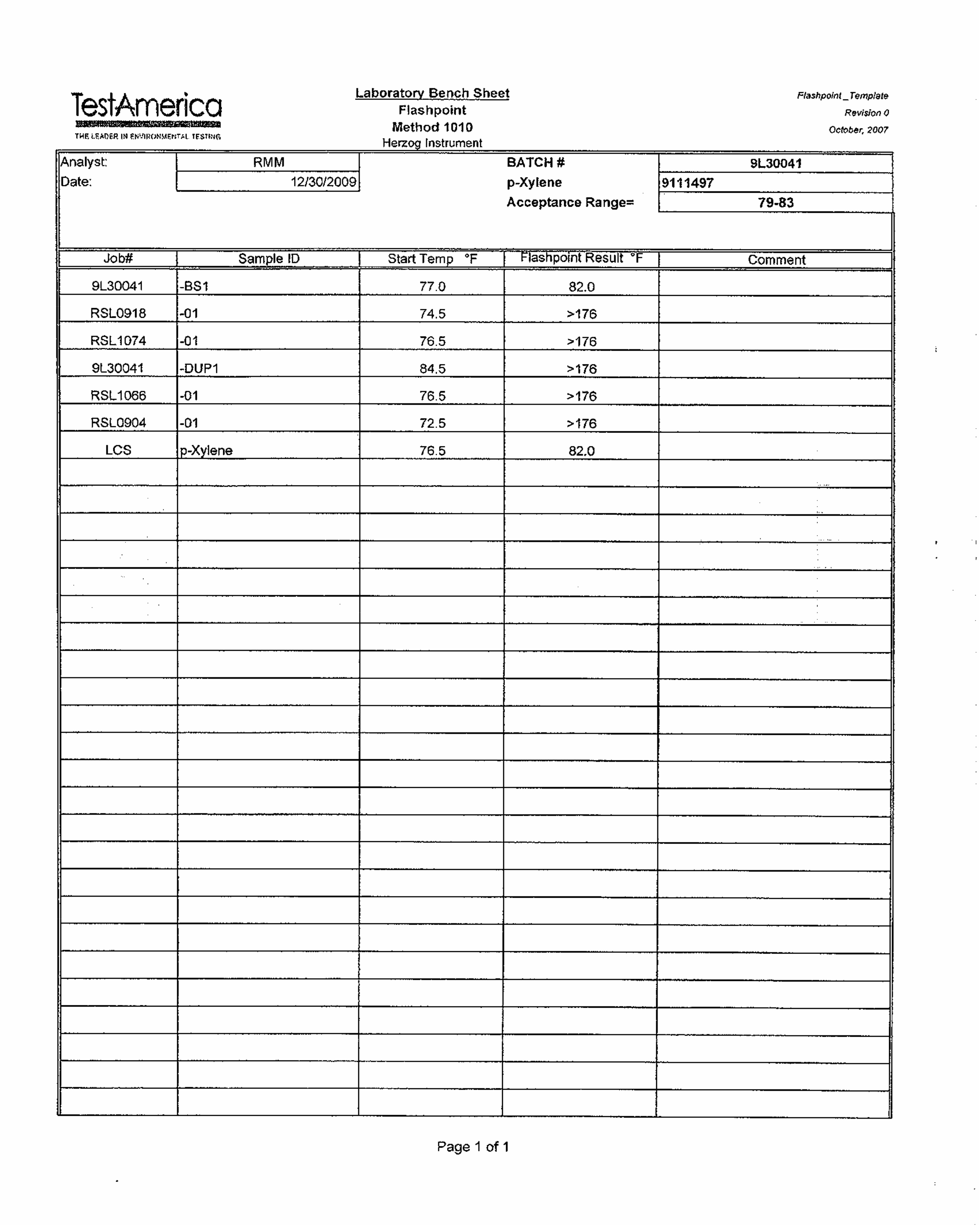
- Revision 1, dated 12/31/2009
    - Updated Attachment 17.2-17.3

- Revision 0, dated 12/17/2007
    - Integration for TestAmerica and STL operations.
    - Revised SOP for new automated flash point tester
    - Updated Attachment 2

Attachment 17.1
Analytical Sequence

LCS
SAMPLE
SAMPLE
SAMPLE
SAMPLE
SAMPLE
SAMPLE
SAMPLE
SAMPLE
SAMPLE
SAMPLE
SAMPLE
SAMPLE
SAMPLE
SAMPLE
SAMPLE
SAMPLE
SAMPLE
SAMPLE
SAMPLE
SAMPLE DUP
LCS

Attachment 17.2
Analytical Batch



TestAmerica
THE LEADER IN ENVIRONMENTAL TESTING

Laboratory Bench Sheet
Flashpoint
Method 1010
Herzog Instrument

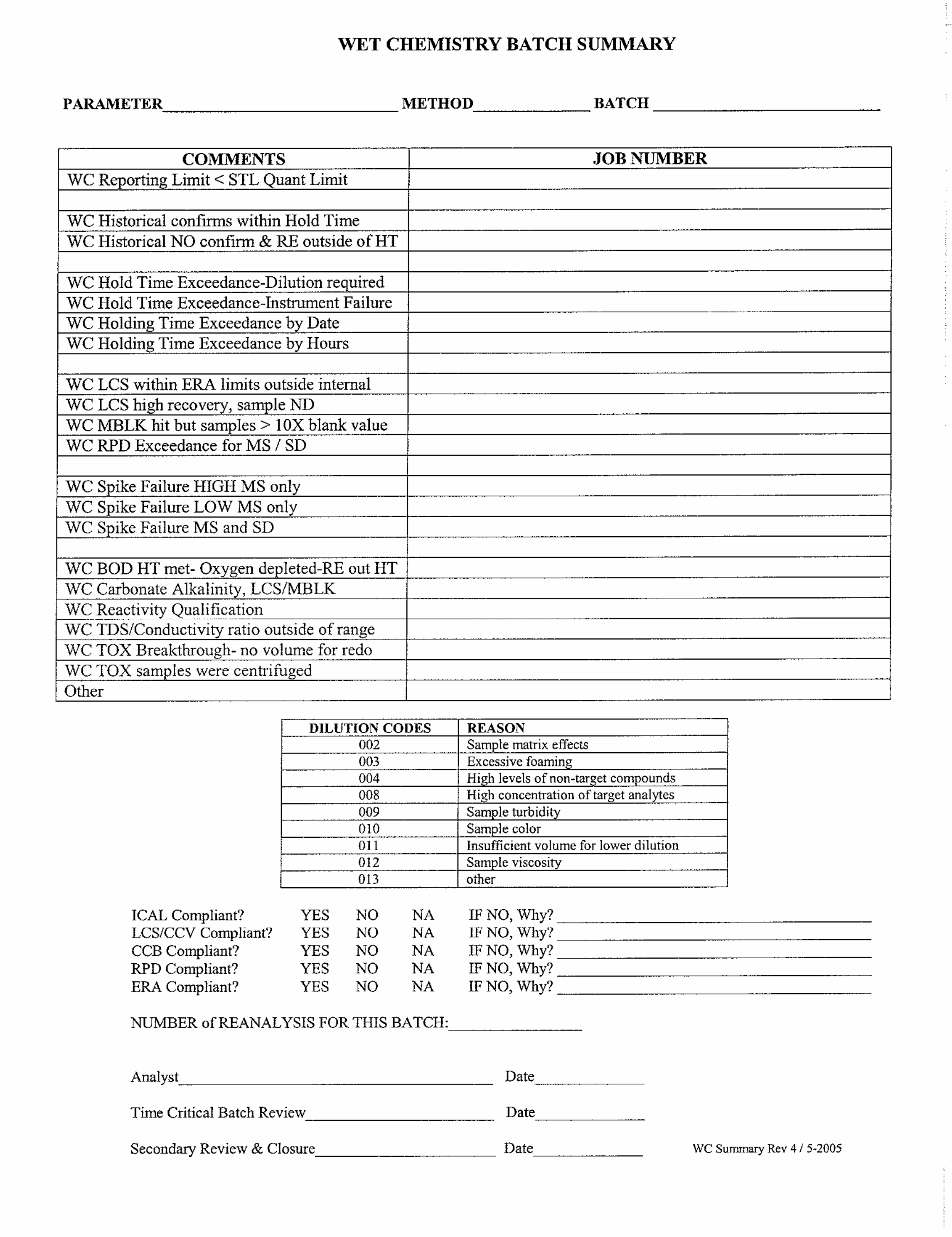
Flashpoint_Template
Revision 0
October, 2007

Analyst:	RMM	**BATCH #**	**9L30041**
Date:	12/30/2009	**p-Xylene**	**9111497**
		Acceptance Range=	**79-83**

Job#	Sample ID	Start Temp °F	Flashpoint Result °F	Comment
9L30041	-BS1	77.0	82.0	
RSL0918	-01	74.5	>176	
RSL1074	-01	76.5	>176	
9L30041	-DUP1	84.5	>176	
RSL1066	-01	76.5	>176	
RSL0904	-01	72.5	>176	
LCS	p-Xylene	76.5	82.0	

Page 1 of 1

Attachment 17.3
Wet Chemistry Batch Summary



WET CHEMISTRY BATCH SUMMARY

PARAMETER_____________________________ **METHOD**______________ **BATCH** ____________________________

COMMENTS	JOB NUMBER
WC Reporting Limit < STL Quant Limit	
WC Historical confirms within Hold Time	
WC Historical NO confirm & RE outside of HT	
WC Hold Time Exceedance-Dilution required	
WC Hold Time Exceedance-Instrument Failure	
WC Holding Time Exceedance by Date	
WC Holding Time Exceedance by Hours	
WC LCS within ERA limits outside internal	
WC LCS high recovery, sample ND	
WC MBLK hit but samples > 10X blank value	
WC RPD Exceedance for MS / SD	
WC Spike Failure HIGH MS only	
WC Spike Failure LOW MS only	
WC Spike Failure MS and SD	
WC BOD HT met- Oxygen depleted-RE out HT	
WC Carbonate Alkalinity, LCS/MBLK	
WC Reactivity Qualification	
WC TDS/Conductivity ratio outside of range	
WC TOX Breakthrough- no volume for redo	
WC TOX samples were centrifuged	
Other	

DILUTION CODES	REASON
002	Sample matrix effects
003	Excessive foaming
004	High levels of non-target compounds
008	High concentration of target analytes
009	Sample turbidity
010	Sample color
011	Insufficient volume for lower dilution
012	Sample viscosity
013	other

ICAL Compliant?	YES	NO	NA	IF NO, Why? ______________________
LCS/CCV Compliant?	YES	NO	NA	IF NO, Why? ______________________
CCB Compliant?	YES	NO	NA	IF NO, Why? ______________________
RPD Compliant?	YES	NO	NA	IF NO, Why? ______________________
ERA Compliant?	YES	NO	NA	IF NO, Why? ______________________

NUMBER of REANALYSIS FOR THIS BATCH:_________________

Analyst____ Date______________

Time Critical Batch Review________________________ Date______________

Secondary Review & Closure________________________ Date______________

WC Summary Rev 4 / 5-2005

AQUEOUS SEPARATORY FUNNEL EXTRACTION PROCEDURE (METHOD No. 3510C)

Once printed, this is considered an uncontrolled document

Approvals (Signature/Date):

Shanna Snider
03/12/10
Shanna Snider — Date
Department Manager

Kenneth E. Kasperek
03/12/10
Kenneth Kasperek — Date
Technical Director

Peggy Gray-Erdmann
03/12/10
Peggy Gray-Erdmann — Date
Quality Assurance Manager

03/12/10
Christopher Spencer — Date
Laboratory Director

Facility Distribution No. ____________ **Distributed To:**_________________________

1.0 Scope and Application

This method is used to extract a broad range of organic compounds from aqueous samples for analysis by either GC or GCMS. This method also describes concentration techniques, which prepare the extract for the appropriate analysis.

1.1 Analytes, Matrix(s), and Reporting Limits

1.2 Matrices: aqueous samples (water)

1.3 Reporting limit: N/A

1.4 On occasion clients may request modifications to this SOP. These modifications are handled following the procedures outlined in the Quality Assurance Manual.

2.0 Summary of Method

2.1 A measured volume of aqueous sample, approximately 1 liter, is extracted with methylene chloride at a specified pH using separatory funnel extraction. The extract is dried through activated anhydrous sodium sulfate, concentrated using a Nitrogen blowdown technique, and if necessary solvent exchanged into a solvent suitable for its cleanup or analysis.

3.0 Definitions

3.1 Standard definitions are found in Section 3.0 of the Laboratory Quality Manual.

3.2 Solvent exchange: The process of exchanging the solvent of the sample extract from the extraction solvent (usually methylene chloride) to the final volume solvent (usually hexane).

4.0 Interferences

4.1 Method interference may be caused by contaminants in solvents, reagents, glassware and other sample processing hardware that lead to discrete artifacts or elevated baselines in gas chromatograms. All these materials must be routinely demonstrated to be free from interference under the conditions of the analysis, by analyzing reagent blanks.

4.2 Matrix interference may be caused by contaminants that are co-extracted from the sample.

4.3 Glassware used for water extractions is kept separate from soil glassware to prevent cross-contamination.

5.0 Safety

5.1 Employees must abide by the policies and procedures in the Corporate Safety Manual, Radiation Safety Manual and this document.

5.2 This procedure may involve hazardous material, operations and equipment. This SOP does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of the method to follow appropriate safety, waste disposal and health practices under the assumption that all samples and reagents are potentially hazardous. Safety glasses, gloves, lab coats and closed-toe, nonabsorbent shoes are a minimum.

5.3 Specific Safety Concerns or Requirements

5.3.1 The use of separatory funnels to extract aqueous samples with Methylene Chloride creates excessive pressure very rapidly. Initial venting should be done immediately after the sample container has been sealed and inverted. Vent the funnel into the hood away from people and other samples.

5.3.2 All parameters of this extraction must be performed in an operational fume hood or within an extraction apparatus that is ventilated by the fume hood system. The following analytes have been tentatively classified as known or suspected, human or mammalian carcinogens: benzo(a)anthracene, benzidine, 3,3'dichlorobenzindine, benzo(a)pyrene, alpha-BHC, beta-BHC, gamma-BHC, delta-BHC, dibenz(a,h)anthracene, N-nitrosodimethylamine, 4,4'-DDT, and polychlorinated biphenyl compounds. Primary standards of these toxic compounds should be prepared in hood.

5.3.3 Safety glasses, gloves, and lab coats must be worn at all times. Nitrile gloves should be used when performing this extraction. Latex and vinyl gloves provide no significant protection against the organic solvents used in this SOP, and should not be used.

5.3.4 All solvents, reagents, and standards must be handled inside a fume hood and with proper personal safety equipment due to their hazardous properties. All samples must be opened inside a fume hood due to their unknown hazardous properties.

5.4 Primary Materials Used

The following is a list of the materials used in this method, which have a serious or significant hazard rating. **Note: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the MSDS for each of the materials listed in the table.** A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the MSDS for each material before using it for the first time or when there are major changes to the MSDS.

Material (1)	**Hazards**	**Exposure Limit (2)**	**Signs and symptoms of exposure**
Hexane	Flammable Irritant	500 ppm-TWA	Inhalation of vapors irritates the respiratory tract. Overexposure may cause lightheadedness, nausea, headache, and blurred vision. Vapors may cause irritation to the skin and eyes.
Methylene Chloride	Carcinogen Irritant	25 ppm-TWA 125 ppm-STEL	Causes irritation to respiratory tract. Has a strong narcotic effect with symptoms of mental confusion, light-headedness, fatigue, nausea, vomiting and headache. Causes irritation, redness and pain to the skin and eyes. Prolonged contact can cause burns. Liquid degreases the skin. May be absorbed through skin.
Sulfuric Acid	Corrosive Oxidizer Dehydrador	1 mg/m^3	This material will cause burns if comes into contact with the skin or eyes. Inhalation of vapors will cause irritation of the nasal and respiratory system.
Sodium Hydroxide	Corrosive Poison	2 ppm, 5 mg/m^3	This material will cause burns if comes into contact with the skin or eyes. Inhalation of Sodium Hydroxide dust will cause irritation of the nasal and respiratory system.

1 – Always add acid to water to prevent violent reactions.
2 – Exposure limit refers to the OSHA regulatory exposure limit.

6.0 Equipment and Supplies

6.1 1 liter graduated cylinder
6.2 250 mL graduated cylinder
6.3 2 liter Teflon separatory funnels, stopcocks, and caps
6.4 Syringes
6.5 Turbovaps and turbovap vessels
6.6 16 oz. French squares
6.7 Steel funnels
6.8 Glass wool
6.9 Disposable pipettes and bulbs
6.10 2 mL vials and caps (amber or clear depending on application)
6.11 40 ml. Glass vials with caps and (PTFE)- lined cap inserts
6.12 Vial crimpers
6.13 Wide range pH paper
6.14 Centrifuge and centrifuge tubes
6.15 Automatic separatory funnel rotators
6.16 Narrow range pH paper
6.17 Aluminum weigh dishes

7.0 Reagents and Standards

7.1 **Note:** All solvents are pesticide grade or equivalent
7.2 Methylene chloride delivered in cycletainers
7.3 Hexane delivered in cycletainers
7.4 Acetone delivered in cycletainers
7.5 Methanol
7.6 10N sodium hydroxide
7.7 1:1 sulfuric acid
7.8 Concentrated Sulfuric Acid
7.9 Anhydrous granular sodium sulfate. **Note:** Sodium sulfate must be baked in a 400°C oven for a minimum of 4 hours before use, or alternately may be purchased pre-baked from Jost chemical.
7.10 Deionized water and/or carbon filtered (volatile free) water
7.11 Surrogate and spike solutions appropriate to the final determinative procedures as assigned by test profile.

8.0 Sample Collection, Preservation, Shipment and Storage

Sample container, preservation techniques and holding times may vary and are dependent on sample matrix, method of choice, regulatory compliance, and/or specific contract or client requests. Listed below are the holding times and the references that include preservation requirements.

Test	Sample Container	Min. Sample Size	Preservation	Holding Time	Reference
8270	1-Liter glass amber w/ Teflon lid	1-Liter	Cool to 4˚C	7 days	SW846
625	1-Liter glass amber w/ Teflon lid	1-Liter	Cool to 4˚C	7 days	SW846
548.1	250 mL amber	250 mLs	Cool to 4˚C	14 days	EPA 548.1
OLM	1-Liter glass amber w/ PTFE screw top	1-Liter	Cool to 4˚C	7 days	OLM 4.3
CLP	1-Liter glass amber w/ PTFE screw top	1-Liter	Cool to 4˚C	7 days	OLM 4.3
TCLP	1-Liter amber w/ Teflon screw top	250 mLs per test	Cool to 4˚C	7 days	EPA 1311
608pcb	1-Liter glass amber w/ Teflon lid	1-Liter	Cool to 4˚C	365 days	SW846
608pest	1-Liter glass amber w/ Teflon lid	1-Liter	Cool to 4˚C	7 days	SW846
8081	1-Liter glass amber w/ Teflon lid	1-Liter	Cool to 4˚C	7 days	SW846
8082	1-Liter glass amber w/ Teflon lid	1-Liter	Cool to 4˚C	7 days	SW846
31013	1-Liter Glass amber	1-Liter	None Required	7 days	NYSDOH
8015	1-Liter Glass amber	1-Liter	None Required	7 days	NYSDOH
8151	1-Liter glass amber w/ Teflon lid	1-Liter	Cool to 4˚C	7 days	SW846

8.1 Typical method holding time for water samples is seven days from sampling. However, the client may impose a more strict time constraint.

8.2 Clients may request CLP QC requirements without Continuous Liquid/Liquid extraction technique.

9.0 Quality Control

The following quality control samples are prepared with each batch of samples. All reagent blanks, method spike blanks, matrix spikes and matrix spike duplicates will undergo the same procedure as the samples.

Quality Controls	Frequency	Control Limit
Method Blank (BLK)	1 in 20 or fewer samples	< Rpt. Limit
Laboratory Control Standard (BS)[1]	1 in 20 or fewer samples	Statistical Limits [4]
Matrix Spike (MS)[2]	1 in 20 or fewer samples	Statistical Limits [4]
Spike Duplicate (SD)[2]	1 in 20 or fewer samples	Statistical Limits [4]
Surrogates	every sample[3]	Statistical Limits [4]

[1] Laboratory Control Duplicate Standard (BSD) is performed only when insufficient sample is available for the MS/SD or when requested by the client/project/contract.

[2] The sample selection for MS/SD are randomly selected, unless specifically requested by a client or predetermined by the extraction lab.

[3] Analytical and QC samples (BLK, BS/BSD, MS/SD)

[4] Statistical control limits are updated annually and are updated into LIMS.

10.0 Procedure

10.1 Assemble a pre-rinsed 2L separatory funnel, stopcock and stopper as well as all other extraction supplies and glassware. All glassware is pre-rinsed with 12.5% nitric acid, DI, methanol and methylene chloride.

10.2 Label each separatory funnel with TestAmerica Laboratories vial label that corresponds with the sample I.D. number.

10.3 Make a powder funnel by placing a glass wool plug in a steel funnel, and fill the funnel 2/3 full with baked granular sodium sulfate. As a precaution the sodium sulfate can be rinsed, in the funnel, with 20-30ml of methylene chloride and allowed to drain. Discard this methylene chloride rinse. Place the powder funnel into a clean French square labeled with TestAmerica Laboratories vial labels.

If humidity is a concern, push the end of the powder funnel through a paper napkin to catch any water that may condense on the sides of the funnel during the extraction procedure.

10.4 Obtain the designated spike and surrogate solutions (See Table 4) and allow them to come to room temperature.

10.5 Using a disposable pipette and wide range pH paper, test and record the initial pH of the sample. Since the sample pH will be unknown, wide range pH paper may be used for the initial process. Narrow range pH paper is used later to verify that sample pH is in the correct range for required test. Refer to table 3 for specific pH ranges.

<u>All pH measurements are to be made by the following method</u>:
Dip the back end of a glass disposable pipette into the liquid.
Tap this end onto a piece of wide-range pH paper.
Record the measurement and discard the paper and pipette.

10.6 Visually inspect samples.
Any sample that has an oil layer needs the oil removed. This can be accomplished by using a disposable pipette (if the oil layer is minimal). If there is significant oil a glass 2-L separatory funnel can be used to drain the aqueous portion. ALL GLASSWARE ASSOCIATED WITH THESE SAMPLES SHOULD BE SEGRAGATED TO PREVENT CONTAMINATION.

10.6.1 If there is a large amount of sediment, the sample volume should be measured by pouring the sample into a pre-rinsed graduated cylinder, leaving as much of the sediment in the sample bottle as possible. Record the sample volume and transfer the sample into the corresponding labeled separatory funnel.

10.6.2 If the sample is relatively free of sediment and not received in ESS bottles mark the meniscus on the bottle. Once the sample is transferred to the corresponding sep funnel, the empty sample bottle is filled to the meniscus mark with tap water. The tap water is then transferred to a graduated cylinder, and the volume is recorded on the bench paperwork.

10.6.3 If the sample is relatively free of sediment and received in ESS bottles hold the volume template to the bottle and read and record the volume at the meniscus.

10.6.4 Record the appearance of the samples on the bench sheet. It may be necessary to pour off a small volume of the sample into the corresponding sep funnel.

10.7 Add the appropriate spike to QC samples except BLK and surrogate solutions to all samples.

10.7.1 Samples that have been transferred already to their separatory funnels have the surrogate added directly to the separatory funnel.

10.7.2 Samples remaining in their sample bottles have the surrogate added directly to the sample bottle, recapped and shaken.

10.7.3 It is important to mark the labels of each sample and blank accordingly when adding spikes and surrogates to avoid error. Once a surrogate has been added (whether it is to the original sample jar or the separatory funnel should the occasion warrant it), an “X” must be drawn on the label affixed to the separatory funnel. After a spike has been added, circle the “X” on the label immediately.

10.8 Transfer all samples to their corresponding labeled separatory funnels.

10.8.1 Measure 1 Liter of appropriate extraction water (see Table 3) for all QC samples (BS,BSD,and BLK). All batch QC will undergo the same procedure as batch samples.

10.8.2 Rinse the internal walls of sample bottle with Methylene Chloride. Transfer this Methylene Chloride rinse to the sep funnel (the first 60 mL aliquot of methylene chloride may be used for this rinse. See section 10.10).

10.9 Verify that samples are in correct pH range using narrow range pH paper.

10.9.1 Make adjustments to sample pH as needed using 1:1 H_2SO_4 or 10N NaOH.. If pH adjustment is required the separatory funnel must be capped and shaken for a moment to ensure homogenization of the newly added acid or base. Once shaken, the sample pH can be verified using narrow range pH paper.

10.9.2 Record any pH adjustments that have been made on the bench sheet.

10.10 Add 60 ml of methylene chloride to the separatory funnel

The method for the delivery of solvent to the extraction container will be as follows: Obtain Teflon graduated cylinders and measure the necessary amount of solvent. Pour this solvent from the graduated cylinder to the extraction vessel.

10.11 Seal the separatory funnels and rotate a few times. Vent all separatory funnels and rotate for an additional 2 minutes.

10.12 Allow the organic layer to separate from the water for a minimum of 10 minutes.

10.12.1 If an emulsion occurs so that it is 1/3 the solvent layer, it must be centrifuged.

10.12.2 Following centrifugation of the emulsion, place the aqueous layer back in the separatory funnel and pour the $MeCl_2$ layer into the powder funnel.

10.12.3 Rinse the centrifuge tube with 5-10 mLs of $MeCl_2$ and add this to the powder funnel to complete the transfer.

10.12.4 Record on bench sheet that sample required to be centrifuged.

10.13 Wet the powder funnel with $MeCl_2$ prior to draining. Drain the $MeCl_2$ layer through the powder funnel.

10.14 Rinse each powder funnel with approximately 20-30ml methylene chloride.

10.15 Perform two more extractions with 60ml methylene, shaking or rotating for 1 minute each time. Rinse the powder funnel with 10 – 20 mLs of $MeCl_2$ after the third drain.

10.16 Pour the extracted samples into the satellite "W" waste containers. Adjust the pH of each container to between 5 and 9 and discard in the main W-waste drum.

10.17 Concentrate the extract using the Turbovaps.

10.17.1 Pour approximately 150ml of extract into pre-rinsed, labeled Turbovap vessel and place in the Turbovap.

10.17.2 Add the remaining extract volume to the vessel as it is concentrating. Rinse the French square with 10-20 mLs of MeCl2 and add to Turbovap vessel.

10.18 Keep the Turbovap nitrogen pressure as high as possible without splashing the extract.

Water temperature should be maintained between 30-40°C.

Splashing of the extracts must be avoided since cross-contamination could occur. Aluminum tins may be used to cover turbovap vessels to assist in the prevention of cross contamination and analyte loss.

During concentration, periodically rinse the walls of the turbovap with a small amount of $MeCl_2$ to push analytes back into solvent.

10.19 Remove the turbovap vessel from the Turbovap as soon as the 1.0 mL calibration mark is reached.

Samples should be closely monitored to ensure that time spent in the Turbovap is minimized.

Over concentrating samples results in the loss of analytes.

10.20 Solvent exchange samples to hexane if required by adding 20-30 mls of hexane and concentrating back to the 1.0ml mark. Refer to Table 3 for specific test requirements.

10.21 Homogenize the extract and perform any necessary cleanup procedures. Adjustment to the appropriate final volume may be done either before or after any required cleanup procedures, depending on the procedures to be performed. Reference the individual cleanup SOP for that information.

10.22 Bring samples to appropriate final volumes.

10.22.1 Final volume of 1.0mL: Concentrate the extract down to the calibrated 1-mL mark on the Turbovap vessel. Transfer the entire extract into a 2-mL vial using a 9 inch disposable pipette.

10.22.2 Final volume of 2.0mL: Concentrate the extract down to the calibrated 1-mL mark on the Turbovap vessel, add 1.0mL of final solvent using a repipetter. Transfer approximately 1.0mL to a labeled 2-mL vial using a 9 inch disposable pipette. Discard remaining volume.

10.22.3 Final volume of 10.0mL: Concentrate the extract down to the calibrated 1-mL mark on the Turbovap vessel, add 9.0mL of the final solvent to the vessel using a repipetter. Transfer 10.0mL into a labeled 40mL vial. Using a disposable pipette, transfer approximately 1.0mL to a labeled 2-mL vial. Retain extra volume for a period no less than 30 days.

10.23 Pesticide (608PEST, 8081) Polychlorinated Biphenyls (608PCB, 8082)

10.23.1 Pesticide (608PEST, 8081)

Require 3 shakes with the sample pH in the range of 5-9

Type of water used for QC samples is Distilled water (DI)

Extracted samples are solvent exchanged to Hexane

Subject to Florisil Cleanup

Final volume of extracts is 10.0 mLs

10.23.2 Polychlorinated Biphenyls (608PCB, 8082, 8082LL)

Require 3 shakes with the sample pH in the range of 5-9

Type of water used for QC samples is Distilled water (DI)

Extracted samples are solvent exchanged to Hexane

Subject to Silica Gel Cleanup (method 3630C) procedures

Acid cleanup (method 3665A) is performed on all 8082 samples and associated QC
Acid cleanup (method 3665A) is NOT performed on 608PCB or 8082LL samples

Final volume for 608PCB extracts us 2.0 mLs

Final volume for 8082 extracts is 10.0 mLs

10.24 Diesel Range Organics (310.13,8015B,DRO)

Require 3 shakes with the sample pH <2.

Type of water used for QC samples is Distilled water (DI).

Final volume is 1.0 mL

The oily nature of the spike and surrogate used for this extraction may result in a greater affinity for the Teflon sep funnels rather than the aqueous matrix.

> If low analyte recoveries are observed 40 mLs of $MeCl_2$ can be added to the empty sep funnel. Rotate the sep funnel for approximately thirty seconds and drain through the powder funnel. Concentrate this with the rest of the extract for that sample.

10.25 Semivolatile (8270, 625, 8270LL)

10.25.1 BNA (Base, Neutral Acid)

Most 8270 and 625 require 6 shakes
3 shakes with the sample pH <2
3 shakes with the sample pH in the range of 11-12

> Care should be taken to add only as much acid or base that is necessary to bring the sample within required range. Over acidifying samples results in the loss of Base/Neutral compounds. Over hydrolyzing samples results in the hydrolysis of compounds.

Type of water used for QC samples is Di water with approximately 1 gram of salt

Final volume is 1.0 mL

10.25.2 BN (Base, Neutral), TEL (TetraEthyl Lead), PAH (Polyaromatic Hydrocarbons) STARS

Require 3 shakes with the sample pH in the range of 7-11

Type of water used for QC samples is Di water with approximately 1 gram of salt

Final Volume is 1.0 mL

10.26 Extraction of TCLP Leachates

Can be applied to 8270, 8081, 8151

Sample volume is 250 mLs

QC sample volume is recorded as 250 mLs,

11.0 Calculations / Data Reduction N/A

12.0 Method Performance

Acceptable performance is monitored through the use of Method Detection Limit Studies as well as recoveries of surrogate and spike compounds.

12.1 Method Detection Limit Study (MDL)

The method detection limit (MDL) is the lowest concentration that can be detected for a given analytical method and sample matrix with 99% confidence that the analyte is present. The MDL is determined according to the laboratory's MDL procedure in the QA Manual. MDLs reflect a calculated (statistical) value determined under ideal laboratory conditions in a clean matrix, and may not be achievable in all environmental matrices. The laboratory maintains MDL studies for analyses performed; these are verified at least annually unless method requirements require a greater frequency.

12.2 Demonstration of Capabilities

A one–time initial demonstration of performance for each individual method for both soils and water matrices must be generated.

12.2.1 This requires quadruplicate analysis of a mid–level check standard containing the standard analytes for the method using the same procedures used to analyze samples, including sample preparation.

12.2.2 Calculate the average recovery and standard deviation of the recovery for each analyte of interest.

12.2.3 Compare these results with the acceptance criteria given in the Method or to laboratory historical limits (if available).

12.2.4 Repeat the test for any analyte that does not meet the acceptance criteria. Repeated failure for any analyte indicates the need for the laboratory to evaluate the analytical procedure and take corrective action.

12.3 Training Requirements

12.3.1 The supervisor has the responsibility to ensure that this procedure is performed by an analyst who has been properly trained in its use and has the required experience.

12.3.2 The following analyst validation information is maintained for this method in the laboratory QA files.

12.3.3 The analyst must complete the laboratory safety orientation training that includes, but is not limited to, chemicals, PPE requirements, and electrical safety.

12.3.4 The analyst must read and understand this SOP.

12.3.5 The analyst must read and understand the Method used as reference for this SOP.

12.3.6 The analyst must complete a DOC or successfully analyze PT samples annually.

13.0 Pollution Control

13.1 It is TestAmerica’s policy to evaluate each method and look for opportunities to minimize waste generated (i.e., examine recycling options, ordering chemicals based on quantity needed, preparation of reagents based on anticipated usage and reagent stability).

14.0 Waste Management

14.1 The following waste streams are produced when this method is carried out.

14.1.1 Methylene Chloride waste. (Spent solvents are stored in satellite “C” waste containers. When full, a designated sample custodian will transfer solvent material from these satellite “C” waste containers to a grounded 55-gallon drum. These are located in the secured waste area and are disposed of according to all state and federal regulations).

14.1.2 Hexane waste. (Spent solvents are stored in satellite “C” waste containers. When full, a designated sample custodian will transfer solvent material from these satellite “C” waste containers to a grounded 55-gallon drum. These are located in the secured waste area and are disposed of according to all state and federal regulations).

14.1.3 Assorted flammable solvent waste from various rinses. (Spent solvents are stored in satellite “C” waste containers. When full, a designated laboratory technician will transfer solvent material from these satellite “C” waste containers to a grounded 55-gallon drum. These are located in the secured waste area and are disposed of according to all state and federal regulations).

14.1.4 Vials containing extracts in solvents. (Extract vials are disposed in BV waste drums and stored in the GC and GCMS SVOA departments. These drums are disposed of according to all state and federal regulations).

14.1.5 Extracted water samples. This material must be neutralized before it is discharged. (All extracted water shall be neutralized and dumped into the designated drum marked as “W” waste. When full, the satellite containers will be transferred to the secure waste storage area and disposed of by appropriately trained laboratory technicians in accordance to all state and federal regulations).

14.1.6 Extracted aqueous samples contaminated with methylene chloride. This material must be neutralized before it is discharged to a POTW. (All extracted water shall be neutralized and dumped into the designated drum marked as “W” waste. When full, the satellite containers will be transferred to the secure waste storage area and disposed of by appropriately trained laboratory technicians in accordance to all state and federal regulations).

14.1.7 Used sodium sulfate and glass wool or filter paper contaminated with methylene chloride from the extract drying step. (Solid wastes are dried in trays inside a fume hood then transferred to 5-gallon satellite containers. Lab generated solid wastes (extracted solid waste, sodium sulfate and glass wool or filter paper) are marked as "BC waste. When full, a designated laboratory technician will transfer all of the lab generated solid waste into a 55-gallon drum. This material will be disposed of according to all state and federal regulations.).

14.1.8 Miscellaneous disposable glassware contaminated with acids, caustics, solvents and sample residue. (All disposable glassware is dried of all solvents inside a fume hood then disposed of in a recycling bin).

14.2 Waste management practices are conducted consistent with all applicable rules and regulations. Excess reagents, samples and method process wastes are disposed of in an accepted manner. Waste description rules and land disposal restrictions are followed. Waste disposal procedures are incorporated by reference to [*].

15.0 References / Cross-References

15.1 Method 3510C, "Separatory Funnel Liquid-Liquid Extraction", Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW-846, Third Edition, Final Update III, December 1996.

15.2 CFR, Title 40, Protection of Environment, Pt. 136, App.A, Method 608, Revised as of July 1, 1996.

15.3 CFR, Title 40, Protection of Environment, Pt. 136, App. A, Method 625, Revised as of July1, 1996.

16.0 Method Modifications: N/A

17.0 Attachments

Table 1: Spike and Surrogate Recipes
Table 2: Spike and Surrogate Reference Sheet
Table 3: Specific Test Requirements
Table 4: Spike and Surrogate Reference
Attachment 1: Bench Sheet

18.0 Revision History

Revision 1, dated 12 March 2010

- Updated section 10.6 to include specific directions on procedure for handling water samples with an oil layer.
- Removed: "Record temperature of Turbovap water bath on the bench sheet." From section 10.18 as it is now recorded daily in a logbook.
- Added section 10.6.3 to include use of bottle template for obtaining sample volumes
- Updated Bench Sheet Attachment
- Updated Table 1,2, 3

- Updated section 10.23.2 to include 8082 LL
- Updated section 10.25 to include 8270LL
- Updated section 10.26 by removing 8082 and replacing with 8151
- Updated type of water used for 8270,625, 8270PAH/STARS extractions from VOA free to Di with approximately 1 gram of salt added.

Revision 0, dated 18 February 2008

- Integration for TestAmerica and STL operations.

Table 1 Spike and Surrogate Recipes

Spike Name	Spike Code	Compound Name	Mix Location	Amount Added (uL)	Final Volume	Solvent	Directions for Verification	Final Conc. (ng/uL)
8270 surrogate	A01	B/N surrogate mix	incubator	20,000 uL	1000 mL	MeOH	S.E.→MeCl2 F.V.=1 mL	100
		ACID surrogate mix	incubator	15,000 uL				150
8270 GPC surrogate (OLMO4.3 GPC)	A26	B/N surrogate mix	incubator	10,000 uL	1000 mL	MeOH	S.E.→MeCl2 F.V.=1 mL	50
		ACID surrogate mix	incubator	5,000 uL				50
8015B surr.	A27	O-Terphenyl Std	incubator	1,000 uL	500 mL	ACETONE	S.E.→MeCl2 F.V.=1 mL	20
CLLE surrogate	A28	B/N surrogate mix HC	drawer	1,000 uL	100 mL	MeOH	S.E.→MeCl2 F.V.=1 mL	50
		ACID surrogate mix HC	drawer	750 uL				75
8151 surr.	A33	2,4-Diclorophenol/acetic acid	incubator	2,500 uL	1000 mL	MeOH	DERIVATIZE F.V.=1mL	5
8081/8082 surr	A35	Pest Surr. Solution	drawer	1,000 uL	1000 mL	MeOH	Bring over as is (1 mL)	0.2
8151 spike	A47	Chlorinated Herb.mix	drawer	2,000 uL	100 mL	MeOH	DERIVATIZE F.V.=1mL	2
Pest/PCB surrogate (OLMO4.3 GPC)	A49	Pest Matrix Spike	incubator	1,000 uL	50 mL	MeOH	S.E.→HEX. F.V.=10 mL	0.5-1.0
8270 short list spike	A55	B/N Matrix Spike mix	incubator	10,000 uL	500 mL	MeOH	S.E.→MeCl2 F.V.=1 mL	100
		ACID Matrix Spike mix	incubator	5,000 uL				100
625 spike	A56	76 Big Mix	freezer	5,000 uL	100 mL	MeOH	S.E.→MeCl2 F.V.=1 mL	50
		XQ-5237	freezer	5,000 uL				50
CLLE spike	A57	B/N matrix spike mix	incubator	5,000 uL	500 mL	MeOH	S.E.→MeCl2 F.V.=1 mL	50
		ACID matrix spike mix	incubator	3,750 uL				75
8270 pre-dilutions spike	A60	Ultra Custom mix	drawer	1,000 uL	20 mL	MeOH	S.E.→MeCl2 F.V.=1 mL	100
	A61	Custom mix		Use As Received				50
8270 TCLP spike	A62	B/N TCLP mix	incubator	1,000 uL	20 mL	MeOH	S.E.→MeCl2 F.V.=1 mL	100
		Acid TCLP Mix	incubator	1,000 uL				100
8015B / 310.13 spike	A95	Diesel Fuel #2	drawer	3,000 uL	100 mL	MeOH	S.E.→MeCl2 F.V.=1 mL	1500
608Pest Dech+ spike	A113	Dechlorane Plus	incubator	1,000 uL	100 mL	ACETONE	S.E.→HEX. F.V.=1 mL	1
8270 Full List spike	A193	76 Big Mix	freezer	5,000 uL	50 mL	MeOH	S.E.→MeCl2 F.V.=1 mL	100
		XQ-5237	freezer	5,000 uL				100

8082 spike	A222	AR 1016/1260 MIX	drawer	2,500 uL	500 mL	1/2MeOH 1/2Acetone	Bring over as is (1 mL)	5
8081 spike	A225	Organochlorine Pest Mix AB #1	incubator	500 uL	200 mL	MeOH	Bring over as is (1 mL)	0.5
8270 1-4 dioxane only spike	A234	1,4-Dioxane	freezer	1,250 uL	25 mL	MeOH	S.E.→MeCl2 F.V.=1 mL	100
8082 wipe surrogate	A278	Pest Surr. Solution	drawer	1,000uL	250 mL	MeOH	Bring over as is (1 mL)	0.8
8082 wipe spike	A279	AR 1016/1260 MIX	drawer	2,500 uL	125 mL	1/2MeOH 1/2Acetone	Bring over as is (1 mL)	20
548.1 spike	A288 Endothall	Acenaphthene-d10	drawer	1,000 uL	200 mL	$MeCl_2$	S.E.→MeCl2 F.V.=1 mL	10
8270 TEL only spike	A302	Tetraethyllead	freezer	5,000 uL	50 mL	MeOH	S.E.→MeCl2 F.V.=1 mL	100
8270 Millseat Spike	A309	EPA 8270/APP IX	freezer	1,000 uL	10 mL	MeOH	S.E.→MeCl2 F.V.=1 mL	100
		8270 APP IX Supp. #1	incubator	500 uL				100
		8270 APP IX Supp #2	incubator	500 uL				100
		Aramite	incubator	500 uL				100
		8270 Benzidines Mix	freezer	500 uL				100
		1,4-Phylenediamine (0.1509g into 1L DiH_20)	freezer	1,000 uL				1500
8270 PAH/STARS	PAH/STARS	PAH Mixtrue	Incubator	5,000 uL	100 mL	MeOH	Bring over as is (1 mL)	100
	A314	O-Terphenyl Std	incubator	200 uL	500 mL	Acetone	S.E.→MeCl2 F.V.=1 mL	100
	A315	Use straight Diesel Fuel #2						
608 PCB surr.	A318	Pest. Surrogate Std.		100 uL	500 mL	MeOH	S.E.→HEX. F.V.=10 mL	
608 PCB spike	A319	Aroclor 1016/1260	drawer	1,000 uL	1000 mL	MeOH	S.E.→HEX. F.V.=2 mL	1
GPC Check	PCB √ Sol.	Aroclor 1016/1260	drawer	1,000 uL	500 mL	$MeCl_2$	NONE	2
GPC calibration	GPC Cal. Sol.	GPC Calibration Mix	incubator	5,000 uL	50 mL	$MeCl_2$	NONE	0.02-2.5

Table 2 Spike and Surrogate Reference Sheet

A27	o-Terphenyl
A28	Nitrobenzene-d5
	2-Fluorobiphenyl
	p-terphenyl
	Phenol-d5
	2-Fluorophenol
	2,4,6-Tribromophenol
	1,2-Dichlorobenzene
	2-Chlorophenol-d4
A33	Dichlorophenyl Acetic Acid
A35	Tetrachloro-m-xylene
	Decachlorobiphenyl
A47	2,4-D
	Dalapon
	Dinoseb
	Pentachlorophenol
	Picloram
	2,4,5-TP (Silvex)
	2,4,5-T
	2,4-DB
	Dicamba
	Dichloroprop
A49	gamma-BHC (Lindane)
	Heptachlor
	Aldrin
	Dieldrin
	Endrin
	4,4'-DDT
A55	Phenol
	2-Chlorophenol
	1,4-Dichlorobenzene
	N-Nitroso-Di-n-Propylamine
	1,2,4-Trichlorobenzene
	4-Chloro-3-methylphenol
	Acenapththene
	4-Nitrophenol
	2,4-Dinitrotoluene
	Pentachlorophenol
	Pyrene

A93	Tetrachloro-m-xylene
	Decachlorobiphenyl
A222	Aroclor 1016
	Aroclor 1260
A225	Endosulfan I
	Endosulfan II
	Endrin Aldehyde
	Endosulfan Sulfate
	Heptachlor epoxide
	Methoxychlor
	Endrin Ketone
	alpha-Chlordane
	gamma-Chlordane
	Gamma-BHC-(Lindane)
	alpha-BHC
	Heptachlor
	Aldrin
	Beta-BHC
	Dieldrin
	Endrin
	4,4;-DDD
	4,4'-DDT
	4,4;-DDE
A318	Tetrachloro-m-xylene
	Decachlorobiphenyl
8270 PAH/STARS	Chrysene
	Acenaphthylene
	Anthracend
	Benzo[a]anthracene
	Benzo[b]fluoranthene
	Acenaphthene
	Benzo[k]fluoranthene
	Pyrene
	Dibenz[a,h]anthracene
	Fluoranthene
	Fluorene
	Indeno[1,2,3-cd]pyrene
	Naphthalene
	Phenanthrene
	Benzo[g,h,i]perylene

Table 2 Continued

A193	Acenaphthene	Dimethyl phthalate
	Aniline	4,6-Dinitro-2-methylphenol
	Acenaphthylene	2,4-Dinitrophenol
	Anthracene	2,4-Dinitrotoluene
	Benzo(a)anthracene	2,6-Dinitrotoluene
	Benzo(b)fluoranthene	Di-n-octyl phthalate
	Benzo(k)fluoranthene	Fluoranthene
	Benzo(ghi)perylene	Fluorene
	Benzo(a)pyrene	Hexachlorobenzene
	Benzoic Acid	Hexachlorobutadiene
	Benzyl alcohol	Hexachlorocyclopentadiene
	Bis(2-chloroethoxy) methane	Hexachloroethane
	Bis(2-chloroethyl) ether	Indeno(1,2,3-cd)pyrene
	Pyridine	Isophorone
	Bis(2-ethylhexyl)phthalate	2-Methylnaphthalene
	4-Bromophenyl phenyl ether	2-Methylphenol
	Butyl benzyl phthalate	4-Methylphenol
	4-Chloroaniline	Naphthalene
	4-Chloro-3-methylphenol	2-Nitroaniline
	2-Chloronaphthalene	3-Nitroaniline
	2-Chlorophenol	4-Nitroaniline
	4-Chlorophenyl phenyl ether	Nitrobenzene
	Benzaldehyde	3-Methylphenol
	Acetophenone	1-Methylnaphthalene
	Caprolactam	Diphenylamine
	1,2,4,5-Tetrachlorobenzene	1,4-Dinitobenzene
	Biphenyl	1,3-Dinitobenzene
	Atrazine	1,2-Dinitrobenzene
	Quinoline	Azobenzene
	Benzidine	bis(2-Chloroisopropyl)ether
	2,3,5,6-Tetrachlorophenol	bis-(2-Ethylhexyl)adipate
	2,3,4,6-Tetrachlorophenol	Carbazole
	N-Nitrosodiphenylamine	2-Nitrophenol
	Chrysene	4-Nitrophenol
	Dibenzo(a,h)anthracene	N-Nitrosodiphenylamine
	Dibenzofuran	N-Nitroso-Di-n-prpoylamine
	Di-n-butyl phthalate	Pentachlorophenol
	1,2-Dichlorobenzene	Phenanthrene
	1,3-Dichlorobenzene	Phenol
	1,4-Dichlorobenzene	Pyrene
	3,3-Dichlorobenzidine	1,2,4-Trichlorobenzene
	2,4;-Dichlorophenol	2,4,5-Trichlorophenol
	Diethyl phthalate	2,4,6-Trichlorophenol
	2,4-Dimethylphenol	3 & 4 Methylphenol
	1,4-Dioxane	Tetraethyl lead

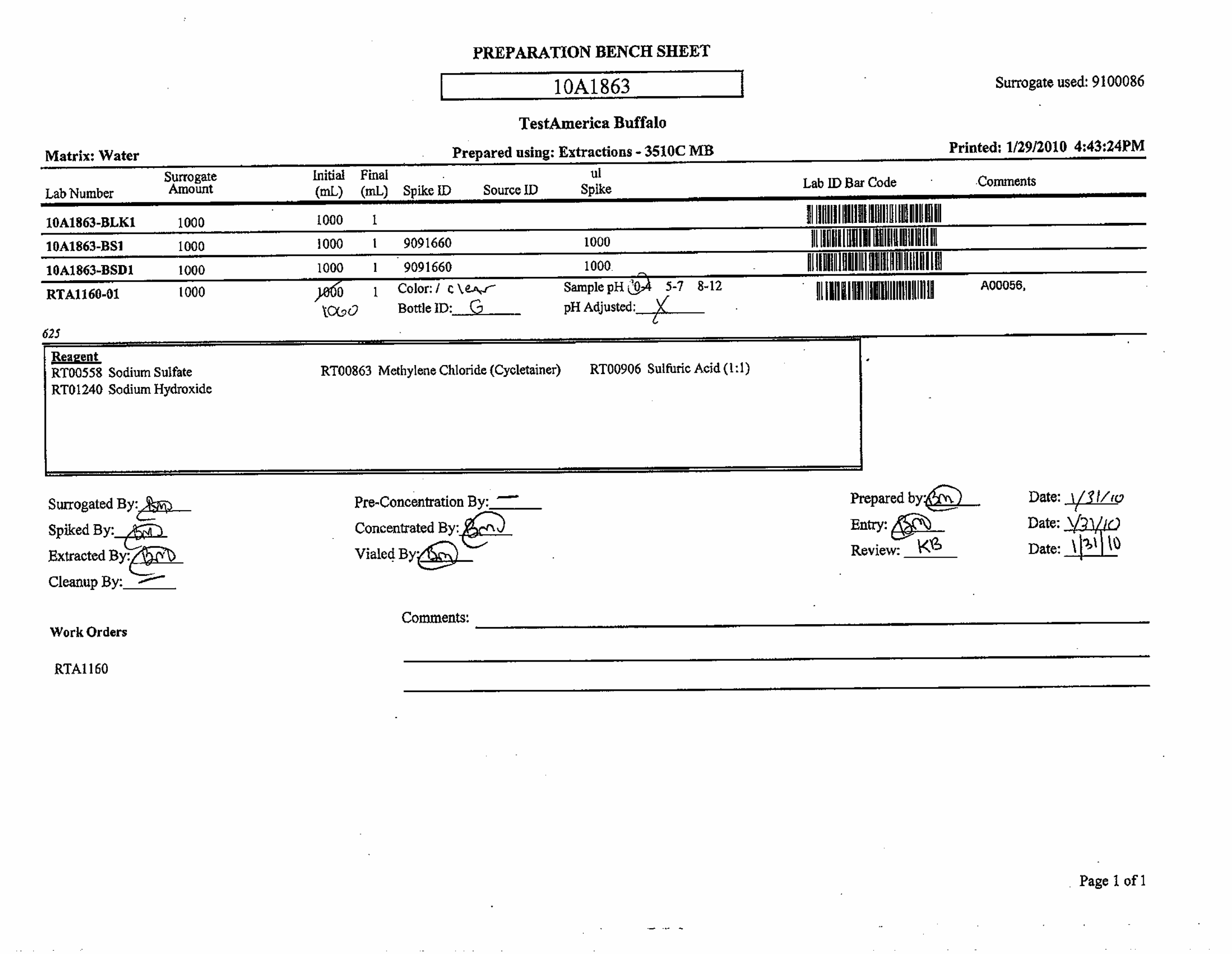
Table 3 Test Requirements Reference

Method	# Shakes	Initial pH	Secondary pH	QC Water Type	Hex. Exchange	Final Volume	Cleanup
608Pest	3	5-9	N/A	DI	Yes	10 mL	Florisil
8081	3	5-9	N/A	DI	Yes	10 mL	Florisil
608PCB	3	5-9	N/A	DI	Yes	2.0 mL	Silica Gel
8082	3	5-9	N/A	DI	Yes	10 mL	Silica Gel/Acid
310.13/8015B	3	<2	N/A	DI	No	1 mL	N/A
625	6	<2	11-12	DI + 1g salt	No	1 mL	N/A
8270	6	<2	11-12	DI + 1g salt	No	1 mL	N/A
8270 TCLP	6	<2	11-12	DI + 1g salt	No	1 mL	N/A
8270 PAH/STARS	3	7-11	N/A	DI + 1g salt	No	1 mL	N/A

Table 4 Spike and Surrogate Reference

	METHOD	SURROGATE	AMOUNT USED	SPIKE	AMOUNT USED	FINAL VOLUME
PEST	608***	A35	1000 uL	A225	1000 uL	10 mL
	608 dech+	A35	1000 uL	A113	1000 uL	10 mL
	8081***	A35	1000 uL	A225	1000 uL	10 mL
	CLP	A093	2000 uL A35	A049	1000 uL	GPC & 5 Ml
PCB	608	A318	1000 uL	A319	1000 uL	2 Ml
	8082	A35	1000 uL	A222	1000 uL	10 mL
HERB	8151	A33	1000 uL	A47	1000 uL	10 mL
DRO/310.13	8015B***	A27	1000 uL	A95	1000 uL	1 mL
	310.13/310.14***	A27	1000 uL	A95	1000 uL	1 mL
	med level*	A314	1000 uL	A315	150 uL (diesel fuel #2)	5 mL
	METHOD	**SURROGATE**	**AMOUNT USED**	**SPIKE**	**AMOUNT USED**	**FINAL VOLUME**
8270	BN/AP ***	A01	1000 uL	A055	1000 uL	1 mL
	FULL LIST ***	A01	1000 uL	**A193**	1000 uL	1 mL
	LOW	A148	100 uL of A26	A147	100 uL of A055	1 mL
	LOW	A148	100 uL of A26	A316	100 uL A193	1 mL
	EXPANDED LIST	A01	1000 uL	A309**	800 uL A309 800 uL of A193	1 mL
	BASE ONLY	A01	1000 uL	A234	1000 uL	1 mL
	TCLP	A01	1000 uL	A62	1000 uL	1 mL
	PAH only	A01	1000 uL	A327	500 uL of A193	1 mL
EPA/CLP	CLLE	A28	1000 uL	A57	1000 uL	1 mL
625	PP BNA	A26	1000 uL	A56	1000 uL A193	1 mL

Attachment 1: Bench Sheet



PREPARATION BENCH SHEET

10A1863

Surrogate used: 9100086

TestAmerica Buffalo

Matrix: Water

Prepared using: Extractions - 3510C MB

Printed: 1/29/2010 4:43:24PM

Lab Number	Surrogate Amount	Initial (mL)	Final (mL)	Spike ID	Source ID	ul Spike	Lab ID Bar Code	Comments
10A1863-BLK1	1000	1000	1					
10A1863-BS1	1000	1000	1	9091660		1000		
10A1863-BSD1	1000	1000	1	9091660		1000		
RTA1160-01	1000	~~1000~~ 1000	1	Color: / clear Bottle ID: G		Sample pH 0-4 5-7 8-12 pH Adjusted: X		A00056,

625

Reagent
RT00558 Sodium Sulfate
RT01240 Sodium Hydroxide
RT00863 Methylene Chloride (Cycletainer)
RT00906 Sulfuric Acid (1:1)

Surrogated By: BM
Spiked By: BM
Extracted By: BM
Cleanup By: —

Pre-Concentration By: —
Concentrated By: BM
Vialed By: BM

Prepared by: BM Date: 1/31/10
Entry: BM Date: 1/31/10
Review: KB Date: 1/31/10

Comments:

Work Orders

RTA1160

Page 1 of 1

Ultrasonic Extraction of Soils and Wipes (Method No 3550B)

Once printed, this is considered an uncontrolled document

Approvals (Signature/Date):

Shanna Snider 03/12/10
Shanna Snider Date
Department Manager

Kenneth E. Kasperek 03/12/10
Kenneth Kasperek Date
Technical Director

Peggy Gray-Erdmann 03/12/10
Peggy Gray-Erdmann Date
Quality Assurance Manager

03/12/10
Christopher Spencer Date
Laboratory Director

Facility Distribution No. ___________ **Distributed To:**________________________

1.0 Scope and Application

1.1 This method is used for the extraction of nonvolatile and semivolatile organic compounds from solids and wipes. The ultrasonic process used ensures thorough contact of the sample with the extraction solvent.

2.0 Analytes, Matrix(s), and Reporting Limits

2.1 This method is used for the extraction of nonvolatile and semivolatile organic compounds from solids and wipes.

2.2 Reporting Limit N/A

On occasion clients may request modifications to this SOP. These modifications are handled following the procedures outlined in the Quality Assurance Manual.

3.0 Summary of Method

3.1 Low Level

A 30 gram sample is mixed with anhydrous sodium sulfate. This is solvent extracted three times using ultrasonic extraction. The extract is then filtered and concentrated. The extract may then be subject to clean-up procedures or sent directly for analysis.

3.2 Medium/High Level

A 2 gram sample is mixed with anhydrous sodium sulfate and solvent extracted once using ultrasonic extraction. A portion of the extract is removed for cleanup and/or analysis.

3.3 Wipes

A wipe sample is mixed with anhydrous sodium sulfate and solvent extracted once using ultrasonic extraction.
A portion of the extract is removed for cleanup and/or analysis.

4.0 Definitions

Standard definitions are found in Section 3.2 of the Laboratory Quality Manual

5.0 Interferences

5.1 Method interference may be caused by contaminants in solvents, reagents, glassware and other sample processing hardware that lead to discrete artifacts or elevated baselines in gas chromatograms. All these materials must be routinely demonstrated to be free from interference under the conditions of the analysis, by analyzing reagent blanks. Matrix interference may be caused by contaminants that are co-extracted from the sample.

5.2 Major organic interferences may be removed during cleanup procedures

6.0 Safety

6.1 Employees must abide by the policies and procedures in the Corporate Safety Manual, Radiation Safety Manual and this document.

6.2 This procedure may involve hazardous material, operations and equipment. This SOP does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of the method to follow appropriate safety, waste disposal and health practices under the assumption that all samples and reagents are potentially hazardous. Safety glasses, gloves, lab coats and closed-toe, nonabsorbent shoes are a minimum.

6.3 Specific Safety Concerns or Requirements

6.3.1 All parameters of this extraction where solvent is being used must be performed in an operational fume hood or within an extraction apparatus that is ventilated by the fume hood system.

6.3.2 Any excess unextracted sample (including dry weights) waste will be disposed of in “BE” waste. Solid waste generated in the extraction process will be disposed of in “BC” waste. All solvent and extract waste is disposed of in “C” waste.

6.3.3 Safety glasses, gloves, and lab coats must be worn at all times. Nitrile gloves should be used when performing this extraction. Latex and vinyl gloves provide no significant protection against the organic solvents used for extractions and should not be used.

6.3.4 All solvents, reagents, and standards must be handled inside a fume hood and with proper personal safety equipment due to their hazardous properties. All samples must be opened inside a fume hood due to their unknown hazardous properties.

6.3.5 Due to the high frequency produced from the sonicators, it is necessary to utilize both hood sashes to keep the noise level to a minimum.

6.4 Primary Materials Used

6.4.1 The following is a list of the materials used in this method, which have a serious or significant hazard rating. **Note: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the MSDS for each of the materials listed in the table.** A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the MSDS for each material before using it for the first time or when there are major changes to the MSDS.

Material	Hazards	Exposure Limit	Signs and symptoms of exposure
Methylene Chloride	Carcinogen Irritant	25 ppm-TWA 125 ppm-STEL	Causes irritation to respiratory tract. Has a strong narcotic effect with symptoms of mental confusion, light-headedness, fatigue, nausea, vomiting and headache. Causes irritation, redness and pain to the skin and eyes. Prolonged contact can cause burns. Liquid degreases the skin. May be absorbed through skin.
Acetone	Flammable	1000 ppm-TWA	Inhalation of vapors irritates the respiratory tract. May cause coughing, dizziness, dullness, and headache.
Hexane	Flammable Irritant	500 ppm-TWA	Inhalation of vapors irritates the respiratory tract. Overexposure may cause lightheadedness, nausea, headache, and blurred vision. Vapors may cause irritation to the skin and eyes.
		1 – Always add acid to water to prevent violent reactions.	
		2 – Exposure limit refers to the OSHA regulatory exposure limit.	

7.0 Equipment and Supplies

7.1 Aluminum Dishes, Foil
7.2 Disposable, wood tongue depressor
7.3 Toploader Balance, capable of accurately measuring to 2.0g
7.4 Syringes
7.5 ¾ in. dual horn Sonicators® with Sonabox® acoustic enclosures
7.6 16 oz. french squares, disposable
7.7 Ovens - 104°C and 400°C
7.8 16 oz. wide mouth jars, disposable
7.9 Turbovap concentrators and vessels
7.10 Stainless steel filter funnels
7.11 Graduated cylinders
7.12 Ear Protection
7.13 2,10 and 25 or 40 ml vials, septa and caps
7.14 Disposable pipets and pipet bulbs
7.15 18.5 cm #41 filter paper
7.16 Microtip horn Sonicators with Sonabox acoustic enclosures.

8.0 Reagents and Standards

8.1 All solvents are pesticide grade or equivalent.
8.2 Hexane delivered in cycletainers
8.3 Compressed Nitrogen

8.4 Anhydrous granular sodium sulfate, previously baked in a 400°C oven for a minimum of 4 hours, cooled and dried in a dessicator,and rinsed with methylene chloride. Or purchased pre-baked from Jost chemical.
8.5 Methylene Chloride delivered in cycletainers
8.6 Acetone delivered in cycletainers
8.7 Surrogate and spike solutions appropriate to the final determinative procedures as assigned by test profile (See Table 2).
8.8 De-ionized water (DI)

9.0 Sample Collection, Preservation, Shipment and Storage

9.1 Sample container, preservation techniques and holding times may vary and are dependent on sample matrix, method of choice, regulatory compliance, and/or specific contract or client requests. Listed below are the holding times and the references that include preservation requirements.

Matrix	Sample Container	Min. Sample Size	Preservation	Holding Time [1]	Reference
Soils	Glass	30 grams	Cool 4 ± 2°C	14 Days from sample	SW-846, third edition
Soils	Glass	30 grams	Cool 4 ± 2°C	10 days from receipt	CLP

10.0 Quality Control

10.1 The following quality control samples are prepared with each batch of samples.

Quality Controls	Frequency	Control Limit
Method Blank (BLK)	1in 20 or fewer samples	< Rpt. Limit
Laboratory Control Standard (LCS)[1]	1in 20 or fewer samples	Statistical Limits [4]
Matrix Spike (MS)[2]	1in 20 or fewer samples	Statistical Limits [4]
Spike Duplicate (SD)[2]	1in 20 or fewer samples	Statistical Limits [4]
Surrogates	every sample[3]	Statistical Limits [4]

10.1.1 Laboratory Control Standard Duplicate (LCSD) is performed only when insufficient sample is available for the MS/SD or when requested by the client/project/contract.
10.1.2 The sample selection for MS/SD is randomly selected, unless specifically requested by a client….predetermined by the extraction lab.
10.1.3 Analytical and QC samples (BLK, LCS,LCSD, MS/SD)
10.1.4 Statistical control limits are updated annually and are updated into LIMS.

11.0 Procedure

Sample volumes and Bottle IDs are directly recorded into Element, and therefore may not be present on the scanned copy of the bench sheets. Also, the Color;/, Sample pH, and pH adjusted fields pertain to water extractions only.

11.1 Low Level Extraction:

11.1.1 Decant and discard any standing water on the sample. Enter into LIMS if sample was decanted. Discard any sticks, leaves, rocks or other foreign matter.

11.1.2 Tare a labeled 16oz wide mouth jar and transfer 30 grams of homogenized soil. For blank samples (LCS,LCSD, or BLK), approximately 30g of sodium sulfate will be used in lieu of soil and shall be taken through the entire analytical procedure

11.1.3 Add granular sodium sulfate to the 30g sample and blend with a wood tongue depressor until the sample is free flowing.

11.1.4 Add surrogate (See Table1) to the samples using the appropriate surrogate as designated on the batch sheet. Write an “X” on the label after adding the surrogate.

11.1.5 Add appropriate spike (See Table1) to samples designated MS, MSD, LCS, LCSD. (The spike code to be used appears on the preparation bench sheets.) Circle the “X” after adding the appropriate spike.

11.1.6 Immediately add 100mls of appropriate solvent to the sample; the solvent for determinative methods is as follows:

All CLP methods –
1:1 methylene chloride/acetone: Combine 500 ml of methylene chloride and 500 ml of acetone in a clean 1 liter glass amber bottle.

8081,8082 - 1:1 acetone/hexane: Combine 500 ml of acetone and 500 ml of hexane in a clean 1liter glass amber bottle.

8270 – Methylene chloride

DRO - Methylene chloride

*8270 soils for specific clients (NiSource) will be extracted with 1:1 Methylene chloride/Acetone

11.1.7 Fold an 18.5cm filter paper into quarters and place it in a stainless steel filter funnel. Place this funnel in a labeled french square bottle.

11.1.8 Before use, clean the sonication horns with 12.5% Nitric acid rinse, DI water, acetone, and the solvent the extraction calls for. Wipe the horns thoroughly with paper towels after the DI water rinse.

11.1.9 Place the 16oz. wide mouth jar under the sonication horn so it is submerged ½ inch. Ideally, the sonicator horn is to be submerged into the solvent ½ inch and still above the soil sample by the ½ inch. In the case of excessively wet samples that needed a great deal of sodium sulfate, more solvent may be added and the position of the sonicator jar adjusted to the ideal parameters.

11.1.10 Sonicate for 3 minutes at out put setting 10, pulsed mode, 50% duty cycle, using ¾ inch horn.

11.1.11 Collect the extract in a labeled french square jar by first decanting the extract through the filter funnel containing the 18.5 cm filter paper folded inside. Rinse the filter paper with the appropriate extraction solvent after the first sonication round is decanted. When using solvents with acetone, add a little sodium sulfate to the filter paper to reduce the amount of water in the extract.

11.1.12 Repeat steps 13.1.9-13.1.11 twice more.

11.1.13 After the third sonication, rinse the contents of the sonication jar into the funnel.

11.1.14 After sample has drained, rinse down the funnel with 20-30mLs of the extraction solvent being used. Allow the sample to drain completely inside a fume hood.

11.1.15 Clean the sonicator horn between samples as describe in section 13.1.8.

11.2 Concentration Procedure

11.2.1 Pour the extract into a labeled turbovap vessel that has been pre-rinsed with $MeCl_2$, rinse the french square with the appropriate solvent and add this to the turbovap vessel.

11.2.2 Place the vessel in the turbovap and turn on the nitrogen to concentrate the extract to approximately 1ml. During concentration, the turbovap vessel should be periodically rinsed with the extraction solvent. The temperature of the turbovap water bath must be maintained between 30˚C and 40˚C.

11.2.3 For 8270 and DROs, concentrate to a final volume of 1ml using the calibrated 1.0ml mark on the turbovap vessel. Transfer entire volume to a 2ml vial using a disposable 9-inch pipette and mark the meniscus. 8270 samples can be relinquished to GC/MS for analysis and DRO samples can be relinquished to GC for analysis.

11.2.4 For 8081/8082 concentrate the extract to 1.0ml using the calibrated 1.0ml mark on the turbovap vessel. Adjust the final volume to 10.0ml by adding 9.0ml of Hexane to the turbovap vessel with a repipettor. If cleanup of samples is required, refer to specific cleanup SOP. If no cleanup is required, transfer 1 ml using a disposable pipette to a 2ml vial. Mark the meniscus on the vial and relinquish to GC for analysis. Transfer remaining extract volume to an appropriately labeled 40ml vial and store for no less than 30 days. All 8082 extracts go through sulfuric acid cleanup. See Sulfuric Acid Cleanup SOP for details.

11.2.5 For all CLP method soils, GPC cleanup is required. Bring the volume to 1.0ml using the calibrated 1.0ml mark on the turbovap vessel then adjust the final volume to 10.0ml by adding 9.0ml of Methylene chloride to the turbovap vessel with a repipetter. Transfer to a 40mL vial. Cap and set aside in a $4^oC \pm 2^o$ C incubator for later clean up by GPC. See GPC SOP for GPC procedure.

11.3 MEDIUM LEVEL EXTRACTION:

No dry weight is required for caulk samples, and samples containing asbestos. The dry weight will be recorded as 100% dry in LIMS.

The sample volume used for caulk samples is 0.1-0.2 grams. This reduced volume aids in minimizing contamination commonly seen from caulk samples.

11.3.1 Decant and discard any standing water on the sample. Enter into LIMS if sample was decanted. Discard any sticks, leaves, rocks or other foreign matter.

11.3.2 Transfer 2 grams of the homogenized sample into a tared 25 mL extraction vial. For blank samples (LCS, LCSD, or BLK), approximately 2 g of sodium sulfate will be used in lieu of soil and shall be taken through the entire analytical procedure.

11.3.3 Add granular sodium sulfate to the 2g sample and blend with a disposable tongue depressor until the sample is free flowing.

11.3.4 Add surrogate to the samples using the appropriate surrogate (See Table 1) as designated on the batch sheet. Write an “X” on the label after adding the surrogate.

11.3.5 Add appropriate spike (See Table 1) to samples designated MS, MSD, LCS, LCSD. (The spike code to be used appears on the preparation bench sheets.) Circle the “X” after adding the appropriate spike.

11.3.6 Add 10.0 mLs of hexane to the sample.

11.3.7 Before use, clean the sonication horns with 12.5% Nitric acid rinse, DI water, acetone, and hexane. Wipe the horns thoroughly with paper towels after the DI water rinse.

11.3.8 Sonicate each sample once for 45 seconds on pulse mode and setting of 5 using a microtip sonicating horn.

11.3.9 Decant the sample into a 40mL vial that is pre labeled with the appropriate vial number. Aliquot 1.0ml using a disposable pipette in a 2mL vial, mark the meniscus and relinquish to GC for analysis. The remaining extract is to be saved for no less than 30 days.
8082 extraction requires acid cleanup. Add 10 mLs of concentrated H_2SO_4, cap and shake for 1 minute. Allow the solvent layer (top layer) to separate from the acid layer (bottom layer) for at least 10 minutes. Aliquot 1.0ml from the top layer using a disposable pipette in a 2mL vial, mark the meniscus, and relinquish to GC for analysis. The remaining extract is to be saved for no less than 30 days.

11.3.10 Clean the sonicator horn between samples as describe in section 13.3.7.

11.4 Wipe Extraction

11.4.1 Place entire sample into a labeled 8oz. wide-mouth jar.

11.4.2 Add anhydrous granular sodium sulfate.

11.4.3 Add 1ml of specific wipe surrogate.

11.4.4 Add appropriate spike to samples designated MS, MSD, LCS, LCSD. Add 4 times the normal amount of spike, unless using a spike made up at 4 times the normal concentration.

11.4.5 For blank samples (LCS, LCSD, and BLK), approximately 30g of sodium sulfate will be used in lieu of soil and shall be taken through the entire analytical procedure.

11.4.6 Add hexane so that the total final volume is 40 mls taking into account the volume of spike and surrogate added. For example, if you add 1 ml of surrogate and 1 ml of spike, the amount of hexane added is 38 mls.

11.4.7 Clean the sonicator horns before beginning extraction, and between samples with 12.5% Nitric Acid rinse, DI water, acetone, hexane. Wipe the horns thoroughly with paper towels after the DI water rinse.

11.4.8 Place the 8oz. wide mouth jar under the sonicator horn so it is submerged ½ inch. Ideally, the sonicator horn is to be submerged into the solvent ½ inch and still above the soil sample by the ½ inch.

11.4.9 Sonicate for 1.5 minutes at out put setting 8 or 9, pulsed mode, 50% duty cycle, using ¾ inch horn.

11.4.10 Transfer approximately 10 mls of solvent to an appropriately labeled 40 ml vial. If 8082 analysis is required perform acid cleanup. Transfer 1.0 ml using a disposable pipette to 2ml vial, mark the meniscus, and relinquish to the appropriate analytical lab. The remaining extract is to be saved for no less than 30 days. Record the final volume as 40.0mls on the batch paperwork.

11.5 Calibration

11.5.1 Analytical Balances are checked on a daily basis, and calibrated by a NIST certified company.

11.5.2 Sonicator horns are checked on a daily basis and tuned at least every 6 months.

12.0 Calculations / Data Reduction N/A

13.0 Method Performance

13.1 Acceptable performance is monitored through the use of Method Detection Limit Studies, as well as, recoveries of surrogate and spike compounds.

13.2 Method Detection Limit Study (MDL)

13.2.1 The method detection limit (MDL) is the lowest concentration that can be detected for a given analytical method and sample matrix with 99% confidence that the analyte is present. The MDL is determined according to the laboratory's MDL procedure in the QA Manual. MDLs reflect a calculated (statistical) value determined under ideal laboratory conditions in a clean matrix, and may not be achievable in all environmental matrices. The laboratory maintains MDL studies for analyses performed; these are verified at least annually unless method requirements require a greater frequency.

13.3 Demonstration of Capabilities

Refer to Buffalo Quality Laboratory Manual

13.4 Training Requirements

Refer to Buffalo Quality Laboratory Manual.

14.0 Pollution Control

14.1 It is TestAmerica's policy to evaluate each method and look for opportunities to minimize waste generated (i.e., examine recycling options, ordering chemicals based on quantity needed, preparation of reagents based on anticipated usage and reagent stability).

15.0 Waste Management

15.1 The following waste streams are produced when this method is carries out.

15.1.1 Waste hexane. Extra volume is stored for a period of no less than 30 days. After 30 days the waste is disposed of according to all state and federal regulations.

15.1.2 Waste Methylene Chloride, Acetone, and/or Hexane. Spent solvents are stored in satellite "C" waste containers. When full satellite containers will be transferred to a grounded 55-gallon drum. These are located in the secured waste area and are disposed of according to all state and federal regulations.

15.1.3 Waste solid material from the extraction process. Solid Wastes are separated into 5-gallon satellite containers. Lab generated solid wastes (extracted solid waste) are marked as "BC waste" and extra solid sample volumes (dry weights and other unextracted solid waste) are marked as "BE waste". When full the satellite containers will be transferred into a 55-gallon drum and disposed of according to all state and federal regulations.

15.1.4 Used sodium sulfate, glass wool, or filter paper contaminated with methylene chloride/acetone or acetone/hexane from the extract drying step. Lab generated solid wastes (extracted solid waste) are marked as "BC waste". When full the satellite containers will be transferred into a 55-gallon drum and disposed of according to all state and federal regulations.

15.1.5 Assorted flammable solvent waste from various glassware rinses. Spent solvents are stored in satellite "C" waste containers. When full satellite containers will be transferred to a grounded 55-gallon drum. These are located in the secured waste area and are disposed of according to all state and federal regulations.

15.1.6 Miscellaneous disposable glassware contaminated with solvents and sample residue. All disposable glassware contaminated with solvent is air dried inside an operational fume hood then disposed in the recycling receptacle.

15.1.7 All waste will be disposed of in accordance with Federal, State, and Local regulations. Where reasonably feasible, technological changes have been implemented to minimize the potential for pollution of the environment. Employees will abide by this method and the policies in section 13 of the Corporate Safety Manual for "Waste Management and Pollution Prevention."

16.0 References / Cross-References

16.1 USEPA Test Methods for Evaluating Solid Waste, Physical/Chemical Methods; SW-846, Third Edition; Revision 2, December 1996; Method 3550B.

16.2 USEPA Contract Laboratory Program, Statement of Work for Organics Analysis, Multi-Media, Multi-Concentration, OLMO4.3.

17.0 Method Modifications:

Item	Method xx	Modification
1	3550B 7.2	Dry weights for samples are kept in a 104 C for 3 hours..
2	3550B 7.3.4	Samples are allowed to gravity drain through filter paper to minimize analyte recovery loss.
3	3550B 7.4.1	Sample volume used for medium level Caulk extraction is reduced from 2 grams to 0.1-0.2 grams to minimize contamination commonly seen from this matrix.

18.0 Attachments

18.1 Table 1: Spike and Surrogate Recipe

18.2 Table 2: Spike and Surrogate Reference Sheet

18.3 Table 3: Organic Prep Worksheet

19.0Revision History

- Revision 0, dated 24, January 2008
    - Integration for TestAmerica and STL operations.
    - Updated section 13.1.8 to include recipe for making 1:1 1:1 methylene chloride/acetone and 1:1 acetone:hexane
    - Updated Table 1,2, and 3

- Revision 1, dated 12, March 2010
    - Updated nomenclature of QC samples
    - Included process for Sulfuric acid cleanup for all 8082 extractions for Section 11.2.4 (removed interim form)
    - Removed dry-weight references in Section 11.0.
    - Removed sections 11.2.6, 11.2.7 concerning GPC cleanup
    - Updated vial sizes
    - Updated Table 1
    - Updated spike/surrogate amount for wipe procedure in Section 11.0.
    - Added Nitric Rinse for cleaning horns between extractions in Section 11.0.
    - Updated power mode and duration settings for sonicators
    - Changed sample volume used for Caulk samples from 2 grams to 0.1-0.2 grams.
    - Updated Method Modifications table to include reduced volume for Caulk samples.
    - Included comment in section 11.0 clarifying bench sheets used for soil extractions.

Table 1 Spike and Surrogate Recipes

Spike Name	Spike Code	Compound Name	Mix Location	Amount Added (uL)	Final Volume	Solvent	Directions for Verification	Final Conc. (ng/uL)
8270 surrogate	A01	B/N surrogate mix	incubator	20,000 uL	1000 mL	MeOH	S.E.→MeCl2 F.V.=1 mL	100
		ACID surrogate mix	incubator	15,000 uL				150
8270 GPC surrogate (OLMO4.3 GPC)	A26	B/N surrogate mix	incubator	10,000 uL	1000 mL	MeOH	S.E.→MeCl2 F.V.=1 mL	50
		ACID surrogate mix	incubator	5,000 uL				50
8015B surr.	A27	O-Terphenyl Std	incubator	1,000 uL	500 mL	ACETONE	S.E.→MeCl2 F.V.=1 mL	20
CLLE surrogate	A28	B/N surrogate mix HC	drawer	1,000 uL	100 mL	MeOH	S.E.→MeCl2 F.V.=1 mL	50
		ACID surrogate mix HC	drawer	750 uL				75
8151 surr.	A33	2,4-Diclorophenol/acetic acid	incubator	2,500 uL	1000 mL	MeOH	DERIVATIZE F.V.=1mL	5
8081/8082 surr	A35	Pest Surr. Solution	drawer	1,000 uL	1000 mL	MeOH	Bring over as is (1 mL)	0.2
8151 spike	A47	Chlorinated Herb.mix	drawer	2,000 uL	100 mL	MeOH	DERIVATIZE F.V.=1mL	2
Pest/PCB surrogate (OLMO4.3 GPC)	A49	Pest Matrix Spike	incubator	1,000 uL	50 mL	MeOH	S.E.→HEX. F.V.=10 mL	0.5-1.0
8270 short list spike	A55	B/N Matrix Spike mix	incubator	10,000 uL	500 mL	MeOH	S.E.→MeCl2 F.V.=1 mL	100
		ACID Matrix Spike mix	incubator	5,000 uL				100
625 spike	A56	76 Big Mix	freezer	5,000 uL	100 mL	MeOH	S.E.→MeCl2 F.V.=1 mL	50
		XQ-3649	freezer	5,000 uL				50
CLLE spike	A57	B/N matrix spike mix	incubator	5,000 uL	500 mL	MeOH	S.E.→MeCl2 F.V.=1 mL	50
		ACID matrix spike mix	incubator	3,750 uL				75
8270 pre-dilutions spike	A60	Ultra Custom mix	drawer	1,000 uL	20 mL	MeOH	S.E.→MeCl2 F.V.=1 mL	100
	A61	Custom mix		Use As Received				50
8270 TCLP spike	A62	B/N TCLP mix	incubator	1,000 uL	20 mL	MeOH	S.E.→MeCl2 F.V.=1 mL	100
		Acid TCLP Mix	incubator	1,000 uL				100
8015B / 310.13 spike	A95	Diesel Fuel #2	drawer	3,000 uL	100 mL	MeOH	S.E.→MeCl2 F.V.=1 mL	1500
608Pest Dech+ spike	A113	Dechlorane Plus	incubator	1,000 uL	100 mL	ACETONE	S.E.→HEX. F.V.=1 mL	1
8270 Full List spike	A193	76 Big Mix	freezer	5,000 uL	50 mL	MeOH	S.E.→MeCl2 F.V.=1 mL	100
		XQ-3649	freezer	5,000 uL				100

8082 spike	A222	AR 1016/1260 MIX	drawer	2,500 uL	500 mL	1/2MeOH 1/2Acetone	Bring over as is (1 mL)	5
8081 spike	A225	Organochlorine Pest Mix AB #1	incubator	500 uL	200 mL	MeOH	Bring over as is (1 mL)	0.5
8270 1-4 dioxane only spike	A234	1,4-Dioxane	freezer	1,250 uL	25 mL	MeOH	S.E.→MeCl2 F.V.=1 mL	100
8082 wipe surrogate	A278	Pest Surr. Solution	drawer	1,000uL	250 mL	MeOH	Bring over as is (1 mL)	0.8
8082 wipe spike	A279	AR 1016/1260 MIX	drawer	2,500 uL	125 mL	1/2MeOH 1/2Acetone	Bring over as is (1 mL)	20
548.1 spike	A288 Endothall	Acenaphthene-d10	drawer	1,000 uL	200 mL	$MeCl_2$	S.E.→MeCl2 F.V.=1 mL	10
8270 TEL only spike	A302	Tetraethyllead	freezer	5,000 uL	50 mL	MeOH	S.E.→MeCl2 F.V.=1 mL	100
8270 Millseat Spike	A309	EPA 8270/APP IX	freezer	1,000 uL	10 mL	MeOH	S.E.→MeCl2 F.V.=1 mL	100
		8270 APP IX Supp. #1	incubator	500 uL				100
		8270 APP IX Supp #2	incubator	500 uL				100
		Aramite	incubator	500 uL				100
		8270 Benzidines Mix	freezer	500 uL				100
		1,4-Phylenediamine (0.1509g into 1L DiH$_2$0)	freezer	1,000 uL				1500
	A314	O-Terphenyl Std	incubator	200 uL	500 mL	Acetone	S.E.→MeCl2 F.V.=1 mL	100
	A315	Use straight Diesel Fuel #2						
608 PCB surr.	A318	Pest. Surrogate Std.		100 uL	500 mL	MeOH	S.E.→HEX. F.V.=10 mL	
608 PCB spike	A319	Aroclor 1016/1260	drawer	1,000 uL	1000 mL	MeOH	S.E.→HEX. F.V.=2 mL	1
GPC Check	PCB √ Sol.	Aroclor 1016/1260	drawer	1,000 uL	500 mL	$MeCl_2$	NONE	2
GPC calibration	GPC Cal. Sol.	GPC Calibration Mix	incubator	5,000 uL	50 mL	$MeCl_2$	NONE	0.02-2.5

Table 2 Spike and Surrogate Reference Sheet

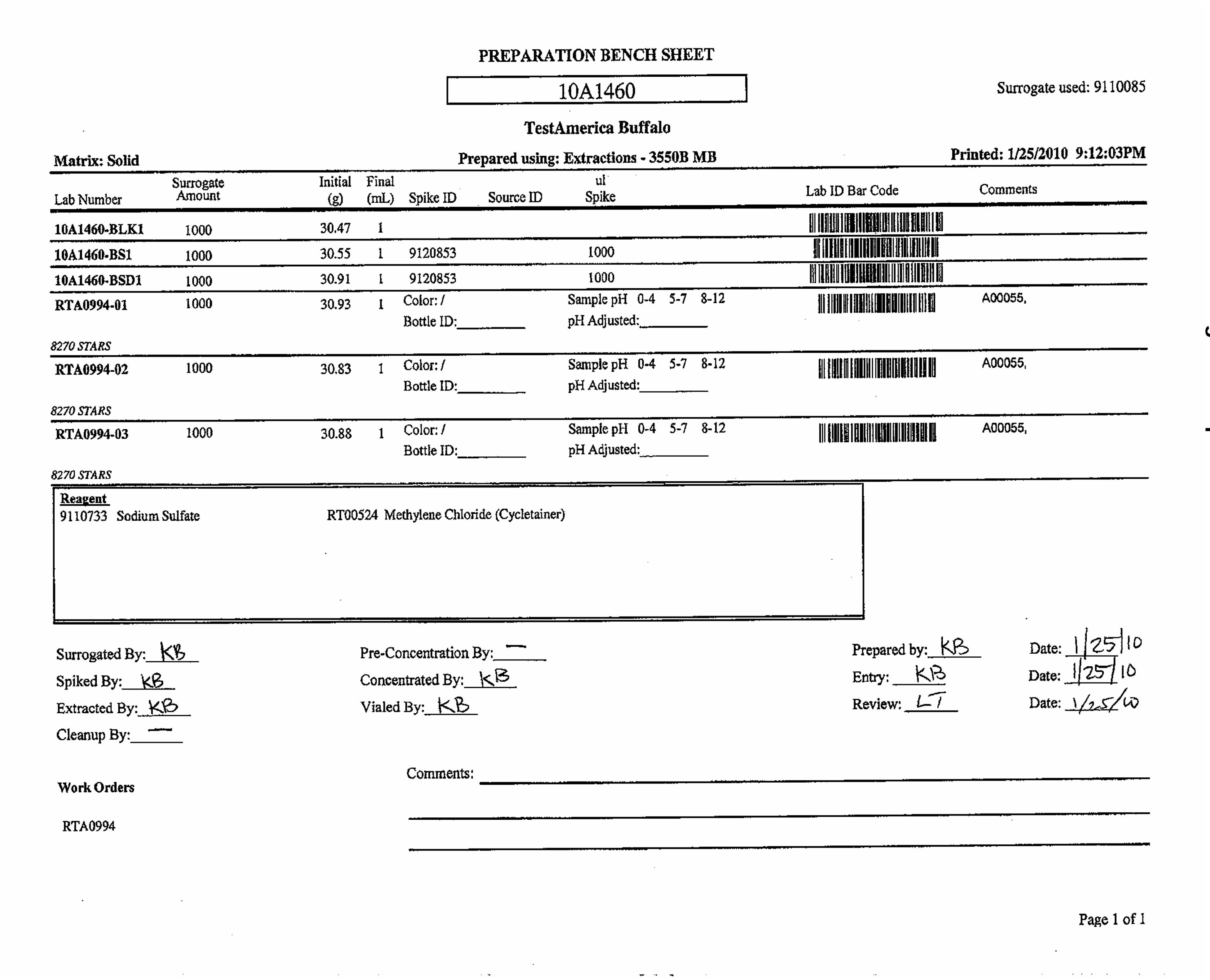
A01	Nitrobenzene-d5
	2-Fluorobiphenyl
	p-terphenyl
	Phenol-d5
	2-Fluorophenol
	2,4,6-Tribromophenol
A27	o-Terphenyl
A33	Dichlorophenyl Acetic Acid
A35	Tetrachloro-m-xylene
	Decachlorobiphenyl
A47	2,4-D
	Dalapon
	Dinoseb
	Pentachlorophenol
	Picloram
	2,4,5-TP (Silvex)
	2,4,5-T
	2,4-DB
	Dicamba
	Dichloroprop
A49	gamma-BHC (Lindane)
	Heptachlor
	Aldrin
	Dieldrin
	Endrin
	4,4'-DDT
A55	Phenol
	2-Chlorophenol
	1,4-Dichlorobenzene
	N-Nitroso-Di-n-Propylamine
	1,2,4-Trichlorobenzene
	4-Chloro-3-methylphenol
	Acenapththene
	4-Nitrophenol
	2,4-Dinitrotoluene
	Pentachlorophenol
	Pyrene

A95	Diesel Fuel #2	
A93	Tetrachloro-m-xylene	
	Decachlorobiphenyl	
A193	Acenaphthene	Dimethyl phthalate
	Aniline	4,6-Dinitro-2-methylphenol
	Acenaphthylene	2,4-Dinitrophenol
	Anthracene	2,4-Dinitrotoluene
	Benzo(a)anthracene	2,6-Dinitrotoluene
	Benzo(b)fluoranthene	Di-n-octyl phthalate
	Benzo(k)fluoranthene	Fluoranthene
	Benzo(ghi)perylene	Fluorene
	Benzo(a)pyrene	Hexachlorobenzene
	Benzoic Acid	Hexachlorobutadiene
	Benzyl alcohol	Hexachlorocyclopentadiene
	Bis(2-chloroethoxy) methane	Hexachloroethane
	Bis(2-chloroethyl) ether	Indeno(1,2,3-cd)pyrene
	2,2'-Oxybis(1-Chloropropane)	Isophorone
	Bis(2-ethylhexyl)phthalate	2-Methylnaphthalene
	4-Bromophenyl phenyl ether	2-Methylphenol
	Butyl benzyl phthalate	4-Methylphenol
	4-Chloroaniline	Naphthalene
	4-Chloro-3-methylphenol	2-Nitroaniline
	2-Chloronaphthalene	3-Nitroaniline
	2-Chlorophenol	4-Nitroaniline
	4-Chlorophenyl phenyl ether	Nitrobenzene
	Chrysene	2-Nitrophenol
	Dibenzo(a,h)anthracene	4-Nitrophenol
	Dibenzofuran	N-Nitrosodiphenylamine
	Di-n-butyl phthalate	N-Nitroso-Di-n-prpoylamine
	1,2-Dichlorobenzene	Pentachlorophenol
	1,3-Dichlorobenzene	Phenanthrene
	1,4-Dichlorobenzene	Phenol
	3,3-Dichlorobenzidine	Pyrene
	2,4;-Dichlorophenol	1,2,4-Trichlorobenzene
	Diethyl phthalate	2,4,5-Trichlorophenol
	2,4-Dimethylphenol	2,4,6-Trichlorophenol
A222	Aroclor 1016	

A57	Phenol
	2-Chlorophenol
	1,4-Dichlorobenzene
	N-Nitroso-Di-n-propylamine
	1,2,4-Trichlorobenze
	4-Chloro-3-methylphenol
	Acenaphthene
	4-Nitrophenol
	2,4-Dinitrotoluene
	Pentachlorophenol
	Pyrene

	Aroclor 1260	
A225	Gamma-BHC-(Lindane)	Endosulfan I
	alpha-BHC	Endosulfan II
	Heptachlor	Endrin Aldehyde
	Aldrin	Endosulfan Sulfate
	Beta-BHC	Heptachlor epoxide
	Dieldrin	Methoxychlor
	Endrin	Endrin Ketone
	4,4;-DDD	alpha-Chlordane
	4,4'-DDT	gamma-Chlordane
	4,4;-DDE	

Organic Prep Worksheet



PREPARATION BENCH SHEET

10A1460

Surrogate used: 9110085

TestAmerica Buffalo

Matrix: Solid

Prepared using: Extractions - 3550B MB

Printed: 1/25/2010 9:12:03PM

Lab Number	Surrogate Amount	Initial (g)	Final (mL)	Spike ID	Source ID	ul Spike	Lab ID Bar Code	Comments
10A1460-BLK1	1000	30.47	1					
10A1460-BS1	1000	30.55	1	9120853		1000		
10A1460-BSD1	1000	30.91	1	9120853		1000		
RTA0994-01 *8270 STARS*	1000	30.93	1	Color: / Bottle ID:________		Sample pH 0-4 5-7 8-12 pH Adjusted:________		A00055,
RTA0994-02 *8270 STARS*	1000	30.83	1	Color: / Bottle ID:________		Sample pH 0-4 5-7 8-12 pH Adjusted:________		A00055,
RTA0994-03 *8270 STARS*	1000	30.88	1	Color: / Bottle ID:________		Sample pH 0-4 5-7 8-12 pH Adjusted:________		A00055,

Reagent

9110733 Sodium Sulfate

RT00524 Methylene Chloride (Cycletainer)

Surrogated By: KB
Spiked By: KB
Extracted By: KB
Cleanup By: —

Pre-Concentration By: —
Concentrated By: KB
Vialed By: KB

Prepared by: KB Date: 1/25/10
Entry: KB Date: 1/25/10
Review: LT Date: 1/25/10

Comments: ____________________

Work Orders

RTA0994

Page 1 of 1

Title: Analytical Methods for GC/MS Semivolatile Samples by SW846 3rd Edition

8270C

Once printed, this is considered an uncontrolled document

Approvals (Signature/Date):

David C. Wilkes	01/29/10	Kenneth E. Kasperek	02/01/10
David Wilkes Department Manager	Date	Kenneth Kasperek Technical Director	Date
Peggy Gray-Erdmann	01/29/10		01/29/10
Peggy Gray-Erdmann Quality Assurance Manager	Date	Christopher Spencer Laboratory Director	Date

Facility Distribution No. ___________ **Distributed To:**________________________

1.0 Scope and Application

1.1 Analytes, Matrix(s), and Reporting Limits

The analytical method is utilized for the analysis of water, air sampling media, sediment and soil from hazardous waste sites for the organic compounds listed in Table 1. Table 1 includes CAS numbers and estimated quantitation limits for each analyte. Typical sample size should be 30 grams for soils and 1 liter for waters. The method begins with the extraction of the sample aliquot either by sonication (soils) or separatory funnel extraction (waters), into 1:1 methylene chloride/ acetone mixture. The extraction volume is then concentrated to 1.0ml final volume for waters and soils. The extracts are prepared for analysis with the addition of internal standard to each vial. One microliter of each extract is then directly injected into a gas chromatograph and the compounds are separated by mass using a capillary column and analyzed using a mass spectrometer. A summary of the analysis procedure is provided in Attachment A.

On occasion clients may request modifications to this SOP. These modifications are handled following the procedures outlined in Section xx in the Quality Assurance Manual.

2.0 Summary of Method

See Scope and Application

3.0 Definitions

Additional definitions scan be found in the TAL Buffalo Laboratory Quality Manual (LQM)

4.0 Interferences

Some of the possible interferences that arise during GCMS Semivolatile analysis include,but are not limited to:

1. Glassware contamination
2. Matrix interference
3. Aldol condensation
4. System air leaks
5. Injection port/liner contamination
6. Warped filament, and/or dirty source and rods
7. APIX analytes Methapyrilene and Phentermine split at all concentrations and require manual integration in calibration standard.

Raw GC/MS data from all blanks, samples, and spikes must be evaluated for interferences. Determine if the source of interference is in the preparation and/or cleanup of the samples and take corrective action to eliminate the problem.
.

4.1 See section 1.4 and 3.0 of method 8270c for other interferences, with the exception that there is no carryover in direct injection GCMS.

5.0 Safety

Employees must abide by the policies and procedures in the Corporate Environmental Health and Safety Manual (CW-E-M-001), Radiation Safety Manual and this document. This procedure may involve hazardous material, operations and equipment. This SOP does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of the method to follow appropriate safety, waste disposal and health practices under the assumption that all samples and reagents are potentially hazardous. Safety glasses, gloves, lab coats and closed-toe, nonabsorbent shoes are a minimum.

5.1 Specific Safety Concerns or Requirements

Chemicals that have been classified as carcinogens or potential carcinogens, under OSHA include: Benzo(a)anthracene, benzidine, 3,3'-dichlorobenzidine, benzo(a)pyrene, dibenzo(a,h)anthracene, and n-nitrosodimethylamine. Primary standards should be purchased in solution. If neat materials must be obtained, they shall be handled in a hood.

Exposure to chemicals must be maintained as low as reasonably achievable; therefore, unless they are known to be non-hazardous, all samples should be opened, transferred, and prepared in a fume hood, or under other means of mechanical ventilation. Solvent and waste containers should be kept closed unless transfers are being made.

Analysts are expected to use caution and common sense while working in a laboratory environment. Each employee is required to read the companies' Corporate Safety Manual. All of the samples to be analyzed have the potential to contain hazardous substances. Most standards also contain hazardous chemicals and many do contain known carcinogens. Employees must use protective equipment when handling standards, samples and extracts including gloves, lab coats and safety glasses. It is the analyst's responsibility to read and familiarize themselves with the MSDS of each chemical and/or reagent involved in this method.

Samples, standards and/or extracts should never be opened or transferred outside of a fume hood.

Waste disposal is all C waste with the exception of some acids used in the cleaning of equipment which is disposed of in AN waste.

Spills should be cleaned up promptly and waste should be disposed of as per the Chemical Hygiene Plan.

There is also the danger of burns while doing repair or maintenance on a gas chromatograph. One must use caution while working on or near the injection port or transfer line.

5.2 Primary Materials Used

The following is a list of the materials used in this method, which have a serious or significant hazard rating. **Note: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the MSDS for each of the materials listed in the table.** A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the MSDS for each material before using it for the first time or when there are major changes to the MSDS.

Material (1)	**Hazards**	**Exposure Limit (2)**	**Signs and symptoms of exposure**
Methanol	Flammable Poison Irritant	200 ppm-TWA	A slight irritant to the mucous membranes. Toxic effects exerted upon nervous system, particularly the optic nerve. Symptoms of overexposure may include headache, drowsiness and dizziness. Methyl alcohol is a defatting agent and may cause skin to become dry and cracked. Skin absorption can occur; symptoms may parallel inhalation exposure. Irritant to the eyes.
Methylene Chloride	Carcinogen Irritant	25 ppm-TWA 125 ppm-STEL	Causes irritation to respiratory tract. Has a strong narcotic effect with symptoms of mental confusion, light-headedness, fatigue, nausea, vomiting and headache. Causes irritation, redness and pain to the skin and eyes. Prolonged contact can cause burns. Liquid degreases the skin. May be absorbed through skin.
Sodium Hydroxide	Corrosive	2 Mg/M3-Ceiling	Severe irritant. Effects from inhalation of dust or mist vary from mild irritation to serious damage of the upper respiratory tract, depending on severity of exposure. Symptoms may include sneezing, sore throat or runny nose. Contact with skin can cause irritation or severe burns and scarring with greater exposures. Causes irritation of eyes, and with greater exposures it can cause burns that may result in permanent impairment of vision, even blindness.
Sulfuric Acid	Corrosive Oxidizer Dehydrator Poison Carcinogen	1 Mg/M3-TWA	Inhalation produces damaging effects on the mucous membranes and upper respiratory tract. Symptoms may include irritation of the nose and throat, and labored breathing. Symptoms of redness, pain, and severe burn can occur. Contact can cause blurred vision, redness, pain and severe tissue burns. Can cause blindness.
1 – Always add acid to water to prevent violent reactions.			
2 – Exposure limit refers to the OSHA regulatory exposure limit.			

6.0 <u>Equipment and Supplies</u>

6.1 Calibrated micro syringes 10, 25, 50, 100, 500, 1,000 microliter.

6.2 2ml amber vials and caps.

6.3 Disposable pipets and pipet bulbs.

6.4 Volumetric flasks.

6.5 Instrumentation

Gas Chromatograph/Mass Spectrometer (GC/MS) System

6.5.1 Gas Chromatograph -
- Hewlett Packard 6890
- Carrier gas Helium UPC grade or equivalent

6.5.2 Gas Chromatography Column
- Analysis: RESTEK Rxi-5Sil MS w/Integra-Guard (cat.#13623-127) or equivalent

6.5.3 Mass Spectrometer
- HP5973 and HP5973 inert
- Tuning compound PFTBA
- Scan Range 35-500 AMU/second

6.5.4 Data System
- HP Chemstation
- Teknivent and HP enviroquant software

7.0 Reagents and Standards

7.1 Methylene Chloride – high purity

7.2 Standards:

7.2.1 Stock Standards
CLP Semivolatile Calibration Mix 1000µg/ml
Calibration Mix #2 2000µg/ml
Benzidines Mix 2000 µg/ml
N-Nitrosodiphenylamines 5000 µg/ml
OLM Mix 2000 µg/ml
Benzoic Acid 2000 µg/ml
BN/AP Surrogate Mix 4000 µg/ml
DFTPP mix 50µg/ml or equivalent
Internal Standard Mix 2.0 mg/ml

All Certificates of Analysis received from the manufacturer are maintained in a laboratory lims system. Stock standards are prepared every twelve months or sooner, if necessary.

7.2.2 Initial and Continuing Calibration Solutions
8270 Stock Solution

Standard	Solvent	Stock Conc.	Initial Wt/Vol.	Final Vol.	Final Conc.	Final Conc. In Samples
CLP Semivo Calibration Mix	$MECL_2$	1000 ng/ul	400µl	2000ul	200 ng/ul	200 ug/L
Calibration Mix # 4	$MECL_2$	2000 ng/ul	200µl	2000ul	200 ng/ul	200 ug/L
Benzidines Mix	$MECL_2$	2000 ng/ul	200µl	2000ul	200 ng/ul	200 ug/L
N-Nitrosodiphenylamine Mix	$MECL_2$	5000 ng/ul	80µl	2000ul	200 ng/ul	200 ug/L
BN/AP Mix	$MECL_2$	4000 ng/ul	100µl	2000ul	200 ng/ul	200 ug/L
OLM Mix	$MECL_2$	2000 ng/ul	200µl	2000ul	200 ng/ul	200 ug/L
Benzoic Acid	$MECL_2$	2000 ng/ul	400µl	2000ul	200 ng/ul	400 ug/L

7.2.3 Working Standards

7.2.3.1

Surrogate Standard Spiking Solution is prepared that contains nitrobenzene-d5, terphenyl1-d14, 2-fluorobiphenyl, and 1,2-dichlorobenzene-d4 at a concentration of 100µg/ml; phenol-d5, 2,4,6-tribromophenol, 2-fluorophenol and 2-chlorophenol-d4 at a concentration of 150µg/ml. Surrogate standards are added to all samples and calibration solutions. Additional surrogates may be added at the laboratory's discretion.

Standard	***Solvent***	Stock Conc.	Initial Wt/Vol.	Final Vol.	Final Conc. In Samples
Semivolatile Acid Surrogate Phenol-d5 2,4,6-Tribromophenol 2-Fluorophenol 2-Chlorophenol-d4	MEOH	10,000ng/ul	1,500ul	100,000ul	150ug/L
Semivolatile B/N Surrogate Nitrobenzene-d5 Terphenyl-d14 2-Fluorobiphenyl 1,2-Dichlorobenzene-d4	MEOH	5000ng/ul	2,000ul	100,000ul	100ug/L

7.2.3.2.

Matrix Spiking Solution (11 compound)

The 11 compound matrix spiking solution consists of the following:

<u>Bases/Neutrals</u>	<u>Acids</u>
1,2,4-Trichlorobenzene	Pentachlorophenol
Acenaphthene	Phenol
2,4-Dinitrotoluene	2-Chlorophenol
Pyrene	4-Chloro-3-methylphenol

N-Nitroso-di-n-propylamine
1,4-Dichlorobenzene
4-Nitrophenol

a. Using the Intermediate Acid and BN Standards, the Matrix Spike solution is prepared that contains each of the base-neutral compounds above at 100µg/ml in methanol and the acid compounds at 100µg/ml in methanol.

Standard	Solvent	Stock Conc.	Initial Wt/Vol.	Final Vol.	Final Conc. in Solution	Final Conc. In Aqueous Samples
Acid Matrix Spike Intermediate BN Matrix Spike Intermediate	MeOH MeOH	10000ng/ul 5000ng/ul	5000uls 10000 uls	500mls 500mls	100 ug/ml 100 ug/ml	100 µg/L 100 ug/L

7.2.3.3 Matrix Spiking Solution (all compound)

The all compound matrix spiking solution contains each of the following SVOA target analytes at 100µg/ml in methanol. Additional compounds may be included in the spike mixture if required for a specific project.

Ancenaphthene	Dibenzo(a,h)anthracene	Indeno(1,2,3-cd)pyrene
Acenaphthylene	Dibenzofuran	Isophorone
Anthracene	di-n-butyl phthalate	2-Methylnaphthalene
Benzo(a)anthracene	1,2-Dichlorobenzene	2-Methylphenol
Benzo(b)fluoranthene	1,3-Dichlorobenzene	4-Methylphenol
Benzo(k)fluoranthene	1,4-Dichlorobenzene	Naphthalene
Benzo(ghi)perylene	3,3'Dichlorobenzidine	2-Nitroaniline
Benzo(a)pyrene	2,4-Dichlorophenol	3- Nitroaniline
Benzoic acid	Diethyl phthalate	4- Nitroaniline
Benzyl alcohol	2,4-Dimethylphenol	Nitrobenzene
Bis(2-chloroethoxy)methane	Dimethyl phthalate	2-Nitrophenol
Bis(2-chloroethyl)ether	4,6-Dinitro-2-methylphenol	4-Nitrophenol
2,2'-oxybix(1-Chloropropane)	2,4-Dinitrophenol	N-nitrosodiphenylamine
Bis(2-ethylhexyl)phthalate	2,4-Dinitrotoluene	N-Nitroso-Di-n-propylamine
4-Bromophenyl phenyl ether	2,6-Dinitrotoluene	Pentachlorophenol
Butyl benzyl phthalate	Di-n-octyl phthalate	Phenanthrene
2-Chloroaniline	Fluoranthene	Phenol
4-Chloro-3-methylphenol	Fluorene	Pyrene
2-Chloronaphthalene	Hexachlorobenzene	1,2,4-Trichlorobenzene
2-Chlorophenol	Hexachlorobutadiene	2,4,5-Trichlorophenol
4-Chlorophenyl phenyl ether	Hexachlorocyclopentadiene	2,4,6-Trichlorophenol
Chrysene	Hexachloroethane	Tetra Ethyl Lead

1,4 Dioxane		

8.0 Sample Collection, Preservation, Shipment and Storage

Sample container, preservation techniques and holding times may vary and are dependent on sample matrix, method of choice, regulatory compliance, and/or specific contract or client requests. Listed below are the holding times and the references that include preservation requirements.

Matrix	Sample Container	Min. Sample Size	Preservation	Holding Time [1]	Reference
Waters	HDPE	50 mLs	HNO_3, pH < 2; Cool 4 $\pm$ 2°C	180 Days	40 CFR Part 136.3
Soils	Glass	3 grams	Cool 4 $\pm$ 2°C	180 Days	N/A

[1] Inclusive of digestion and analysis.

8.1 Water samples may be collected in 1L (or more) amber glass containers with Teflonlined, screw-caps.

8.2 Soil/Sediment Samples may be collected in glass containers fitted with Teflon-lined screw-caps or closed end tubes.

8.3 All samples are stored at 4 C (+/-2C) from the time of collection until extraction

8.4 Aqueous samples must be extracted within 7 days of collection and analyzed within 40 days of extraction.

8.5 Soil samples must be extracted within 14 days of collection and analyzed within 40 days of extraction.

9.0 Quality Control

9.1 Sample QC - The following quality control samples are prepared with each batch of samples.

Quality Controls	Frequency	Control Limit
Method Blank (MB)	1 in 20 or fewer samples	< Rpt. Limit
Laboratory Control Sample (LCS)[1]	1 in 20 or fewer samples	Statistical Limits [4]
Matrix Spike (MS)[2]	1 in 20 or fewer samples	Statistical Limits [4]
MS Duplicate (MSD)[2]	1 in 20 or fewer samples	Statistical Limits [4]
Surrogates	every sample[3]	Statistical Limits [4]

[1] LCS Duplicate (LCD) is performed only when insufficient sample is available for the MS/MSD or when requested by the client/project/contract.
[2] The sample selection for MS/MSD are randomly selected, unless specifically requested by a client….predetermined by the extraction lab.
[3] Analytical and QC samples (MB, LCS, MS/MSD)
[4] Statistical control limits are updated annually and are updated into LIMS.

Method Blanks - A method blank is a volume of a clean reference matrix (reagent water for water samples, or purified sodium sulfate/clean sand for soil/sediment samples) that is carried through the entire analytical procedure. The volume or weight of the reference matrix must be approximately equal to the volume or weight of samples associated with the blank. The purpose of a method blank is to determine the levels of contamination associated with the processing and analysis of samples.

9.1.1 For semivolatile analysis, a method blank for water samples consists of 1 L volume of reagent water spiked with 1.0mL of the surrogate spiking solution. For medium or low level soil/sediment samples, a method blank consists of 1g or 30g of sodium sulfate/clean sand spiked with 1.0mL of the surrogate spiking solution, respectively. Extract, concentrate, cleanup and analyze the blank according to procedures for water and soil samples.

9.1.2 Acceptance Criteria – levels of target analytes in the method blank must be less than the required reporting limit or less than one-tenth the concentration of the respective analyte in the associated samples. For USACE all target analytes must be less than one half of the MRL (Method Reporting Limit) and common laboratory contaminants must be less than the MRL. The MRL is set at either the MDL or the MDL Check.

9.1.3 Corrective Actions for Method Blank Analyses - If the acceptance criteria for method blank analysis are not met, the analytical system may be assumed to be out of control. The following corrective actions may be taken:

- If contamination is the problem, then the source of the contamination must be investigated and appropriate corrective measures must be taken and documented before further sample analysis proceeds. It is the laboratory's responsibility to ensure that method interferences caused by contaminants in solvent, reagents, glassware, and sample storage and processing hardware that lead to discrete artifacts and/or elevated baselines in the GC/MS be eliminated. Samples associated with the contaminated blank must be re-extracted and re-analyzed.

- If surrogate recoveries in the method blank do not meet the acceptance criteria, first reanalyze the method blank. If the surrogate recoveries do not meet the acceptance criteria after reanalysis, re-extract and re-analyze the blank and all associated samples OR the samples may be reported as estimated, and noted in the case narrative.

- If the method blank does not meet internal standard response requirements, check calculations, the internal standard spiking solutions, and the instrument operation. If the calculations were incorrect, correct the calculations and verify that the internal standard responses meet their acceptance criteria. If the internal standard compound spiking solution was improperly prepared, concentrated, or degraded, re-prepare solutions and re-extract/reanalyze samples. If the instrument malfunctioned, correct the instrument problem and reanalyze the method blank. If the instrument malfunction affected the calibration, recalibrate the instrument before reanalyzing the blank

9.2 Matrix Spike Blank/Matrix Spike/Matrix Spike Duplicate(MSB/MS/MSD)

9.2.1 A matrix spike blank, matrix spike and matrix spike duplicate are analyzed to evaluate the analytical system and the effects of sample matrix on the methods used for semivolatile analysis.

9.2.2 The matrix spike blank, matrix spike, and matrix spike duplicate are spiked with the compounds of interest (at concentrations noted in the standard preparation section).

9.2.3 A matrix spike blank, matrix spike and matrix spike duplicate are extracted and analyzed for every batch of 20 samples of a similar matrix. Matrix spike and matrix spike duplicates are not performed for field QC samples such as rinsates, or field/trip blanks

9.2.4 If insufficient sample amount is received to perform matrix spike and matrix spike duplicate analysis, duplicate matrix spike blanks may be processed.

9.2.5 Dilutions

Dilutions of MS/MSD samples are performed only if the unspiked sample requires a dilution in order to maintain any target compound concentrations in the upper half of the calibration. MS/MSD samples will not be diluted to get spiked or non-spiked compounds below the highest calibration standard.

9.2.6 Calculations for MS/MSD

The concentrations of spiked compounds are determined using equations described for sample analysis. After determining the compound concentrations, the percent recovery is calculated using Equation 1.

Equation 1

$$\text{Matrix Spike Recovery} = \frac{\text{SSR - SR}}{\text{SA}} x100$$

Where,

SSR= Spike Sample Result

SR = Sample Result

SA = Spike Added

The relative percent difference between the matrix spike and matrix spike duplicate is calculated using Equation 2.

Equation 2

$$RPD = \frac{[MSR - MSDR]}{1/2\,(MSR + MSDR)} \text{ x } 100$$

Where,

RPD = Relative Percent Difference

MSR = Matrix Spike Recovery

MSDR = Matrix Spike Duplicate Recovery

The vertical bars in the formula above indicate the absolute value of the difference, hence RPD is always expressed as a positive value

9.2.7 Technical Acceptance Criteria for MS/MSD

The acceptance criteria for sample analysis (retention time, surrogate and IS recovery) must be met for matrix spike and matrix spike duplicate analysis also.

The matrix spike recovery limits are based on historical data and are updated annually.

The matrix spike recovery limits are advisory. If the recovery limits are not met, no further corrective action will be necessary. However, frequent occurrences of this nature should be investigated.

Re-extraction and re-analysis of the matrix spike and matrix spike duplicate may be necessary if, in the technical judgment of the analyst and/or supervisors, an error was made during the extraction procedure

9.2.8 Technical Acceptance Criteria for MSB:

The acceptance criteria for sample analysis (retention time, surrogate and IS recovery) must be met for the matrix spike blank analysis also.

The matrix spike blank recovery limits are based on historical data and are updated annually.

If the Matrix Spike Blank was found to be unacceptable all samples in the associated batch must be re-extracted and re-analyzed. If the sample was not within extraction hold time, a job exception must be filed and both analysis must be included with the report.

9.2.9 Surrogate Recoveries

The surrogate compound concentrations are determined using calculations found in Section 9.1.1. The recoveries are then determined using Equation 3

Equation 3

$$\% \; Recovery = \frac{Concentration\,(\vee\, amount)\; found}{Concentration\,(\vee\, amount)\; spiked}$$

Recovery limits for surrogate compounds are based on historical data and are updated annually.

9.2.10 QC Acceptance Criteria for AFCEE or USACE projects are provided in Attachments B and C respectively.

9.3 Instrument QC

Instrument Operating Conditions

- Gas Chromatograph; The following are recommended GC conditions that may vary slightly depending on the compound list and the column film thickness.

Initial Temperature: 40-50°C
Initial Hold Time: 3 minutes (hold time may vary to ensure proper chromatographic separation).
Temperature program 40-50°C to 70°C at 20°C/min to 195 at 16°C/min to 325 at 30°C/min
Final Temperature: 325°C
Final Hold Time: As necessary for TCL compound identification
Injector Temperature: 250°C
Source Temperature: 230°C
Transfer Line Temperature: 310°C
Injector: splitless
Front Inlet Pressure: 7.00 psi
Purge Flow: 15.0 mL/min
Purge Time: 0.50 min
Total flow: 19.2 mL/min
Injection Volume: 1µl
Carrier Gas: Helium
Carrier Flow: 36 cm/sec

- Mass Spectrometer

Electron Energy: 70 volts (nominal)
Mass Range: 35 to 500 amu
Scan Time: Not to exceed 1 second per scan

9.4 Instrument Performance Check

The GC/MS system is tuned using Perfluorotributylamine (PFTBA) such that an injection of 50ng of DFTPP will meet the abundance criteria listed in Table 2.

Prior to the analysis of standards or samples, the mass calibration and resolution of the GC/MS system is verified by the analysis of DFTPP. This analysis will verify the proper tuning of the system for 12 hours. After 12 hours, the instrument performance must be verified before standard and sample analysis may continue.

The mass spectrum of DFTPP may be background subtracted to eliminate column bleed or instrument background ions.

Breakdown of 4,4'-DDT into 4,4'-DDD and 4,4'-DDE may be used to assess GC column performance and injection port inertness and must be less than 20%.

The compounds Benzidine and Pentachlorophenol should be present and at their normal responses for this concentration. Peak tailing should not be visible (PCP tailing factor <5 and Benzidine <3). If responses are poor and excessive peak tailing is present, corrective actions for the GC/MS instrument performance check solution may be required. Benzidine and Pentachlorophenol tailing may also be verified in the CCV.

All subsequent standards and samples must be acquired under the same GC/MS tuning conditions that were used for the analysis of the instrument performance check solution.

9.4.2 Technical Acceptance Criteria for the GC/MS Instrument Performance Check (DFTPP) is listed in Table 2.

9.4.3 Corrective Actions for the GC/MS Instrument Performance Check
If any of the acceptance criteria are not met, the DFTPP should be re-injected to insure that the injection made was not a cause for failure. If, after reinjection, acceptance criteria has not been met, one or more of the following corrective actions may be taken:

1. Retune the GC/MS
2. Clean the source; replace parts, etc...
3. Cut the column at the injector end
4. Replace the column
5. Replace the septum in the injector
6. Replace the injector liner
7. Clean injection port with $MeCl_2$
8. Change injection port seal
9. An instrument service call may be placed.

9.5 Initial Calibration
After the instrument performance check criteria has been met and prior to the analysis of samples, the GC/MS system is calibrated at a minimum of five concentration levels in order to establish instrument sensitivity and linearity.

The initial calibration shall be performed when major instrument maintenance has been performed or if continuing calibration criteria cannot be met.

Major instrument maintenance may consist of source cleaning, column changing, or quadrapole rod adjustment. Preventative maintenance such as septum changes, injector liner changes or column cutting may not require an initial calibration to be performed.

9.5.1 Procedure

Five calibration standards are prepared which contain all target and surrogate compounds. A 20µl aliquot of internal standard solution is added to a 1mL aliquot of each calibration standard solution. The resulting concentration of internal standards is 40ng. A 1µl injection would result in a final concentration of 40ng on column. The internal standards used are given in Table 3.

The relative response factors (RRF) for each target and surrogate compound is determined using equation 4. The characteristic ions for a given compound are listed in Tables 3 and 6. Internal standard assignments are listed in Table 4.

Equation 4

$$RRF = \frac{A_x}{A_{is}} \, x \, \frac{C_{is}}{C_x}$$

Where,
A_x = Area of the characteristic ion for the compound to be measured (see Table 4)
A_{is} = Area of the characteristic ion for specific internal standard (see Table 3)
C_{is} = Amount of the internal standard injected (ng)
C_x = Amount of the compound to be measured injected (ng)

The mean relative response factor (RRF) must be calculated for all compounds. Calculate the % Relative Standard Deviation (%RSD) of the RRF values for the initial calibration using the following equation:

Equation 5

$$\%RDS = \frac{Standard\ Deviation}{Mean} \, x \, 100$$

Where,

$$Standard\ Deviation = \sqrt{\frac{\sum_{i+1}^{n} (X_i - \overline{X})^2}{(n-1)}}$$

x_i = each individual value used to calculate the mean
x = the mean of n values
n = the total number of values

9.5.2 Acceptance Criteria for Initial Calibration

The average response factor (RRF) for each System Performance Check Compound (listed in Table 5) must be greater than or equal to the compound's minimum acceptable relative response factor of 0.050.

The %RSD over the initial calibration range for relative response factor for each Calibration Check (Table 5) compound %RSD must be less than or equal to the 30%.

The %RSD over the initial calibration range for the relative response factor for all other compounds must be less than or equal to 15%.

OR

A least squares regression correlation coefficient of greater than 0.990 for all compounds greater than 15% RSD.

OR

A non-linear coefficient of determination of greater than 0.990 for all compounds greater than 15% RSD. For a 2nd order non-linear regression, 6 calibration points must be used and for a 3rd order non-linear regression, 7 calibration points must be used.

9.5.3 Corrective Actions for Initial Calibration

If any of the acceptance criteria for initial calibration are not met, it may be necessary to reanalyze one or more of the calibration standards. If after reanalysis, the acceptance criteria have not been met, it may be necessary to take further corrective actions.

The following corrective actions may be taken if the acceptance criteria for initial calibration cannot be met.

1. Prepare fresh standards and reanalyze the initial calibration.
2. Replace the septum on the injector
3. Replace the injector liner
4. Cut the column at the injector end
5. Retune the GC/MS system and reanalyze the instrument performance check
6. Clean the source
7. An instrument service call may be placed

The acceptance criteria must be met before sample analysis may proceed.

9.5.4 Initial Calibration Verification

To verify the accuracy of the initial calibration, a standard is obtained from a source different from the calibration standards.

Immediately following analysis of an acceptable initial calibration curve, a 80ng/µl aliquot of this independent standard is injected.

Recoveries of all compounds shall fall within ±25% of the expected value, however, recoveries of up to 40% are allowable for up to four compounds.

9.5.5 Continuing Calibration

If there is no time left in the 12-hour time period after initial calibration, the instrument performance check may be analyzed and a 50ng/1µl standard may be analyzed to verify the calibration of the instrument.

The continuing calibration check must be analyzed once every 12-hour time period of operation. This check must be analyzed prior to the analysis of samples for a given 12-hour time period.

9.5.6 Procedure for Continuing Calibration

The 50ng/µl standard is used for the continuing calibration. The relative response factor is calculated using procedures described for initial calibration.

If quantitation is performed using response factor, calculate the percent difference between the mean relative response factor from the most recent initial calibration and the continuing calibration relative response factor for each semivolatile target and surrogate compound using Equation 6.

Equation 6

$$\%\ Difference_{RRF} = \frac{RRF_c - \overline{RRF_i}}{\overline{RRF_i}} \ x\ 100$$

Where,

$\overline{RRF_i}$ = Mean relative response factor from the most recent initial calibration meeting technical acceptance criteria
RRF_c = Relative response factor from continuing calibration standard

If quantitation is performed using a least squares regression or a non-linear model, calculate the concentration of all analytes and surrogates in the continuing calibration as described in section 8.3.2 of this SOP. Calculate the percent drift using Equation 7.

Equation 7:

$$\%\mathrm{Drift} = \frac{\mathrm{Conc}_E - \mathrm{Conc}_A}{\mathrm{Conc}_E} x 100$$

Where:

Conc_E = Expected Concentration
Conc_A = Actual Concentration

9.5.7 Acceptance Criteria for Continuing Calibration

The relative response factor (RRF) for each System Performance Check Compound must be greater than or equal 0.050.

The RRF of percent drift for Calibration Check Compounds must be less than 20%. The RRF percent difference or percent drift for all other compounds including TCL list compounds must be within ±25%, with up to four compounds within ±40%D. For expanded list and additional compounds not on the EPA TCL list a percent drift of 40% is allowed. Any analyte may have an elevated response >40%D if it is not detected in the associated samples, with the exception of APIX and priority pollutant compounds +/- 100%D.

Internal Standard retention times and responses are evaluated after acquisition of the continuing calibration check. If the retention time of any internal standard shifts by more than 30 seconds or the response of any internal standard is outside of the-50%to +100% range, the system shall be inspected and corrected as needed. The CCV will be reanalyzed after inspection. If the problem is not resolved, a new initial calibration must be performed.

9.5.8 Corrective Actions for Continuing Calibration

If any of the technical acceptance criteria for continuing calibration are not met, it may be necessary to reanalyze the continuing calibration standard. If after reanalysis the acceptance criteria cannot be met, further corrective actions may be required.

The following corrective actions may be taken if the acceptance criteria for continuing calibration cannot be met.

1. Replace the septum on the injector
2. Replace the injector liner
3. Replace injection port seal
4. Cut the column at the injector end
5. Retune the GC/MS system and reanalyze the instrument performance check
6. Prepare fresh standards
7. Reanalyze the initial calibration

9.5.9 Calibration acceptance criteria for AFCEE and USACE projects are provided in Attachments B and C respectively.

9.6 Calibration Acceptance Summary

Include a line for each appropriate parameter you used above.

Step	***Standards***	***Type***	***Control Limit***	***Frequency***
Method #8270				
Initial Cal	*Conc and # of stds*	*Type of Cal: Linear, …*		*How often performed?*
ICV	*80ng*	*LINEAR*	*+/- 25%*	*After initial cal.*

CCV	*50ng*	*LINEAR*	*+/- 20%*	

10.0 Procedure

10.1 Sample extracts shall be analyzed only after the GC/MS system has met the instrument performance check, initial calibration, continuing calibration and second source calibration verification requirements. The same instrument conditions must be employed for the analysis of samples as were used for calibration.

Internal standard solution is added to each sample extract. 20µL of internal standard solution is added to each accurately measured 1.0mL of water sample extract. For soil/sediment samples and water samples subjected to GPC, 10µL of internal standard solution is added to each accurately measured 0.5mL of sample extract. This will result in a concentration of 40ng/µL of each internal standard. The amount of internal standard needs to be adjusted according to how much extract volume was present in the extract vial. The exact volume of extract is measured using a syringe. The amount of Internal Standard solution to be added is then adjusted accordingly. The calculation to determine the amount of IS to add is provided below:

Vol. Extract (ml) X 20 ul = FV of IS

Necessary dilutions are made prior to adding internal standard solution. The internal standard solution must be added so that the concentration of each internal standard is 40ng/µL.

10.2. Dilutions

Dilutions of sample extracts are required if any target compound exceeds the initial calibration range.
The dilution chosen should keep the response of the largest target compound within the calibration range.

10.2.1 Sample Log Book Entry
Samples are logged into the electronic instrument logbook prior to the start of the analysis.

10.3. Qualitative Identification

10.3.1 Target Compounds
Target compound identification is done by comparing the sample mass spectrum to that of the standard. The following criteria must be satisfied in order to verify identifications.
Elution of the sample analyte within GC relative retention time unit window established from the 12-hour calibration standard.

Correspondence of the sample analyte and calibration standard component mass spectra.

To establish correspondence of the GC relative retention time (RRT), the sample component RRT must compare with ±0.06 RRT units of that of the standard RRT. If samples are analyzed within the same 12-hour period as the initial calibration, the 50ng standard is used to verify relative retention times.

To establish correspondence of the sample component mass spectra to that of the standard, the following criteria must be met:

- All ions present in the standard mass spectrum at a relative intensity greater than 10.0 percent (most abundant ion in the spectrum equals 100.0 percent) must be present in the sample spectrum.
- The relative intensities of ions specified in the paragraph above must agree within ±20.0 percent between the standard and sample spectrum. (Example: For an ion with an abundance of 50.0 percent in the standard spectrum, the corresponding sample ion abundance must be between 30.0 and 70.0 percent).
- Ions greater than 10.0 percent in the sample spectrum but not present in the standard spectrum must be considered and accounted for by the analyst making the comparison. The verification process should favor false positives. All compounds meeting the identification criteria must be reported with their spectra. When target compounds are below contract required quantitation limits (CRQL) but the spectrum meets the identification criteria, report the concentration with a "J".

If a compound does not meet all of the above criteria, but in the technical judgement of the mass spectral interpretation specialist the identification is correct, the compound will be identified. Documentation of such by the specialist on the raw data is required.

10.3.2 Non-Target Compounds

A library search may be executed for non-target sample components for the purpose of tentative identification. For this purpose, the NIST/EPA/NIH mass spectral library is used to identify non-target compounds of greatest apparent concentration by a forward search of the library. The following compounds will not be identified by a library search routine:

a. Internal standard compounds
b. Surrogate compounds
c. Volatile target compounds

Peaks that are suspected to be aldol-condensation reaction products (i.e., 4-methyl-4-hydroxy-7-pentanone and 4-methyl-3-pentene-2-one) are searched and reported as part of the 30 tentatively identified compounds.

10.3.3 Guidelines for Making Tentative Identifications

Major ions in the reference spectrum (ions greater than 10 percent of the most abundant ion) should be present in the sample spectrum.

The relative intensities of the major ions should agree within ±20 percent. Molecular ions present in reference spectrum should be present in sample spectrum.

Ions present in the sample spectrum but not in the reference spectrum should be reviewed for possible background contamination or presence of co-eluting compounds.

Ions present in the reference spectrum but not in the sample spectrum should be reviewed for possible subtraction from the sample spectrum because of background contamination or co-eluting compounds.

If, in the technical judgement of the mass spectral interpretation specialist, no tentative identification can be made the compound will be reported as unknown. Further identification may be possible, such as molecular weights or classifications (i.e., unknown hydrocarbon, unknown acid, etc.)

Pesticide target compounds may be tentatively identified by a library search.

10.4 Technical Acceptance Criteria For Sample Analysis

The samples must be analyzed on a GC/MS system meeting the DFTPP initial calibration, continuing calibration, and blank technical acceptance criteria. The sample must undergo cleanup procedures, when required, on a GPC meeting the acceptance criteria for GPC calibration.

The sample must be extracted and analyzed within the holding times.

The sample must have an associated method blank meeting the blank acceptance criteria. All Matrix Spike Blank recoveries must fall within the laboratory derived limits. Recoveries above the upper control limit are acceptable as long as the analyte was not detected in the associated samples above the quantitation limit.

All surrogates should fall within the laboratory derived limits (Up to one BN and/or one AP surrogate may fall outside the control limit as long as the recovery is greater than 10%).

The relative retention time of each surrogate must be within ±0.06 RRT units of its relative retention time in the continuing calibration standard.

The instrumental response (EICP area) for each of the internal standards must be within the inclusive range of -50.0 percent and +100.0 percent of the response of the internal standards in the most recent continuing calibration analysis.

The retention time shift for each of the internal standards must be within ±0.50 minutes (30 seconds) between the sample and the most recent continuing calibration standard analysis.

Excluding those ions in the solvent front, no ion may saturate the detector. No target compound concentration may exceed the upper limit of the 12-hour standard calibration range unless a more dilute aliquot of the sample extract is also analyzed.

10.5 Corrective Actions for Sample Analysis

The technical acceptance criteria must be met before data are reported. Contamination from laboratory sources requires re-extraction and reanalysis.

10.5.1 Surrogate Compounds

If the technical acceptance criteria for surrogate compound recoveries is not met, the following corrective actions are taken in the given order:

a. Calculations, injection volumes, preparation volumes are checked to insure that an error was not made; if all calculations, volumes, etc., were correct the analyst will proceed to the next step in the corrective action process.

b. The sample is re-injected to insure that an error during injection was not made. If after re-injection, surrogate recoveries are outside of the acceptance criteria, the analysis will proceed to the next step in the corrective action process.

c. The sample is re-extracted. Exceptions: (1) in the case where the recoveries in a sample, MS/MSD agree (i.e., all samples exhibited recoveries outside of criteria limits) it will be noted in the Case narrative. (2) Insufficient sample remains for re-extraction. In this instance, the client will be contacted in order to determine the next procedure to follow. If this situation should arise, it will be documented in the Case narrative. (see form B: Re-extraction request form).

d. After re-extraction, the sample is re-injected. If after re-analysis surrogate recoveries are within criteria limits, this extract is considered the first because the original problem may have been due to a laboratory error. If, after re-analysis surrogate recoveries are not within criteria limits, a matrix effect may be assumed. If this should occur, both analyses may be reported. The instance will be documented in the Case Narrative.

10.5.2 Internal Standard Compounds

If the technical acceptance criteria for internal standard recoveries is not met, the following corrective actions are taken in the given order:

a. Calculations, internal standard solution volumes and injected volumes are checked to insure that an error was not made. If all calculations and volumes were correct the analyst will proceed to the next step in the corrective action process.

b. The sample is re-injected to insure that the instrument was working properly. If after re-analysis, the internal standard recoveries are with criteria limits, the second analysis will be reported only. If after re-analysis the internal standard recoveries are outside of criteria limits, both analyses will be reported and it may be assumed that a matrix effect was involved. If this instance should arise, it will be documented in the Case Narrative.

Exception: If internal standard recoveries of a sample, MS/MSD agree (i.e., recoveries are outside of criteria limits for all three samples, it may be assumed that a matrix effect is involved and no corrective action is necessary. The instance will be documented in the Case Narrative.

10.5.3 Relative Retention Times

If the technical acceptance criteria for the relative retention times of the internal standard compounds or surrogate compounds are not met, the following corrective actions are taken in the given order:

a. Carrier gas, zone temperatures and instrument temperature programs are checked to insure that an error was not made or that the gas tank was not dry or clogged. If no errors are found the analyst will proceed to the next step in the corrective action process.

b. The sample is re-analyzed to insure that an error was not made during the first injection. If, after reanalysis, the relative retention times are not within the technical acceptance criteria, it may be assumed that a matrix effect was involved. Both analyses will be reported and the instance will be documented in the Case Narrative. If, after re-analysis, the relative retention times are within the technical acceptance criteria, the second analysis will be reported only.

Exception: If the relative retention times of a sample, MS/MSD agree (i.e., relative retention times are outside of criteria limits for the sample, MS and MSD, it may be assumed that a matrix effect was involved and further corrective action is not necessary.

10.5.4 Matrix Spike Blanks.

If the Matrix Spike Blank was found to be unacceptable all samples in the associated batch must be re-extracted and re-analyzed. If the sample was not within extraction hold time, a job exception must be filed and both analysis must be included with the report.

10.6 Injection Logs: Injection Logs must contain the following information:

a. Date, time, and analyst initials
b. File number (FRN), sample ID, vial #, and work order #
c. Injection volume, final volume, initial volume and dilution factor
d. Indicate if tailing of degradation was present in the tune
e. References for the standard, tune mix, IS mix
f. Daily maintenance performed
g. Any non-conformances with the samples

11.0 Calculations / Data Reduction

11.1 Target Compounds

Target compounds identified shall be quantitated by the internal standard method. The internal standard used shall be the one assigned to that analyte for quantitation (see Table 4). The EICP area of primary characteristic ions of analytes listed in Tables 3 and 6 are used for quantitation.

In instances where manual quantitation is necessary due to co-elution baseline noise or matrix interferences, all instances will be initialed and dated by the analyst. The quantitation report is documented as such by an "m" next to the compound that has been edited. In all instances of manual integration, a hardcopy of the EICP for that compound will be supplied with the raw data, this applies to all target compounds, internal standards and surrogate compounds.

The average response factor (RRF) from the initial calibration analysis (linear model) is used to calculate the concentration in the sample. Secondary ion quantitation is allowed ONLY when there are sample interferences with the primary ion. If secondary ion quantitation is performed, the reason is then documented in the case Narrative. The area of a secondary ion cannot be used for the area of a primary ion unless a relative factor is calculated using the secondary ion.

11.2 Water Samples

The following Equation (Eq. 8) is used to determine the concentration of target compounds identified in water samples:

Equation 8

$$Concentration\ \mu g/L = \frac{(A_x)(I_s)(V_c)(Df)(GPC)}{(A_{is})(RRFi)(V_o)(V_i)}$$

Where,
A_x = Area of the characteristic ion for the compound to be measured
A_{is} = Area of the characteristic ion for the internal standard
I_s = Amount of internal standard injected in nanograms (ng)
V_o = Volume of water extracted in milliliters (mL)
V_i = Volume of extract injected in microliters (µL)
V_c = Volume of the concentrated extract in microliters (µL) (V_c = 1,000 µL if sample was not subjected to GPC; V_t = 500 µL if sample was subjected to GPC)
RRFi= Relative response factor determined from the initial calibration
GPC= GPC factor.
GPC= 1.0 if water sample was not subjected to GPC;
Df = Dilution factor. The dilution factor for analysis of water samples for semivolatiles by this method is defined as follows:

<u>µL most conc. extract used to make dilution + µL clean solvent</u>

µL most conc. extract used to make dilution

If no dilution is performed, Df = 1.0

11.3 Soil/Sediment Samples

The following Equation (Eq. 9) is used to determine the concentration of target compounds in soil/sediment samples:

Equation 9

$$Concentration\ \mu g/Kg\ (Dry\ weight\ basis) = \frac{(A_x)(I_s)(V_c)(Df)(GPC)}{(A_{is})(RRF_i)(V_i)(W_s)(D)}$$

Where,
A_x, I_s, A_{is} are as given for water, above.
V_c = Volume of the concentrated extract in microliters (µL) (V_t = 500 µL)
V_i = Volume of the extract injected in microliters (µL)
D = $\frac{100 - \%\ moisture}{100}$
W_s = Weight of sample extracted in grams (g)
GPC= GPC factor (GPC = 2.0 to account for GCP cleanup)
RRFi= Relative response factor determined from the initial calibration.
Df = Dilution factor. The dilution factor for analysis of soil/sediment samples for semivolatile by this method is defined as follows:

$$\frac{\mu L\ most\ conc.\ Extract\ used\ to\ make\ dilution + \mu L\ clean\ solvent}{\mu L\ most\ conc.\ Extract\ used\ to\ make\ dilution}$$

If no dilution is performed, Df = 1.0.

The factor of 2.0 in the numerator is used to account for the amount of extract not recovered from the use of GPC cleanup. Concentrating the extract collected after GPC to 0.5mL maintains the sensitivity of the soil/sediment method.

11.4 Tentatively Identified Compounds

Non-Target Compounds
An estimated concentration for non-target compounds tentatively identified is quantitated by the internal standard method. For quantitation, the nearest internal standard free of interferences is to be used. The equations for calculating concentrations are the same as equations 8 and 9. Total area counts (or peak heights) from the total ion chromatograms are used for both the compounds to be measured and the internal standard. A relative response factor (RRF) of one (1) is assumed. The resulting concentration is to be qualified as “J” (estimated, due to lack of a compound specific response factor), and “N” (Presumptive evidence of presence), indicating the quantitative and qualitative uncertainties is calculated for all tentatively identified compounds as well as those identified as unknowns.

11.5 Rounding is performed automatically in the LIMs system

11.6 Organic Significant Figures

For volatile and semivolatile results, report analytical results to one significant figure if the value is less than 10, and two significant figures if the value is above 10.

12.0 Method Performance

12.1 Data assessment and acceptance criteria for quality control measures::

12.1.1 When internal standards are out of range a re-injection is required unless the problem can be determined to be a result of excessive matrix interference.

12.1.2 When surrogates are out of range, a re-extraction is required unless excessive visible chromatographic matrix interference is present. In this case, the Project Manager should be consulted to decide how to proceed.

12.1.3 When a positive hit for an analyte is above the calibration range a dilution must be performed to bring the value within calibration range .

12.1.4 When there are low spike recoveries in the matrix spike blank the entire extraction batch needs to be re-extracted. If there are high spike recoveries the associated sample data needs to be examined to assess if it may be biased.

12.2 Corrective actions for out of control data require Project Manager, Laboratory Director and/or QA Officer Notification. This can be accomplished either verbally, written using a Job Exception Report or both.

12.3 Contingency measures for handling out of control or unacceptable data requires the Project Manager to notify the client for input.

12.4 Method Detection Limit Study (MDL)

The method detection limit (MDL) is the lowest concentration that can be detected for a given analytical method and sample matrix with 99% confidence that the analyte is present. The MDL is determined according to the laboratory's MDL procedure in Section xx of the QA Manual. MDLs reflect a calculated (statistical) value determined under ideal laboratory conditions in a clean matrix, and may not be achievable in all environmental matrices. The laboratory maintains MDL studies for analyses performed; these are verified at least annually unless method requirements require a greater frequency.

12.5 Demonstration of Capabilities

Initial Demonstration of Capability (IDOC): The initial demonstration with each sample preparation and determinative method combination utilized must be performed by generating data of acceptable accuracy and precision for target analytes in a clean matrix. This is also done for new staff or when significant changes in instrumentation are made as stated in section 8.0 of Method 8000.

13.0 Pollution Control

It is TestAmerica's policy to evaluate each method and look for opportunities to minimize waste generated (i.e., examine recycling options, ordering chemicals based on quantity needed, preparation of reagents based on anticipated usage and reagent stability). Employees must abide by the policies in Section 13 of the Corporate Environmental Health and Safety Manual (CW-E-M-001) for "Waste Management and Pollution Prevention."

14.0 Waste Management

Waste management practices are conducted consistent with all applicable rules and regulations. Excess reagents, samples and method process wastes are disposed of in an accepted manner. Waste description rules and land disposal restrictions are followed. Waste disposal procedures are incorporated by reference to section 13 of the Corporate Safety Manual. The following waste streams are produced when this method is carried out.

There are two types of aqueous waste generated in the lab:

1. A-Waste: All non-nitric acid and alkaline aqueous waste.
2. AN-Waste: All aqueous waste containing nitric acid.

These types of waste are to be disposed of into appropriately market plastic containers.

The following are the other types of lab waste and where to dispose of:

1. C-Waste: all solvent waste gets dumped into appropriately marked metal cans. These cans need to be grounded whenever they are emptied to reduce explosion hazards. Discarded standards will also be dumped into C-waste cans.

2. Solid Waste: all contaminated paper, solid sample waste, sodium sulfate and all other non-glass material that has been contaminated is to be wrapped in foil and gathered to be dumped into 55 gallon drums.

3. Glass: contaminated glass needs to be rinsed off with methylene chloride and disposed of with all other glass in glass specific containers with special extra thick polypropylene liners. These containers are for glass only.

4. Extract Vials: extract vials are to be archived after they have been shot. After archival period, vials are to be crushed into a 55 gallon drum.

15.0 References / Cross-References

15.1 USEPA Methods for Evaluating Solid Waste; SW-846, Third Edition, Update III, Method 8270C, 12/96.

16.0 Method Modifications N/A

17.0 Attachments

17.1 Table 1: Semi-volatile Target Compound List and Contract Estimated Quantitation limits
17.2 Table 2: Ion Abundance
17.3 Table 3: Internal Standards and Corresponding Target Compounds Assigned for Quantitation
17.4 Table 4: Relative Response Factor Criteria for ICV and CCV
17.5 Table 5: Characteristic Ions for Target Compounds and Surrogates
17.6 Attachment A: SOP Procedure Summary
17.7 Attachment C: Job Summary Checklist
17.8 Attachment D: Log Book Copy

18.0 Revision History

Revision 1, dated June 10, 2009

- Removal of grand mean reference
- Integration for TestAmerica and STL operation
- Change to QA Manager, signature updated
- Change to Department Manager, signature updated

Revision 2, dated January 29, 2010

- Removed AFCEE attachment
- Removed ACOE attachment
- Added log book copy attachments, referenced in section 10.2.1
- Addition of 69 ion criteria to table
- Section 11.2 and 11.3 updated to state that the relative response factor is taken from the initial calibration
- Equations in section 11.2 and 11.3 were updated to reflect correct subscript for (RRF) to (RRFi) and for (Vc) to (Vt) and for (I3) to (Is)
- Updated attachment 1 to include Element and deleted AIMS reference
- Added APIX ,TCL list and priority pollutant %D statement in section 9.5.7

TABLE 1

Semivolatiles Target Compound List and Contract Estimated Quantitation Limits

			Estimated Quantitation Limits	
	Semivolatiles	CAS Number	Water µg/L	Low Soil µg/Kg
34.	Phenol	108-95-2	5	170
35.	bis-(2-Chloroethyl)ether	111-44-4	5	170
36.	2-Chlorophenol	95-57-8	5	170
37.	1,3-Dichlorobenzene	541-73-1	5	170
38.	1,4-Dichlorobenzene	106-46-7	5	170
39.	1,2-Dichlorobenzene	95-50-1	5	170
40.	2-Methylphenol	95-48-7	5	170
41.	Bis(2-chloroisopropl)ether	108-60-1	5	170
42.	4-Methylphenol	106-44-5	5	170
43.	N-Nitroso-di-n-propylamine	621-64-7	5	170
44.	Hexachloroethane	67-72-1	5	170
45.	Nitrobenzene	98-95-3	5	170
46.	Isophorone	78-59-1	5	170
47.	2-Nitrophenol	88-75-5	5	170
48.	2,4-Dimethylphenol	105-67-9	5	170
49.	bis(2-Chloroethoxy) methane	111-91-1	5	170
50.	2,4-Dichlorophenol	120-83-2	5	170
51.	1,2,4-Trichlorobenzene	120-82-1	5	170
52.	Naphthalene	91-20-3	5	170
53.	4-Chloroaniline	106-47-8	5	170
54.	Hexachlorobutadiene	87-68-3	5	170
55.	4-Chloro-3-methylphenol	59-50-7	5	170
56.	2-Methylnaphthalene	91-57-6	5	170
57.	Hexachlorocyclopenta-diene	77-47-4	5	170
58.	2,4,6-Trichlorophenol	88-06-2	5	170
59.	2,4,5-Trichlorophenol	95-95-4	10	330
60.	2-Chloronaphthalene	91-58-7	5	170
61.	2-Nitroaniline	88-74-4	10	330
62.	dimethylphthalate	131-11-3	5	170
63.	Acenaphthylene	208-96-8	5	170
64.	2,6-Dinitrotoluene	606-20-2	5	170
65.	3-Nitroanline	99-09-2	10	330
66.	Acenaphthene	83-32-9	5	170
67.	2,4-Dinitrophenol	51-28-5	10	330
68.	4-Nitrophenol	100-02-7	10	330

			Estimated Quantitation Limits	
	Semivolatiles	CAS Number	Water µg/L	Low Soil µg/Kg
69.	Dibenzofuran	132-64-9	5	170
70.	2,4-Dinitrotoluene	121-14-2	5	170
71.	Diethlphthalate	84-66-22	5	170
72.	4-Chlorophenyl-phenyl ether	7005-72-3	5	170
73.	Fluorene	86-73-7	5	170
74.	4-Nitroaniline	100-01-6	10	330
75.	4,6-Dinitro-2-methylphenol	534-52-1	10	330
76.	N-Nitroso-diphenylamine	86-30-6	5	170
77.	4-Bromophenyl-phenylether	101-55-3	5	170
78.	Hexachlorobenzene	118-74-1	5	170
79.	Pentachlorophenol	87-86-5	10	330
80.	Phenanthrene	85-01-8	5	170
81.	Anthracene	120-12-7	5	170
82.	Benzyl Alcohol	100-51-6	5	170
83.	Di-n-butylphthalate	84-74-2	5	170
84.	Fluoranthene	206-44-0	5	170
85.	Pyrene	129-00-0	5	170
86.	Butylbenzylphthalate	85-68-7	5	170
87.	3,3-Dichlorobenzidine	91-94-1	5	170
88.	Benzo(a)anthracene	56-55-3	5	170
89.	Chrysene	218-01-9	5	170
90.	bis(2-Ethylhexyl)phthalate	117-81-7	5	170
91.	Di-n-octylphthalate	117-84-0	5	170
92.	Benzo(b)fluoranthene	205-99-2	5	170
93.	Benzo(k)fluoranthene	207-08-9	5	170
94.	Benzo(a)pyrene	50-32-8	5	170
95.	Indeno(1,2,3-cd)-pyrene	193-39-5	5	170
96.	Dibenzo(a,h)-anthracene	53-70-3	5	170
97.	Benzo(g,h,i)perylene	191-24-2	5	170
98.	Benzoic Acid	65-85-0	150	4800
99.	Benzaldehyde	100-52-7	5	170
100.	Acetophenone	98-86-2	5	170
101.	Caprolactam	105-60-2	5	170
102.	1,1’-Biphenyl	92-52-4	5	170
103.	1,2,4,5-Tetrachlorobenzene	95-94-3	5	170
104.	2,3,4,6-Tetrachlorophenol	58-90-2	5	170
105.	Atrazine	1912-24-9	5	170

TABLE 2

DFTPP Key Ions and Ion Abundance Criteria

Mass	Ion Abundance Criteria
51	30.0 - 60.0 percent of mass 198
68	Less than 2.0 percent of mass 69
69	0-100 percent of the mass 198
70	Less than 2.0 percent of mass 69
127	40.0 - 60.0 percent of mass 198
197	Less than 1.0 percent of mass 198
198	Base peak, 100 percent relative abundance (see Note)
199	5.0-9.0 percent of mass 198
275	10.0-30.0 percent of mass 198
365	Greater than 1.0% of than mass 198
441	Present but less than mass 443
442	40.0 – 110.0 percent of mass 198
443	17.0 - 23.0 percent of mass 442

Note: All ion abundances MUST be normalized to m/z 198, the nominal base peak, even though the ion abundance of m/z 442 may be greater to 110 percent that of m/z 198.

TABLE 3
Semivolatile Internal Standards with Corresponding Target Compounds and Surrogates Assigned for Quantitation

1,4-Dichlorobenzene-d$_4$	Naphthalene-d$_8$	Acenaphthene-d-$_{10}$	Phenanthrene-d	Chrysene-d$_{12}$	Perylene-d$_{12}$
Phenol	Nitrobenzene	Hexachlorocyclopentadiene	4,6-Dinitro-2-methylphenol	Pyrene	Benzo(b)fluoranthene
bis(2-Chloroethyl)ether	Isophorone	2,4,6-Trichlorophenol	N-nitroso-di-phenylamine	Butylbenzylphthalate	Benzo(k)fluoranthene
2-Chlorophenol	2-Nitrophenol	2,4,5-Trichlorophenol	4-Bromophenylphenolether	3,3'-Dichlorobenzidine	Benzo(a)phyrne
1,3-Dichlorobenzene	2,4-Dimethylphenol	2-Chloroaphthalene	Hexachlorobenzene	Benzo(a)-anthracene	Indeno(1,2,3-cd)-pyrene
1,4-Dichlorobenzene	bis(2-Chloroethoxy) methane	2-Nitroaniline	Pentachlorophenol	bis(2-ethyl-hexyl)phthalate	Benzo(g,h,i)-perylene
1,2-Dichlorobenzene	2,4-Dichlorophenol	Dimethylphthalate	Carbzole	Chrysene	Dibenzo(a,h)-anthracene
2-Methylphenol	1,2,4-Trichlorobenzene	Acenaphthylene	Phenanthrene	Terphenyl-d$_{14}$ (surr)	
2,2'-oxybis-(1-Chloropropane)	Naphthalene	3-Nitroaniline	Anthracene	Di-n-octyl-phthalata	
4-Methylphenol	4-Chloroanaline	Acenaphthene	Di-n-butylphthalate		
N-Nitroso-Di-n-propylamine	Hexachlorobutadiene	2,4-Dinitorphenol	Fluoranthene		
Hexachloroethane	4-Chloro-3-methylphenol	4-Nitrophenol	Atrazine		
2-Fluorophenol(surr)	2-Methylnaphthalene	Dibenzofuran			
Phenol-d$_5$ (surr)	Nitrobenzene-d$_5$ (surr)	2,4-Dinitrotoluene			
4-methylphenol	Benzoic acid	2,6-Dinitrotoluene			
Aniline	4-chloroaniline	Diethylphthalate			
Benzyl Alcohol	N-Nitrosobutyl-amine	4-Chlorophenyl-phenylether			
Benzaldehyde	Caprolactam	Fluorene			
Acetophenone	1,2,4,5-Tetrachlorbenz.	4-Nitroaniline			
		2-Fluorobiphenyl (surr)			
		2,4,6-Tribromophenol (surr)			
		1,1’-Biphenyl			
		2,3,4,6-Tetrachlorophenol			

2-Fluorobi

TABLE 4

Relative Response Factor Criteria for Initial and Continuing
Calibration of Semivolatile Target Compounds and Surrogates

Semivolatile Compounds	Minimum RRF	Maximum % RSD	Maximum % Diff
Acenaphthene (CCC)	none	30	±20
1,4-Dichlorobenzene (CCC)	none	30	±20
Hexachlorobutadiene (CCC)	none	30	±20
N-Nitrosodiphenylamine (CCC)	none	30	±20
Di-n-octylphthalate (CCC)	none	30	±20
Flouranthene (CCC)	none	30	±20
Benzo(a)pyrene (CCC)	none	30	±20
4-Chloro-3-methylphenol (CCC)	none	30	$^{+}$20
2,4-Dichlorophenol (CCC)	none	30	±20
2-Nitrophenol (CCC)	none	30	±20
Phenol (CCC)	none	30	±20
Pentachlorophenol(CCC)	none	30	±20
2,4,6-Trichlorophenol (CCC)	none	30	±20
N-Nitroso-di-n-propylamine (SPCC)	0.050	None	none
Hexachlorocyclopentadiene (SPCC)	0.050	None	none
2,4-Dinitrophenol (SPCC)	0.050	None	none
4-Nitrophenol (SPCC)	0.050	None	none

Characteristic Ions for Semivolatile
Target Compounds and Surrogates

Parameters	Primary Quantitation Ion	Secondary Ion(s)
Phenol	94	65, 66
bis(2-Chloroethyl)ether	93	63, 95
2-Chlorophenol	128	64, 130
1,3-Dichlorobenzene	146	148, 113
1,4-Dichlorobenzene	146	148, 113
1,2-Dichlorobenzene	146	148, 113
2-Methylphenol	108	107
Bis(2-chloroisopropyl)ether	45	77, 79
4-Methylphenol	108	107
N-Nitroso-di-n-propylamine	70	42, 101, 130
Hexachloroethane	117	201, 199
Nitrobenzene	77	123, 65
Isophorone	82	95, 138
2-Nitrophenol	139	65, 109
2,4-Dimethylphenol	107	121, 122
bis(2-Chloroethoxy)methane	93	95, 123
2,4-Dichlorophenol	162	164, 98
1,2,4-Trichlorobenzene	180	182, 145
Naphthalene	128	129, 127
4-Chloroaniline	127	129
Hexachlorobutadiene	225	223, 227
4-Chloro-3-methylphenol	107	144, 142
2-Methylnaphthalene	142	141
Hexachlorocyclopentadiene	237	235, 272
2,4,6-Trichlorophenol	196	198, 200
2,4,5-Trichlorophenol	196	198, 200
2-Chloronaphthalene	162	164, 127

Parameters	Primary Quantitation Ion	Secondary Ion(s)
2-Nitroaniline	65	92, 138
Dimethylphthalate	163	194, 164
Acenaphthylene	152	151, 153
3-Nitroaniline	138	108, 92
Acenaphthene	153	152, 154
2,4-Dinitrophenol	184	63, 154
4-Nitrophenol	109	139, 65
Dibenzofuran	168	139
2,4-Dinitrotoluene	165	63, 182
2,6-Dinitrotoluene	165	89, 121
Diethylphthalate	149	177, 150
4-Chlorophenyl-phenylether	204	206, 141
Fluorene	166	165, 167
4-Nitroaniline	138	92, 108
4,6-Dinitro-2-methylphenol	198	182, 77
N-Nitrosodiphenylamine	169	168, 167
4-Bromophenyl-phenylether	248	250, 141
Hexachlorobenzene	284	142, 249
Pentachlorophenol	266	264, 268
Phenanthrene	178	179, 176
Anthracene	178	179, 176
Benzyl Alcohol	108	79, 77
Di-n-butylphthalate	149	150, 104
Fluoranthene	202	101, 100
Pyrene	202	101, 100
Butylbenzylphthalate	149	91, 206
3,3'-Dichlorobenzidine	252	254, 126
Benzo(a)anthracene	228	229, 226
bis(2-Ethylhexyl)phthalate	149	167, 279
Chrysene	228	226, 229
Di-n-octylphthalate	149	---
Benzo(b)fluoranthene	252	253, 125

Parameters	Primary Quantitation Ion	Secondary Ion(s)
Benzo(k)fluoranthene	252	253, 125
Benzo(a)pyrene	252	253, 125
Indeno(1,2,3-cd)pyrene	276	138, 227
Dibenzo(a,h)anthracene	278	139, 279
Benzo(g,h,i)perylene	276	138,277
Benzoic Acid	122	105, 77
SURROGATES		
Phenol-d5	99	42, 71
2-Fluorophenol	112	64
2,4,6-Tribormophenol	330	332, 141
Nitrobenzene-d5	82	128, 54
2-Fluorobiphenyl	172	171
Terphenyl-d14	244	122, 212

Table 6: Characteristic Ions for Semivolatile Target Compounds and Surrogates

Parameters	Primary Quantitation Ion	Secondary Ion(s)
Phenol	94	65, 66
bis(2-Chloroethyl)ether	93	63, 95
2-Chlorophenol	128	64, 130
1,3-Dichlorobenzene	146	148, 113
1,4-Dichlorobenzene	146	148, 113
1,2-Dichlorobenzene	146	148, 113
2-Methylphenol	108	107
Bis(2-chloroisopropyl)ether	45	77, 79
4-Methylphenol	108	107
N-Nitroso-di-n-propylamine	70	42, 101, 130
Hexachloroethane	117	201, 199
Nitrobenzene	77	123, 65
Isophorone	82	95, 138
2-Nitrophenol	139	65, 109
2,4-Dimethylphenol	107	121, 122
bis(2-Chloroethoxy)methane	93	95, 123
2,4-Dichlorophenol	162	164, 98
1,2,4-Trichlorobenzene	180	182, 145
Naphthalene	128	129, 127
4-Chloroaniline	127	129
Hexachlorobutadiene	225	223, 227
4-Chloro-3-methylphenol	107	144, 142
2-Methylnaphthalene	142	141
Hexachlorocyclopentadiene	237	235, 272
2,4,6-Trichlorophenol	196	198, 200
2,4,5-Trichlorophenol	196	198, 200
2-Chloronaphthalene	162	164, 127
2-Nitroaniline	65	92, 138
Dimethylphthalate	163	194, 164
Acenaphthylene	152	151, 153

Parameters	Primary Quantitation Ion	Secondary Ion(s)
3-Nitroaniline	138	108, 92
Acenaphthene	153	152, 154
2,4-Dinitrophenol	184	63, 154
4-Nitrophenol	109	139, 65
Dibenzofuran	168	139
2,4-Dinitrotoluene	165	63, 182
2,6-Dinitrotoluene	165	89, 121
Diethylphthalate	149	177, 150
4-Chlorophenyl-phenylether	204	206, 141
Fluorene	166	165, 167
4-Nitroaniline	138	92, 108
4,6-Dinitro-2-methylphenol	198	182, 77
N-Nitrosodiphenylamine	169	168, 167
4-Bromophenyl-phenylether	248	250, 141
Hexachlorobenzene	284	142, 249
Pentachlorophenol	266	264, 268
Phenanthrene	178	179, 176
Anthracene	178	179, 176
Benzyl Alcohol	108	79, 77
Di-n-butylphthalate	149	150, 104
Fluoranthene	202	101, 100
Pyrene	202	101, 100
Butylbenzylphthalate	149	91, 206
3,3'-Dichlorobenzidine	252	254, 126
Benzo(a)anthracene	228	229, 226
bis(2-Ethylhexyl)phthalate	149	167, 279
Chrysene	228	226, 229
Di-n-octylphthalate	149	---
Benzo(b)fluoranthene	252	253, 125
Benzo(k)fluoranthene	252	253, 125
Benzo(a)pyrene	252	253, 125
Indeno(1,2,3-cd)pyrene	276	138, 227

Parameters	Primary Quantitation Ion	Secondary Ion(s)
Dibenzo(a,h)anthracene	278	139, 279
Benzo(g,h,i)perylene	276	138,277
Benzoic Acid	122	105, 77
SURROGATES		
Phenol-d5	99	42, 71
2-Fluorophenol	112	64
2,4,6-Tribormophenol	330	332, 141
Nitrobenzene-d5	82	128, 54
2-Fluorobiphenyl	172	171
Terphenyl-d14	244	122, 212

ATTACHMENT A - SOP PROCEDURE SUMMARY

I. Preparing the instrument;
   1. Cut column, change liner and septa, inject conditioning solution
   2. Ramp GC oven temp. to 325°C and ramp GC inj. Port pressure to 80 psi to see if pressure holds.

II. Shoot DFTPP tune mix
   1. Shoot 1 ul of the dftpp tune mix
   2. Evaluate the DFTPP peak using the 3rd Edition or criteria
   3. Evaluate the tailing factors of pentachlorophenol and benzidine.
   4. Evaluate the degradation of 4,4'-DDT to 4,4'-DDD and 4,4'-DDE.

III. Shoot single or 5pt. calibration;
   1. Shoot 1 ul of the 50ng continuing standard (CCC)
   2. Evaluate the continuing; 4pts may be out but none over 40%d.
   3. If CCC does not pass criteria, then a 5pt. curve (ICC) must be shot.

IV. Load Samples;
   1. Load blanks and MSBs in the beginning and dark samples toward the end.
   2. Very thick samples may be diluted.
   3. All samples must be shot within 12 hours of the tune injection.

V. Analyze data;
   1. Quantitate all samples; need raw and enhanced spectra for positive and negative hits and 20 TICs .
   2. Shoot dilutions on any samples with positive hits over 160ng.
   3. Shoot reinjections (RI's) on any sample that has internal standards out, unless there is severe matrix interference that accounts for the low recovery.
   4. Samples with more than one BN or AP surrogate out needs to be re-extracted (RE).

VI. Element Entry;
   1. Enter tunes, ICC's and CCC's.
   2. Enter all samples to be included with the job.
   3. Identify and enter all TIC's
   4. Calculate, close and run data validator.

VII. Review Data;
   1. Correct or explain any errors on the data validator.
   2. Make copies of logbooks, tunes, curves and standards and include them with the report.
   3. Check that all calculations have been made correctly.
   4. Turn in job for validation.

Attachment C: Job Summary Checklist

Job Number: ______________________ Instrument # ____________________
Method: _____________________

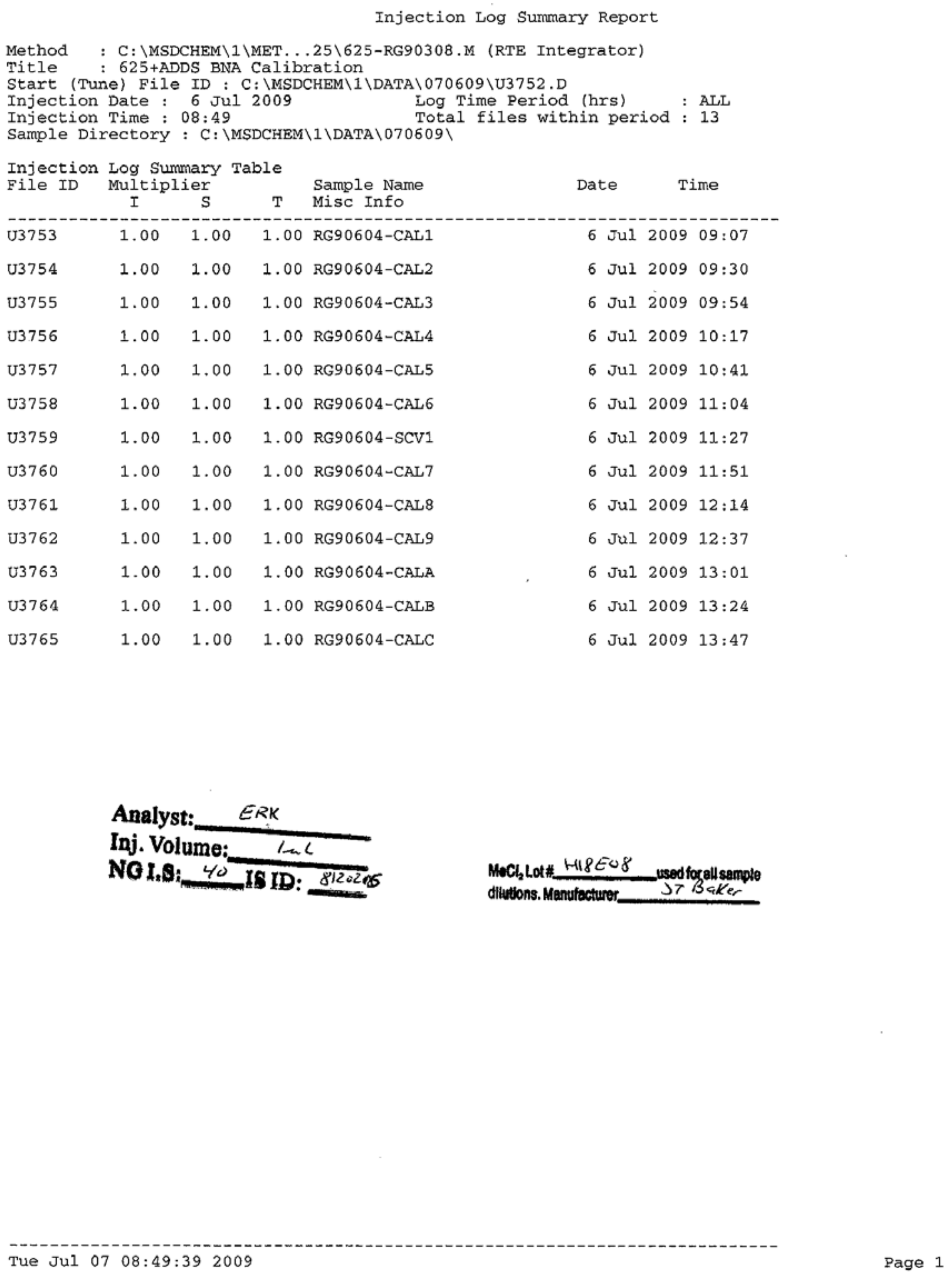
Yes/No	Primary Review
	Tunes passed?
	ICC's passed?
	CCV's passed?
	Quantitations have been performed correctly?
	Qualitative identifications are accurate?
	Client specific requirements have been followed?
	Method and process SOP's have been followed?
	Method and/or QUAPP specific QC criteria have been met?
	QC samples are within established limits?
	Dilution factors are correctly recorded and applied?
	Non-conformances and/or anomalous data have been properly documented and communicated?
	Job folder complete?

Check	Secondary Review
	Qualitative identification.
	Quantitative accuracy.
	Calibration.
	QC samples.
	Method and/or QUAPP specific QC criteria.
	Adherence to method and process SOP's.

Comments: ____
____

Analyst: ____________________ Date: _____________
Element Entry: ________________ Date: _____________
Review: ____________________ Date: _____________

Attachment D: Log Book Copy



```
                                    Injection Log Summary Report

Method    : C:\MSDCHEM\1\MET...25\625-RG90308.M (RTE Integrator)
Title     : 625+ADDS BNA Calibration
Start (Tune) File ID : C:\MSDCHEM\1\DATA\070609\U3752.D
Injection Date :  6 Jul 2009             Log Time Period (hrs)      : ALL
Injection Time : 08:49                   Total files within period : 13
Sample Directory : C:\MSDCHEM\1\DATA\070609\

Injection Log Summary Table
File ID   Multiplier          Sample Name                Date       Time
            I      S      T   Misc Info
---------------------------------------------------------------------------
U3753       1.00   1.00   1.00 RG90604-CAL1              6 Jul 2009 09:07

U3754       1.00   1.00   1.00 RG90604-CAL2              6 Jul 2009 09:30

U3755       1.00   1.00   1.00 RG90604-CAL3              6 Jul 2009 09:54

U3756       1.00   1.00   1.00 RG90604-CAL4              6 Jul 2009 10:17

U3757       1.00   1.00   1.00 RG90604-CAL5              6 Jul 2009 10:41

U3758       1.00   1.00   1.00 RG90604-CAL6              6 Jul 2009 11:04

U3759       1.00   1.00   1.00 RG90604-SCV1              6 Jul 2009 11:27

U3760       1.00   1.00   1.00 RG90604-CAL7              6 Jul 2009 11:51

U3761       1.00   1.00   1.00 RG90604-CAL8              6 Jul 2009 12:14

U3762       1.00   1.00   1.00 RG90604-CAL9              6 Jul 2009 12:37

U3763       1.00   1.00   1.00 RG90604-CALA              6 Jul 2009 13:01

U3764       1.00   1.00   1.00 RG90604-CALB              6 Jul 2009 13:24

U3765       1.00   1.00   1.00 RG90604-CALC              6 Jul 2009 13:47
```

Analyst: ERK
Inj. Volume: 1uL
NG I.S: 40 **IS ID:** 8120205

MeCl$_2$ Lot # H18E08 used for all sample dilutions. Manufacturer JT Baker

```
---------------------------------------------------------------------------
Tue Jul 07 08:49:39 2009                                             Page 1
```

TestAmerica
THE LEADER IN ENVIRONMENTAL TESTING

TestAmerica Buffalo

SOP No. BF-MV-005, Rev. 2
Effective Date: 03/30/2010
Page No.: 1 of 58
221T

Title: Analytical Methods for the Analysis of GC/MS Volatiles [SW-846 Method 8260B]

Once printed, this is considered an uncontrolled document

Approvals (Signature/Date):

John A. Sch
03/30/10
John Schove — Date
Department Manager

Kenneth E. Kasperek
03/30/10
Kenneth Kasperek — Date
Technical Director / Health & Safety Manager

Peggy Gray-Erdmann
03/30/10
Peggy Gray-Erdmann — Date
Quality Assurance Manager

03/30/10
Christopher Spencer — Date
Laboratory Director

Copyright Information:

This documentation has been prepared by TestAmerica Laboratories, Inc. and its affiliates ("TestAmerica"), solely for their own use and the use of their customers in evaluating their qualifications and capabilities in connection with a particular project. The user of this document agrees by its acceptance to return it to TestAmerica upon request and not to reproduce, copy, lend, or otherwise disclose its contents, directly or indirectly, and not to use it for any other purpose other than that for which it was specifically provided. The user also agrees that where consultants or other outside parties are involved in the evaluation process, access to these documents shall not be given to said parties unless those parties also specifically agree to these conditions.

THIS DOCUMENT CONTAINS VALUABLE CONFIDENTIAL AND PROPRIETARY INFORMATION. DISCLOSURE, USE OR REPRODUCTION OF THESE MATERIALS WITHOUT THE WRITTEN AUTHORIZATION OF TESTAMERICA IS STRICTLY PROHIBITED. THIS UNPUBLISHED WORK BY TESTAMERICA IS PROTECTED BY STATE AND FEDERAL LAW OF THE UNITED STATES. IF PUBLICATION OF THIS WORK SHOULD OCCUR THE FOLLOWING NOTICE SHALL APPLY:

Facility Distribution No. ____________

Distributed To:__________________________

1.0 Scope and Application

1.1 Analytes, Matrix(s), and Reporting Limits

1.1.1 Methods 8260B -5 mL aqueous purge, 8260B - 25mL aqueous purge, 8260B – 5gr soil and 8260B – medium level soil.

1.1.2 Applicable matrices include all aqueous samples, sediment, and soil.

1.1.3 The standard reporting limit (RL) is established at or above the low-level standard in the calibration curve. For a 5-ml purge volume, the RL for the majority of compounds is 1 ug/l.

On occasion clients may request modifications to this SOP. These modifications are handled following the procedures outlined in the Quality Assurance Manual.

2.0 Summary of Method

2.1 This analytical method is utilized for the analysis of water, sediment and soil from hazardous waste sites for the organic compounds listed in Table 1.

2.2 The method includes sample preparation and analyses by purge and trap gas chromatograph/mass spectrometer (GC/MS). Method can be used for 5mL purge or 25mL purge (concentrations adjusted accordingly).

2.3 Volatile compounds are extracted from sample matrix by the purge and trap method. Analytes are desorbed onto a capillary column. An appropriate ramping temperature program is applied to maximize separation and achieve the correct resolution between the analytes. A mass spectrometer detector (MSD) interfaced to the gas chromatograph (GC) is utilized to detect analytes of interest.

2.4 Analytes eluted from the capillary column are introduced into the mass spectrometer via a direct connection. Identification of target analytes is accomplished by comparing their mass spectra with the electron impact spectra of authentic standards. Quantitation is accomplished by comparing the response of a major (quantitation) ion relative to an internal standard using a minimum of a five-point calibration curve.

3.0 Definitions

3.1 **VBLK - Volatile blank**: VBLK's are made from laboratory produced volatile free water. They are analyzed before samples to ensure a clean laboratory environment and analytical system.

3.2 **IBLK – Instrument Blank:** IBLK's are made from laboratory produced volatile free water. They are analyzed after high level samples to verify that the system is clean and demonstrate the absence of carryover.

3.3 **LCS – Laboratory Control Sample:** An LCS consists of a sample of volatile free water that is spiked with a group of target compounds representative of the method analytes. It is used to monitor the accuracy of the analytical process, independent of matrix effects.

3.4 **Surrogates (System Monitoring Compounds):** Surrogates are organic compounds which are similar to the target analytes in chemical composition and behavior in the analytical process, but which are not normally found in environmental samples. Each sample, VBLK, LCS and MS/MSD are spiked with surrogates.

3.5 **MS/MSD – Matrix Spike/Matrix Spike Duplicate:** A Matrix Spike is an environmental sample which is spiked with a group of target compounds representative of the method analytes. A Matrix Spike Duplicate is a second aliquot of the same sample, which is spiked with the same target compounds. These samples are used to evaluate accuracy and precision in environmental samples.

3.6 **Batch:** A batch is a set of 20 samples using the same procedures within the same time period. Using this method each BFB analysis will start a new batch. Batches for medium level soils are defined at the sample preparation stage and may be analyzed on multiple instruments over multiple days, although reasonable effort must be made to keep the samples together.

4.0 Interferences

4.1 Airborne contamination may result from solvent vapors. VBLKs and IBLKs will be utilized to demonstrate a clean system and laboratory environment.

4.2 Some volatile compounds can permeate through a sample septum seal during storage or shipment. A weekly volatile holding blank is stored in all sample incubators to monitor contamination.

4.3 Contamination by carryover can occur whenever a sample with high concentrations of target compounds precedes a sample with low levels. The purging device, syringe and lines are flushed between every analysis to reduce carry over contamination. The trap is baked between each analysis.

5.0 Safety

Employees must abide by the policies and procedures in the Corporate Environmental Health and Safety Manual (CW-E-M-001) and this document. This procedure may involve hazardous material, operations and equipment. This SOP does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of the method to follow appropriate safety, waste disposal and health practices under the assumption that all samples and reagents are potentially hazardous. Safety glasses, gloves, lab coats and closed-toe, nonabsorbent shoes are a minimum.

5.1 Specific Safety Concerns or Requirements

5.1.1 The gas chromatograph and mass spectrometer contain zones that have elevated temperatures. The analyst needs to be aware of the locations of those zones, and must cool them to room temperature prior to working on them.

5.1.2 The mass spectrometer is under deep vacuum. The mass spectrometer must be brought to atmospheric pressure prior to working on the source.

5.1.3 There are areas of high voltage in both the gas chromatograph and the mass spectrometer. Depending on the type of work involved, either turn the power to the instrument off, or disconnect it from its source of power.

5.2 Primary Materials Used

The following is a list of the materials used in this method, which have a serious or significant hazard rating. **Note: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the MSDS for each of the materials listed in the table.** A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the MSDS for each material before using it for the first time or when there are major changes to the MSDS.

Material	Hazards	Exposure Limit (1)	Signs and symptoms of exposure
Methanol	Flammable Poison Irritant	200 ppm-TWA	A slight irritant to the mucous membranes. Toxic effects exerted upon nervous system, particularly the optic nerve. Symptoms of overexposure may include headache, drowsiness and dizziness. Methyl alcohol is a defatting agent and may cause skin to become dry and cracked. Skin absorption can occur; symptoms may parallel inhalation exposure. Irritant to the eyes.
1 – Exposure limit refers to the OSHA regulatory exposure limit.			

6.0 Equipment and Supplies

6.1 Instrumentation

6.1.1 Purge and trap devices

- Varian Archon Auto sampler
- Encon Concentrator
- O/I Analytical Auto sampler and Concentrator
- Centurion Auto sampler

6.1.2 Trap Packing

- Vocarb 3000
    - Carbpack B
    - Carboxen 1000
    - Carboxen 1001
- OI #10
    - Tenax
    - Silica Gel
    - cms
- Other traps may be used if the Quality Control criteria are met.

6.1.3 Gas Chromatograph/Mass Spectrometer (GC/MS) - GC: HP5890, MS:

- Gas chromatograph Column - J&W Scientific DB-624 or Phenomenex ZB-624
- Internal diameter: 0.25mm or 0.18mm
- Length: 20m, 30m or 60m.
- Coating: Cyanopropylphenyl Methyl Silicone
- Film thickness: 1.0um or 3.0μm

6.1.4 Data System

- Computer with Chemstation enviroquant software
- Gas Chromatograph/Mass Spectrometer (GC/MS)-GC: HP6890 or HP7890, MS: Hewlett-Packard/Agilent 5973N or 5975.
- ProLab Resources software

6.1.5 Analytical Balance Mettler - Toledo Inc. Mettler AE160

6.2 Supplies

- Syringes - Hamilton Syringes size, 10ul, 25ul, 50ul, 100ul, 500ul, 1ml, 5ml, 10ml, 25ml
- Pasteur Pipettes – disposable
- Vials and caps - 2ml disposable
- Vials and caps - 40ml disposable
- Volumetric flasks - Pyrex 2ml, Pyrex 10ml, Pyrex 50ml, Pyrex 100ml
- pH paper - wide range -.EM Science

7.0 Reagents and Standards

7.1 Reagent Water - For volatile analysis, the reagent water is volatile free and is prepared by passing water through a carbon trap.

7.2 Methanol - Burdick & Jackson, purge and trap grade

7.3 Stock Standards - Are purchased as certified standard mixtures. Traceability is documented following the procedures in the "Standards Traceability and Preparation Logbooks" SOP# AGP-STD-14. Individual compounds are prepared using reagent grade chemicals following the "Primary Standards Preparation" SOP# AMV-STD-25.

7.3.1 Stock Target Compound Mix – Is composed of three different mixtures.

7.3.1.1 Gas Mix (See Table 7 for component list) is purchased at a concentration of 2000ug/ml.

7.3.1.2 54 Component Mix (See Table 8 for component list) is purchased at a concentration of 2000ug/ml.

7.3.1.3 8260+ Mix (See Table 9 for component list) is purchased and is composed of four separate mixtures.

- 8260+ Mix #1 is purchased at a concentration of 1000ug/ml.
- 8260+ Mix #2 is purchased at a concentration of 5000ug/ml.

- 8260+ Mix #3 is purchased at a concentration of 20000ug/ml.
- 8260+ Mix #4 is purchased at a concentration of 5000ug/ml.

7.3.2 **Stock Calibration Verification Mix** – Is composed of two different mixtures.

7.3.2.1 The Second Source Mix (See Table 10 for component list) is purchased at a concentration of 2000ug/ml.

7.3.2.2 The 8260+ Second Source Mix (See Table 11 for component list) is purchased and is composed of two separate mixtures.

- 8260+ Second Source Mix #1 is purchased at a concentration of 1000ug/ml.
- 8260+ Second Source Mix #2 is purchased at a concentration of 5000ug/ml.

7.3.3 **Stock Internal Standard Solution** – A mixture of 1,4-Dichlorobenzene-d4, Chlorobenzene-d5 and 1,4-Difluorobenzene in Methanol is purchased at a concentration of 2500ug/ml.

7.3.4 **Stock System Monitoring Solution** – A mixture of Toluene-D8, 4-Bromofluorobenzene and 1,2-Dichloroethane-d4 in Methanol is purchased at a concentration of 2500ug/ml.

7.3.5 **Stock Matrix Spike Solution** – A 5 component mixture of 1,1-dichloroethene, trichloroethene, chlorobenzene, toluene, and benzene in Methanol is purchased at a concentration of 2500ug/ml.

7.3.6 **Stock BFB Solution** – A solution of 4-Bromofluorobenzene in Methanol is at a concentration of 25000ug/ml.

7.4 **Secondary IS and System Monitoring Calibration Dilution Standards** – these solutions are used for the manual injections required to prepare the initial calibration.

7.4.1 **Internal Standard Solution** - 80ul of stock standard IS solution (2500ug/ml) is added to approximately 1 ml of purge and trap grade methanol in a 2 ml Class A volumetric, and then brought up to final volume of 2 ml with additional purge and trap grade methanol for a final concentration of 100ng/ul.

7.4.2 **System Monitoring Compound Solution** - 80ul of stock standard Surrogate solution (2500ug/ml) is added to approximately 1 ml of purge and trap methanol in a 2 ml Class A volumetric, and then brought up a final volume of 2ml with additional purge and trap grade methanol for a final concentration of 100ng/ml.

7.4.3 To calculate appropriate expiration dates, refer to “Standards Traceability and Preparation Logbooks”.

7.5 **Working Standards**

7.5.1 **Intermediate Calibration Solution** (Three individual mixtures)

7.5.1.1 250ul of stock standard Gas Mix solution (2000ug/ml) is added to approximately 4 ml of purge and trap methanol in a 5ml Class A volumetric, and then brought up to a final volume of 5ml with additional purge and trap grade methanol for a final concentration of 100ng/ul.

7.5.1.2 500ul of stock standard 54 Component Mix solution (2000ug/ml) is added to approximately 9ml of purge and trap methanol in a 10ml Class A volumetric, and then brought up a final volume of 10ml with additional purge and trap grade methanol for a final concentration of 100ng/ul.

7.5.1.3 1000ul of each of the four stock standard 8260+ Mixes are added to approximately 5ml of purge and trap methanol in a 10ml Class A volumetric, and then brought up a final volume of 10ml with additional purge and trap grade methanol.

7.5.2 **Matrix Spike Solution** - 100ul of stock standard 5 component solution (2500ug/ml) is added to approximately 4 ml of purge and trap methanol in a 5 ml Class A volumetric, and then brought up a final volume of 5ml with additional purge and trap grade methanol for a final concentration of 50ng/ul.

7.5.3 **A Full List Matrix Spike Standard** is made from stock Calibration Verification Standards and is composed of two mixes.

7.5.3.1 250ul of stock standard Gas Mix solution (2000ug/ml) is added to approximately 4 ml of purge and trap methanol in a 5ml Class A volumetric, and then brought up a final volume of 5ml with additional purge and trap grade methanol for a final concentration of 100ng/ul.

7.5.3.2 200ul of each of the two stock standard 8260+ Second Source Mixes are added to approximately 1ml of purge and trap methanol in a 2ml Class A volumetric, and then brought up a final volume of 2ml with additional purge and trap grade methanol.

7.5.4 **Working Internal Standard and System Monitoring Compound Solutions** – for auto injection by instrument.

7.5.4.1 **Working Internal Standard Solution** - An Internal Standard Mixture is made from IS stock standard (2500ug/ml). For water analysis a concentration between 20 and 30ng/ul is prepared, depending on sample loop size of the auto sampler, to produce a final concentration of 25ug/L in the sample. For low level soil analysis a concentration between 45 and 55ng/ul is prepared, depending on sample loop size of the auto sampler, to produce a final concentration of 50ug/Kg in the sample.

7.5.4.2 **Working System Monitoring Calibration Solution** - A System Monitoring Compounds Mixture is made from Surrogate stock standard (2500ug/ml). For water analysis a concentration between 20 and 30ng/ul is prepared, depending on sample loop size of the auto sampler, to produce a final concentration of 25ug/L in the sample. For low level soil analysis a concentration between 45 and 55ng/ul is prepared, depending on sample loop size of the auto sampler, to produce a final concentration of 50ug/Kg in the sample.

7.5.5 **Tuning Mixture** - 4ul of stock solution 4-Bomofluorobenzene (BFB) tuning mixture is added to approximately 1 ml of purge and trap grade methanol in a 2 ml Class A volumetric, and then brought up to final volume of 2 ml with additional purge and trap grade methanol for a final concentration of 50ng/ul.

7.5.6 Working Initial Calibration Standards

7.5.6.1 Water: 25 mL

7.5.6.1.1 20ul, 10ul and 5ul each of Intermediate Calibration Solution (7.5.1) and 15ul, 5ul and 0ul of System Monitoring Compound Solution (7.4.2) is added to reagent water in each of three 50ml volumetric flasks. The flasks are brought to volume with reagent water to prepare the 40, 20 and 10 ug/L standards respectively.

7.5.6.1.2 4ul and 1ul each of Intermediate Calibration Solution (7.5.1) and System Monitoring Compound Solution (7.4.2) are added to reagent water in 100 ml volumetric flasks. The flasks are brought to volume with reagent water to prepare the 4ug/L and 1ug/L standards respectively.

7.5.6.1.3 Each standard is then transferred into a 40ml vial and loaded onto the auto sampler.

7.5.6.2 Water: 5 mL (5 point curve)

7.5.6.2.1 50ul, 25ul 12.5ul and 5ul each of Intermediate Calibration Solution (7.5.1) and 37.5ul, 12.5ul, 0ul and 5ul of System Monitoring Compound Solution (7.4.2) is added to reagent water in each of four 50ml volumetric flasks. The flasks are brought to volume with reagent water to prepare the 100, 50, 25 and 10 ug/L standards respectively.

7.5.6.2.2 1ul of each Intermediate Calibration Solution (7.5.1) and 1.0ul of System Monitoring Compound Solution (7.4.2) is added to reagent water in a 100ml volumetric flask. The flask is brought to volume with reagent water to prepare the 1ug/L standard.

7.5.6.2.3 The standard is then transferred into a 40ml vial and loaded onto the auto sampler.

7.5.6.3 Water: 5 mL (6 point curve)

7.5.6.3.1 50ul, 25ul 12.5ul and 5ul each of Intermediate Calibration Solution (7.5.1) and 37.5ul, 12.5ul, 0ul and 5ul of System Monitoring Compound Solution (7.4.2) is added to reagent water in each of four 50ml volumetric flasks. The flasks are brought to volume with reagent water to prepare the 100, 50, 25 and 10 ug/L standards respectively.

7.5.6.3.2 5ul and 1ul of each Intermediate Calibration Solution (7.5.1) and 5.0ul and 1.0ul of System Monitoring Compound Solution (7.4.2) is added to reagent water in each of two 100ml volumetric flasks. The flasks are brought to volume with reagent water to prepare the 5.0 and 1.0ug/L standards respectively.

7.5.6.3.3 Each standard is then transferred into a 40ml vial and loaded onto the auto sampler.

7.5.6.4 Soil: (5 point curve)

7.5.6.4.1 100ul, 50ul, 25ul, 10ul and 2.5ul each of Intermediate Calibration Solution (7.5.1) and 75ul, 25ul, 0ul, 10ul and 2.5ul of System Monitoring Compound Solution (7.4.2) is added to reagent water in each of five 50ml volumetric flasks. The flasks are brought to volume with reagent water to prepare the 200, 100, 50, 20 and 5 ug/kg, standards respectively.

7.5.6.4.2 5 ml of each standard is then transferred into five individual 40ml vials and loaded onto the auto sampler.

7.5.6.5 Soil: (6 point curve)

7.5.6.5.1 100ul, 50ul, 25ul, 10ul, 5ul and 2.5ul each of Intermediate Calibration Solution (7.5.1) and 75ul, 25ul, 0ul, 10ul, 5ul and 2.5ul of System Monitoring Compound Solution (7.4.2) is added to reagent water in each of six 50ml volumetric flasks. The flasks are brought to volume with reagent water to prepare the 200, 100, 50, 20, 10 and 5 ug/kg, standards respectively.

7.5.6.5.2 5 ml of each standard is then transferred into six individual 40ml vials and loaded onto the auto sampler.

7.5.7 Continuing Calibration Standard

7.5.7.1 Water: 25 ml

7.5.7.1.1 5ul of stock target compound mix is added to approximately 49mls of reagent water in a 50ml volumetric flask. The volumetric is brought to a final volume of 50ml to make a final concentration of 10ppb. Pour the standard into a 40ml vial. The auto sampler adds the internal standard and system monitoring compounds.

7.5.7.2 Water: 5 ml

7.5.7.2.1 12.5ul of stock target compound mix is added to approximately 49mls of reagent water in a 50ml volumetric flask. The volumetric is brought to a final volume of 50ml to make a final concentration of 25ppb. Pour the standard into a 40ml vial. The auto sampler adds the internal standard and system monitoring compounds.

7.5.7.3 Soil:

7.5.7.3.1 25ul of stock target compound is added to approximately 49mls of reagent water in a 50ml volumetric flask. The volumetric is brought to a final volume of 50ml to make a final concentration of 50ppb. Take 5ml and transfer it into a 40ml vial. The auto sampler adds the internal standard and system monitoring compounds.

7.6 Storage of Standards

7.6.1 Stock standards are stored in flame sealed ampoules at 22^0 C to -20^0 C according to the vendor's specifications.

7.6.2 Secondary dilution standards are stored in Teflon-sealed crimp cap vials at < 0^0 C.

7.6.3 Aqueous standards are stored in Teflon-sealed vials at 4^0 C $\pm$ 2^o C.

8.0 Sample Collection, Preservation, Shipment and Storage

8.1 Samples are collected in 40 mL vials with caps and septa, preserved to a pH < 2 with Hydrochloric Acid and stored at 4 ± 2 degrees C until time of analysis.

8.2 Holding time for unpreserved samples is 7 days from sample date. For preserved samples the holding time is 14 days from sample date.

8.3 For some clients, regulatory agencies or QAPPS, the specified holding times may be different than those described in 8.2. In those cases, consult the specific Protocol/Method/QAPP or Project Manager for holding time details.

8.4 Sample Storage

- Volatile samples are stored at 4 ± 2^0C from the time of collection until analysis.
- Volatile samples are stored together in refrigerators specifically designated for volatiles only.
- Storage blanks are stored with samples until analysis.
- Samples and extracts are stored separately.
- Volatile samples and standards are stored separately.

8.5 Preparation Of MS/MSD Samples

8.5.1 Water Samples**:** 40ml vial is spiked with 8ul of 50ng/ul or 4ul of 100ng/ul matrix spike standard for 25ml purge and 22ul of 50ng/ul or 11ul of 100ng/ul for the 5ml purge. This corresponds to a final concentration in the samples of 10ug/L and 25ug/L respectively. Analysis proceeds according to procedures described for water analysis.

8.5.2 Low Level Soil/Sediment Samples: 5ul of 50ng/ul or 2.5ul of 100ng/ul of matrix spiking solution is added to a 5g aliquot of sample. This corresponds to a final concentration in the samples of 50 ug/kg. Analysis proceeds according to procedures described for low-level soil/sediment samples.

8.5.3 Medium Level Soil/Sediment Samples: 1ml of methanol containing the soil spiking solution is combined with 50 mL of water. Sample analysis proceeds according to procedures described for medium level soil/sediment samples.

9.0 Quality Control

9.1 Blank Analysis

9.1.1 **Method Blank:** A method blank consisting of a clean reference matrix (reagent water or purified quartz sand) must be analyzed prior to the analysis of samples but following any standard analysis.

- Target compounds detected in a method blank must fall below the reporting limit, unless specified in client QAPP.
- If internal standard or systems monitoring compound recoveries are not met, the method blank must be reanalyzed before the analysis of samples.

9.1.2 **Storage (Holding) Blank:** A weekly holding blank is analyzed to determine if cross contamination occurs within the volatile holding area. The results are reviewed by the quality assurance department and deemed acceptable or not acceptable. Corrective action, if necessary, will be taken.

9.1.3 **Instrument Blank:** An instrument blank consisting of a clean reference matrix analyzed after the analysis of samples containing target compounds which exceed the calibration range. Multiple instrument blanks are shot until the instrument blank meets the criteria for method blanks.

9.2 **Matrix Spike Blank (MSB/LCS)** An aliquot of clean reference material spiked with the matrix spiking solution is analyzed with each analytical batch.

9.2.1 If a compliant Second Source Calibration Verification (SCV) has already been analyzed, then standards from the primary (CCV) source may be used. The solution is spiked at a concentration of 10ug/L for 25ml analysis, 25ug/L for 5ml analysis and 50ug/Kg for soil analysis.

9.2.2 Alternatively, a standard that is purchased from an alternate vender (or where not available from a second vendor an alternate lot will be used) from the continuing (CCV) standard may be used. The solution is spiked at a concentration of 10ug/L for 25ml analysis, 25ug/L for 5ml analysis and 50ug/Kg for soil analysis.

9.2.3 The MSB/LCS must fall within internally derived statistical control limits or where applicable the limits specified by a project QAPP.

9.2.4 Analytes that have been identified as a Poor Performing Compounds (Table 5) will be considered compliant as long as their percent recovery exceeds 10%.

9.2.5 Routine compounds included in the MSB/LCS are:

1,1-Dichloroethene; Chlorobenzene; Toluene; Benzene; Trichloroethene

9.2.6 When required, the MSB/LCS a 'full-compound' spike will be prepared and the MSB/LCS will be spiked with all compounds of interest. Due to the potentially large number of target compounds for method 8260B, it is possible that a few of the spiking compound could fall outside limits in the MSB/LCS. If a compound falls outside limits biased high and that compound is not found in the samples, a comment will be made in the case narrative and the data will be found to be acceptable.

9.2.7 If the results of sample matrix spikes fall outside of the quality control range due to matrix, the MSB is used to verify that the laboratory can perform a spike on a clean matrix.

9.3 **Matrix Spike And Matrix Spike Duplicate Analysis** A matrix spike and matrix spike duplicate consisting of an actual field sample which has been spiked with the matrix spiking solution.

9.3.1 Matrix spike and matrix spike duplicate analysis will not be performed on rinsates or field/trip blanks.

9.3.2 If a sample has not been designated for MS/MSD analysis by the client, a sample will be selected at the analyst's discretion. MS/MSD analysis will be performed at a minimum of every 20 samples.

9.3.3 If insufficient sample was received for a designated MS/MSD the client will be contacted with the laboratories in-house designated sample for MS/MSD analysis. If no MS/MSD is required, the instance will be documented in the SDG narrative.

9.3.4 If medium level analysis is required on the client designated sample, the laboratory analyst will choose a low level sample on which to perform the quality control analysis. Medium level QC will also be performed.

9.4 Data Assessment & Acceptance Criteria for QC Measures

9.4.1 Technical Acceptance Criteria For Initial Calibration

9.4.1.1 **SPCCs** (System performance check compounds) are compounds used to check compound instability degradation. The following average minimum average response factors must be met before the curve can be used.

Chloromethane	0.10
1,1-Dichloroethane	0.10
Bromoform	0.10
Chlorobenzene	0.30
1,1,2,2-Tetrachloroethane	0.30

9.4.1.2 **CCCs** (Calibration Check Compounds) evaluate the calibration based on the integrity of the system. The % RSD for the CCCs MUST be equal or less than 30%. The CCCs are:

Vinyl chloride
1,1-Dichloroethene
Chloroform
1,2-Dichloropropane
Toluene
Ethyl benzene

If the % RSD of any of the target analytes is 15% or less, the average response factor is assumed constant and the average response factor may be used for quantitation.

OR

If the % RSD of a target analyte is greater than 15%, linear regression or quadratic regression (forcing through the origin is advisable in both these models, to increase the accuracy at the low end of the calibration curve) may be used providing the coefficient of determination is greater than or equal to 0.99. If quadratic regression is used, a minimum of 6 calibration points must to be analyzed.

9.4.1.3 Non-standard analytes are sometimes requested for analysis by this method. For these analytes it is acceptable to analyze a single point standard at the reporting limit with each continuing calibration rather than a five point calibration. If the analyte is not detected in the associated samples a non-detect will be reported and no further action is required. If the analyte is detected in any of the samples, a five point calibration will be analyzed and the samples with a positive detection will be re-analyzed against this compliant curve.

9.4.1.4 **Second Source Calibration Verification** To verify the accuracy of the initial calibration, a standard is obtained from a source different from the Calibration Standards. This is also referred to as an SCV.

9.4.1.4.1 Following the analysis of an acceptable initial calibration curve, an aliquot of this independent standard is analyzed at the CCV level.

9.4.1.4.2 Recoveries of all compounds shall fall within ±30% of the expected values. However, the recoveries of up to 40% are allowable for up to four compounds.

9.4.2 Technical Acceptance Criteria For Continuing Calibration

9.4.2.1 **SPCCs** - A system performance check is made daily or during every 12 hour analytical shift. Each compound must meet its minimum response factor (see Initial Calibration Criteria).

9.4.2.2 **CCCs** - Used to check the validity of the initial calibration. The % Difference for each CCC shall be less than or equal to 20% from the initial calibration for the continuing calibration to be valid. All non-CCC target compounds must be less than 50% difference (or Drift) with allowance for up to six target analytes to be greater than 50%.

9.4.2.3 **Internal Standard Retention Time** – The retention times for all internal standards must be evaluated to make sure that they are no more than 30 seconds from that of the midpoint of the initial calibration. If the retention time shift is greater than 30 seconds, the system must be inspected for malfunctions and maintenance must be performed, as required.

9.4.2.4 **Internal Standard Response** – The EICP area for all internal standards must be evaluated to make sure that they have not change by a factor greater than two (-50% to +100%) from that of the midpoint of the initial calibration. If the response exceeds these limits, the system must be inspected for malfunctions and maintenance must be performed, as required.

9.4.3 Technical Acceptance Criteria of Quality Control Samples

Samples, blanks, matrix spikes, and matrix spike duplicates must meet internal standard and system monitoring compound recovery limits. Where the Internal Standard recovery limit equals sample internal standard characteristic ion area (EICP) divided by the CCV internal standard characteristic ion area (EICP), multiplied by 100.

9.5 Corrective Action for Out-of-Control Data

9.5.1 Corrective Actions For MS/MSD

9.5.1.1 If the recoveries of the internal standards and system monitoring compounds do not agree with the unspiked sample (i.e. the sample recoveries were within control limits and MS/MSD recoveries were outside of control limits) the MS/MSD will be evaluated. The analyst will use their technical judgment to determine if the non-conformance is due to sample matrix or laboratory error. If it is determined that the QC failure was due to laboratory error, then reanalysis will occur.

9.5.1.2 If the recoveries of the internal standards and system monitoring compounds agree with the unspiked sample (i.e. both the sample and MS/MSD recoveries were outside of control limits) re-analysis is not required. The instance will be documented in the SDG narrative.

9.5.1.3 The laboratory on an annual basis establishes limits for the matrix spiking compounds. If the concentrations determined in the MS/MSD do not meet the control limits, no corrective action is necessary as long as the MSB/LCS was within control limits. The instance will be documented in the job narrative.

9.5.2 Corrective Actions For Initial Calibration

9.5.2.1 If technical acceptance criteria cannot be met, it may be necessary to re-analyze the initial calibration. If after re-analysis, the criteria have not been met, it may be necessary to inspect the GC/MS system for possible problems.

9.5.2.2 Corrective actions may require one or several of the following procedures:

- Open new/remake standard mixes
- The ion source may be cleaned
- The column may be cut at the injection port end
- Change the purge trap on the purge and trap unit
- Correct purge gas flow to optimize response
- The column may be baked out

- The purge trap may be baked out
- The column may be replaced

9.5.3 Corrective Actions for Failure to Meet the Continuing Calibration Acceptance Criteria

9.5.3.1 If the technical acceptance criteria given above are not met, it may be necessary to re-analyze the continuing calibration check. If, after re-analysis, the given criterion has not been met, it may be necessary to re-analyze the initial calibration.

9.5.3.2 A single point standard at the reporting limit may be analyzed before the analysis of any samples. If the analyte is not detected in the associated samples a non-detect will be reported and a comment in the case narrative will be made. If the analyte is detected in any of the samples, a five point calibration will be analyzed and the samples with a positive detection will be re-analyzed against this compliant curve.

9.5.3.3 Other Corrective actions may be taken. The following details possible corrective actions:

- Open new/remake standard mixes
- The ion source may be cleaned
- The column may be cut at the injection port end
- The trap on the purge and trap unit may be replaced
- The purge gas flow may be adjusted
- The column may be baked out
- The trap may be baked out
- The column may be replaced

9.5.4 Corrective Actions For Samples

9.5.4.1 If the internal standard or system monitoring criteria are not met, the sample must be re-analyzed to insure that it was not an internal problem that affected recoveries. If, after re-analysis, recoveries are outside of control limits, a matrix effect can be assumed.

9.5.4.2 When dilutions are performed, target compound concentration must fall within the upper range of the initial calibration. If any target compound exceeds the calibration range, the sample would require dilution. The sample immediately following a sample with target compounds above the calibration range must be monitored to insure that there is no carryover present. If there is a possibility of carryover, that sample must be re-analyzed.

9.5.4.3 If matrix effects exist, and both analyses exhibit recoveries outside of control limits, both analyses will be reported and documented in the job narrative.

9.5.4.4 If, after re-analysis, recovery criteria are met, only the second analyses will be reported. If the second analyses occur outside of the contract required holding time, both analyses will be reported in that instance.

9.5.4.5 In the case of a matrix spike or matrix spike duplicate, these samples should only be reanalyzed if an error was identified in preparation or analysis of the sample. Failures will be documented in the SDG narrative.

9.5.5 Corrective Actions for Failure to Meet the Laboratory Control Sample (Matrix Spike Blank) Acceptance Criteria

9.5.5.1 The laboratory on an annual basis establishes limits for the matrix spiking compounds. The LCS must fall within these control limits. When required, the LCS will be spiked with all compounds of interest, otherwise spiked to include a minimum of 1,1-dichloroethene, trichloroethene, chlorobenzene, toluene, and benzene. Due to the potentially large number of target compounds for method 8260B, it is possible that a few of the spiking compounds could fall outside limits in the MSB/LCS. If a compound falls outside limits biased high and that compound is not found in the samples, a comment will be made in the case narrative and the data will be found to be acceptable

9.5.5.2 If the technical acceptance criteria are not met, it may be necessary to re-analyze the matrix spike blank. If, after re-analysis, the given criterion has not been met, it may be necessary to re-analyze the initial calibration.

9.5.5.3 Other Corrective actions may be taken. The following details possible corrective actions:

- Open new/remake standard mixes
- The ion source may be cleaned
- The column may be cut at the injection port end
- The trap on the purge and trap unit may be replaced
- The purge gas flow may be adjusted
- The column may be baked out
- The trap may be baked out
- The column may be replaced

9.5.6 Corrective Actions for Failure to Meet the Method Blank (VBLK) Acceptance Criteria

9.5.6.1 If the technical acceptance criteria are not met, it may be necessary to re-analyze the associated samples.

9.5.6.2 If the analyte is a common laboratory contaminant (Methylene Chloride, Acetone, 2-Butanone) the data may be reported with qualifiers if the concentration of the analyte is less than five times the reporting limit.

9.5.6.3 If the target analyte is not greater than the reporting limit in the samples with the non-compliant blank, the data may be reported with the analyte qualified.

9.5.6.4 If surrogate recoveries are not acceptable, the data may be evaluated to determine if the method blank has served the purpose of demonstrating that the analysis is free of contamination.

9.5.7 Contingencies for Handling Out-of-Control or Unacceptable Data

- Inform project manager for client input and fill out job exception report.
- Rerun samples to confirm results.
- Resample if client or project manager requests.

10.0 Procedure

10.1 Calibration & Standardization

10.1.1 Instrument Tuning and Performance Check:

The GC/MS system is calibrated using Perflurotributylamine (PFTBA) according to the recommended tuning conditions suggested by the vendor.

An instrument performance check of Bromofluorobenzene (BFB) is analyzed at the beginning of each 12-hour analysis period.

The analysis of the instrument performance check is performed using the following procedure:

- 1ul of a 50ng/ul solution is directly injected, resulting in a 50ng injection of BFB into the GC/MS.
- A blank containing 50 ng BFB is purged.

10.1.2 The mass spectrum of BFB is acquired using the following procedure:

- The apex scan, one scan immediately preceding the apex and one scan immediately following the apex are averaged. The spectrum is background subtracted using a single scan no more than 20 scans prior to the elution of BFB.

- A scan across the peak at one half the peak height may be averaged. The spectrum is background subtracted using a single scan no more than 20 scans prior to the elution of BFB. Background correction cannot include any part of the target peak.

- A single scan of the peak may also be used for the evaluation of the tune. The spectrum is background subtracted using a single scan no more than 20 scans prior to the elution of BFB. Background correction cannot include any part of the target peak

- The mass spectrum of BFB must pass the technical acceptance criteria given in Table 2.

10.1.3 Initial Calibration (ICAL):

The instrument performance check must meet the technical acceptance criteria prior to the analysis of an initial curve or samples. The GC/MS system is calibrated using a minimum of five levels of concentrations. All compounds of interest are included. (See section 9.4 for initial calibration acceptance criteria.)

Solutions containing target compounds and system monitoring compounds are analyzed at the following concentrations:

5 ml Purge Analysis

Standard	Solvent	Working Standard Conc.	Amount Added (ul)	Final Vol. (mL)	Final Conc. (ug/L)
VSTD001	MeOH	100ng/ul	1	100	1
VSTD005*	MeOH	100ng/ul	5	100	5
VSTD010	MeOH	100ng/ul	5	50	10
VSTD025	MeOH	100ng/ul	12.5	50	25
VSTD050	MeOH	100ng/ul	25	50	50
VSTD100	MeOH	100ng/ul	50	50	100

5 gram (soil) Purge Analysis

Standard	Solvent	Working Standard Conc.	Amount Added (ul)	Final Vol. (mL)	Final Conc. (ug/kg)
VSTD005	MeOH	100ng/ul	2.5	50	5
VSTD010*	MeOH	100ng/ul	5	50	10
VSTD020	MeOH	100ng/ul	10	50	20
VSTD050	MeOH	100ng/ul	25	50	50
VSTD100	MeOH	100ng/ul	50	50	100
VSTD200	MeOH	100ng/ul	100	50	200

25 ml Purge Analysis

Standard	Solvent	Working Standard Conc.	Amount Added (ul)	Final Vol. (mL)	Final Conc. Water (ug/L)
VSTD001	MeOH	100ng/ul	1	100	1
VSTD004	MeOH	100ng/ul	4	100	4
VSTD010	MeOH	100ng/ul	5	50	10
VSTD020	MeOH	100ng/ul	10	50	20
VSTD040	MeOH	100ng/ul	20	50	40

* optional 6th point for the initial calibration

10.1.4 Continuing Calibration Verification (CCV):

Every 12 hours of sample analysis the laboratory must demonstrate that the instrument has drifted or changed minimally by performing an instrument performance check and continuing calibration verification. (See section 9.4 for continuing calibration acceptance criteria.)

10.2 Before Analysis

10.2.1 Once initial calibration criteria has been met, and prior to analyzing samples and required blanks, Each GC/MS system must be routinely checked by analyzing a Continuing Calibration Verification (CCV) standard containing all compounds (including internal standards and system monitoring compounds) at a concentration of 25ug/L for 5ml analysis, 10ug/L for 25ml analysis or 50ug/Kg for soil.

10.2.2 If time remains after initial calibration criteria have been met, it may not be necessary to perform a CCV. The 25 ug/L (10ug/L for 25ml or 50ug/Kg for soil) standard may be evaluated against the new initial curve and used as the CCV.

10.2.3 If there is no time remaining in the 12-hour period, the instrument performance check (BFB) must be analyzed along with a new CCV.

10.2.4 Procedure for Continuing Calibration:

10.2.4.1 **5ml Water:** 12.5ul of target compound mixture is added to a 50ml volumetric flask. A 5ml aliquot is analyzed. Internal standards and system monitoring compounds are added by the auto sampler prior to analysis.

10.2.4.2 **25ml Water:** 5ul of target compound mixture is added to a 50ml volumetric flask. A 25ml aliquot is analyzed. Internal standards and system monitoring compounds are added by the auto sampler prior to analysis.

10.2.4.3 **Soil:** 25ul of target compound mixture is added to a 50ml volumetric flask. A 5ml aliquot is transferred to a sample vial. Internal standards and system monitoring compounds are added by the auto sampler prior to analysis

10.3 Sample Analysis

10.3.1 BFB tuning criteria and GC/MS calibration verification must be met before sample analysis begins.

10.3.2 The acquisition time of the BFB tune establishes a 12hr. batch. The CCV, MSB, and VBLK must be analyzed within 12hrs, unless specified by the client request. The remaining time in the 12hr batch is utilized to run samples of similar matrix. The time of initiation of purging is considered the injection time. All aqueous samples are considered a water matrix. All solid samples, with the exception of sludges, are considered soil matrix. Sludges are run medium level.

10.3.3 Samples and standard solutions are brought to ambient temperature before analysis.

10.3.4 Prior to the analysis of samples, a method blank must be analyzed in accordance with the associated procedures for a given matrix. Technical criteria for method blanks must be met prior to sample analysis.

10.3.5 Within the analytical batch an LCS must be analyzed in accordance with the associated procedures for a given matrix. Technical criteria for the LCS must be met with each batch.

10.4 Water Sample Analysis

10.4.1 A 5ml sample aliquot is spiked with internal and system monitoring compounds to a final concentration of 25 ug/L each. 25ml analysis requires a final concentration of 10ug/L. The spike may be performed manually with a Hamilton gas tight syringe or the auto sampler may be used. The sample is then loaded onto the auto sampler where it is in turn transferred to the purge chamber.

10.4.2 The sample is purged for 11.0 ± 1 minute at ambient temperature.

10.4.3 At the end of the purge time, the sample is desorbed onto the gas chromatograph column by rapidly heating the trap from 190C to 250^{0}C (depending on manufacturer specifications) while the trap is back flushed with Helium between 20 - 60 ml/minute according to the manufactures specifications. The sample is desorbed onto the column and the gas chromatograph temperature ramping program is initiated.

10.4.4 While the trap is in the bake mode, the purge chamber is flushed with two 5ml aliquots of reagent water in order to avoid possible contamination from carryover of target compounds.

10.4.5 After the sample has desorbed, the trap is conditioned from 190^{0}C to 260^{0}C according to the manufactures specifications. After baking, the trap is ready for the next sample.

10.4.6 Dilutions may be necessary if the concentration of any target compound exceeds the working range of the calibration.

10.4.7 In the event that a dilution is required, a measured volume of sample is added to a volumetric flask then brought to volume with reagent water and inverted 3 times. The sample in the neck portion is discarded and the remainder of the sample is transferred into a 40ml VOA vial. Analysis may then proceed as previously described.

10.5 Low Level Soil/Sediment Sample Analysis

10.5.1 The low level soil method is based on a heated purge of a 5g sample mixed with reagent water containing a final concentration of 50 ug/L of internal and system monitoring compounds.

10.5.2 If a dilution of the soil/sediment is required, a smaller portion of soil may be used. The smallest amount of soil that may be used is 0.5g. If a higher dilution is required, the sample must be analyzed as a medium level soil/sediment.

10.5.3 Initial and continuing calibrations that are used for the quantitation of low soils/sediments are analyzed using the same purge and trap conditions as samples.

10.5.4 The sample consists of the entire contents of the sample container. A 5g portion is removed using a narrow metal spatula or wooden tongue depressor. This aliquot is then placed into an empty VOA vial and the weight is recorded to the nearest 0.01g. 5ml of reagent water is added to the vial and it is capped.

10.5.5 Internal standards and system monitoring compounds are added to the sample immediately prior to heating and purging by the auto sampler..

10.5.6 After reagent water is added, the soil/sediment sample is heated to 40^0C ± 1^0C then purged for 11 ± 1 minutes.

10.5.7 After purging, the sample is subjected to desorbing as described for water analysis.

10.6 Medium Level Soil/Sediment Samples

10.6.1 The medium level soil/sediment method is based on an extraction of the sample with methanol. An aliquot of the extract is then added to a 50ml of reagent water.

10.6.2 The sample consists of the entire contents of the sample container. A 5g portion is removed using a narrow metal spatula or wooden tongue depressor. This aliquot is then placed into an empty 20ml vial and the weight is recorded to the nearest 0.01g.

10.6.3 1ml of system monitor compound mixture is then added to the sample.

10.6.4 A 9ml aliquot of methanol is quickly added to the sample, bringing the final volume to 10ml. The vial is capped and the sample is shaken for 2 minutes.

10.6.5 A pre-determined amount of methanol extract is added to a 50ml volumetric flask, brought to volume with reagent water and inverted 3 times. The sample in the neck portion is discarded and the remainder of the sample is transferred into a 40ml VOA vial. Analysis may then proceed as previously described in section 10.4.

10.6.6 If sample extracts are prepared in the field (e.g. Terracore kits) then both system monitoring compounds and internal standards are added by the auto-sampler prior to analysis.

10.6.7 Table 3 may be used to determine the volume of methanol extract required for a given dilution factor.

10.7 pH Determinations For Water Samples

10.7.1 After the sample aliquots are taken from the VOA vials, the pH of the sample is determined using wide range pH paper. A checkmark will be entered in the injection logbook if the sample pH is <2, however if the sample demonstrates a pH>2, the actual pH will be noted in the injection logbook.

11.0 Calculations / Data Reduction

11.1 Calculations For MS/MSD Samples

11.1.1 The calculations to determine concentrations are the same equations described for sample analysis of a given matrix.

11.1.2 The percent recovery of the matrix spiking compounds is determined using equation:

$$\text{Matrix Spike Recovery} = \frac{SSR - SR}{SA} \times 100$$

Where: SSR = Spiked sample result
SR = Sample results
SA = Spike added

11.1.3 The relative percent difference (RPD) of the recoveries of each compound between the matrix spike and matrix spike duplicate is determined using equation:

$$RPD = \frac{|MSR - MSDR|}{\underline{1/2}\,(MSR + MSDR)} \times 100$$

Where: MSR = Matrix spike recovery
MSDR = Matrix spike duplicate recovery

11.2 Calculations For Initial Calibration

11.2.1 The relative response factor (RRF) for each target compound and each system monitoring compound is calculated using equation.

$$RRF = \frac{Ax}{Ais} \times \frac{Cis}{Cx}$$

Where,
Ax = Area of the characteristic ion (EICP) for the compound to be measured (see Table 4)
Ais = Area of the characteristic ion (EICP for the specific internal standard (see Table 4)
Cis = Concentration of the internal standard
Cx = Concentration of the compound to be measured

11.2.2 The relative response factor of the Xylenes requires the use of the area response and the concentration of the peak that represents the single isomer.

11.2.3 The relative response factor of 1,2-dichloroethene is calculated using the sum of the areas of both isomers and the sum of the concentrations.

11.2.4 The average response factor (RRF) is calculated for all compounds of interest.

11.2.5 The relative standard deviation (% RSD) is calculated over the working range of the curve for all compounds using equation:

$$\%RSD = \frac{\text{Standard Deviation}}{\text{Mean}} \times 100$$

$$\text{Standard Deviation} = \sqrt{\frac{\sum_{i=1}^{n}(\chi i - \overline{\chi})2}{n-1}}$$

Where,
Xi = each individual value used to calculate the mean
X = the mean of n values
n = the total number of values

11.3 Calculations For Continuing Calibration

11.3.1 The relative response factor (RRF) for all target compounds and system monitoring compounds is calculated using equation 11.2.1.

11.3.2 The percent difference between the initial calibration and the continuing calibration is determined for all target compounds and system monitoring compound using equation:

$$\%Difference = \frac{RRFc - RRFi}{RRFi} \times 100$$

Where,
RRFc = Relative response factor from continuing calibration standard
RRFi = Mean relative response factor from the most recent initial calibration meeting technical acceptance criteria

11.4 Percent Moisture Determinations

11.4.1 Immediately after weighing the sample for analysis, a 5-10g portion is weighed into a tarred aluminum weigh pan. The sample is then dried at 105^0C. The sample is allowed to cool. The final weight is recorded. Using the equation for % moisture, concentrations relative to the dry weight of the soil/sediment samples, may be determined.

$$\%moisture = \frac{\text{g of wet sample - g of dry sample}}{\text{g of wet sample}} \times 100$$

11.5 **Quantitation of volatile target compounds** is done using the internal standard method. The Internal Standard RRF of the continuing calibration is used in the quantitation calculation.

11.5.1 **Water Samples:** The following equation is used to calculate water samples:

$$\text{Concentration ug/L} = \frac{(Ax)\,(Is)\,(DF)}{(Ais)\,(RRF)\,(Vo)}$$

Where,

Ax = Area of the characteristic ion (EICP) for the compound to be measured (see Table 4)

Ais = Area of the characteristic ion (EICP) for the specific internal standard (see Table 4)

Is = Amount of internal standard added in nanograms (ng)

RRF= Relative response factor from the ambient temperature purge of the calibration standard.

Vo = Volume of water purged in milliliters (mL)

Df = Dilution factor. The dilution factor for analysis of water samples for volatiles by this method is defined as the ratio of the number of milliliters (mL) of water purged (i.e., Vo above) to the number of mL of the original water sample used for purging. For example, if 2.0 mL of sample is diluted to 5 mL with reagent water and purged, Df = 5 mL/2.0 mL = 2.5. If no dilution is performed, Df = 1.

11.5.2 **Low Level Soil/Sediment Samples** - The following equation is used for low level soil/sediment samples:

$$\text{Concentration ug/Kg (dry weight basis)} = \frac{(Ax)\,(Is)}{(Ais)\,(RRF)\,(Ws)\,(D)}$$

Where,

Ax, Is, Ais are as given for water.

RRF = Relative response factor form the heated purge of the calibration standard.

D = $\frac{100 - \%\ \text{moisture}}{100}$

Ws = Weight of sample added to the purge tube, in grams (g).

11.5.3 Medium Level Soil/Sediment Samples

The following equation is used for quantitation of medium level soil/sediment samples:

$$\text{Concentration ug/Kg (Dry weight basis)} = \frac{(Ax)\,(Is)\,(Vt)\,(1000)\,(Df)}{(Ais)\,(RRF)\,(Va)\,(Ws)\,(D)}$$

Where,

Ax, Is, Ais are as given for water.

RRF = Relative response factor from the ambient temperature purge of the calibration standard.

Vt = Total volume of the methanol extract in milliliters (mL).

NOTE: This volume is typically 10 mL, even though only 1 mL is transferred to the vial.

Va = Volume of the aliquot of the sample methanol extract (i.e., sample extract not including the methanol added to equal 100 uL) in micro liters (ul) added to reagent water for purging.

Ws = Weight of soil/sediment extracted, in grams (g).

D = $\frac{100 - \%\ \text{moisture}}{100}$

Df = Dilution factor. The dilution factor for analysis of soil/sediment samples for volatiles by the medium level method is defined as:

$$\frac{\text{ul most conc. extract used to make dilution + ul clean solvent}}{\text{ul most conc. extract used to make dilution}}$$

(The dilution factor is equal to 1.0 in all cases other than those requiring dilution of the sample methanol extract (Vt). The factor of 1,000 in the numerator converts the value of Vt from mL to ul.)

11.6 When quantitating the sample concentration of Xylenes (total), the areas of both the m & p Xylene peak and the o-Xylene peak are summed and the RRF determined using equation 11.2.1 are used. The concentration of each peak may be determined separately and then summed to determine the concentration of Xylene (total).

11.7 When quantitating the concentration of 1,2-Dichloroethene (total), the concentrations of the two isomers (cis and trans) are summed.

11.8 Secondary ion quantitation may be used if interferences (such as matrix effects) may cause a bias in quantitation.

11.9 If manual integration of any compound (including internal standards, system monitoring compounds, target or tentatively identified compounds) is required, the EICP of that compound will be provided. All manual integrations will be identified with an "m" and initialed and dated by the GC/MS analyst.

11.10 Tentatively Identified Compounds

11.10.1 An estimated concentration for tentatively identified compounds will be determined using the equations described above for a given matrix using the total area counts of both the tentatively identified compound and the nearest internal standard which is free of interferences.

11.10.2 The RRF used to determine all concentrations of tentatively identified compounds will be an assumed RRF of one (1).

11.10.3 All tentatively identified compounds will be qualified as "J" (estimated) and "N" (presumptive evidence).

11.11 System Monitoring Compounds

11.11.1 The recovery of all system monitoring compounds in samples, blanks matrix spikes and matrix spike duplicates, is calculated using equation:

$$\% \text{ Recovery} = \frac{\text{Concentration (amount) found}}{\text{Concentration (amount) spiked}} \times 100$$

11.11.2 The recovery limits for each system monitoring compound are laboratory established on an annual basis. The recoveries must be within the criteria limits. If they fall outside criteria limits, the results must be evaluated and the sample reanalyzed, if necessary.

11.11.3 The relative retention time (RRT) of each system monitoring compound must be within the acceptance windows of ±0.06 RRT.

11.12 Internal Standards

11.12.1 The internal standards of all samples, blanks, matrix spikes and matrix spike duplicates must be monitored. The EICP area of each internal standard must be within the range of -50.0 percent to 200.0 percent of those in the continuing calibration.

11.12.2 The relative retention time (RRT) of each internal standard must be within 0.5 minutes (30 seconds) of those in the continuing calibration.

11.13 Verification of Calculated Result

11.13.1 The laboratory analyst/data entry analyst will print out and review sample worksheets and hand calculate the result for positive hits, internal standards and surrogates for comparison to the LIMS calculated result. Corrective action will result, if needed.

12.0 Method Performance

Each analyst prior to sample analysis will perform 4 replicate QC check standards as an Initial Demonstration of Capability. The average recovery and standard deviation are calculated in the LIMS system and kept with each analyst's training file.

12.1 Method Detection Limit Study (MDL)

The method detection limit (MDL) is the lowest concentration that can be detected for a given analytical method and sample matrix with 99% confidence that the analyte is present. The MDL is determined according to the laboratory's MDL procedure in the QA Manual. MDLs reflect a calculated (statistical) value determined under ideal laboratory conditions in a clean matrix, and may not be achievable in all environmental matrices. The laboratory maintains MDL studies for analyses performed; these are verified at least annually unless method requirements require a greater frequency.

12.2 Demonstration of Capabilities

12.2.1 A one–time initial demonstration of performance for each individual method for both soils and water matrices must be generated.

12.2.2 This requires quadruplicate analysis of a mid–level check standard containing all of the standard analytes for the method using the same procedures used to analyze samples, including sample preparation.

12.2.3 Compare these results with the acceptance criteria given in the Method or to laboratory historical limits (if available).

12.2.4 Repeat the test for any analyte that does not meet the acceptance criteria. Repeated failure for any analyte indicates the need for the laboratory to evaluate the analytical procedure and take corrective action.

12.3 Training Requirements

12.3.1 The supervisor has the responsibility to ensure that this procedure is performed by an analyst who has been properly trained in its use and has the required experience.

12.3.2 The following analyst validation information is maintained for this method in the laboratory QA files.

12.3.3 The analyst must complete the laboratory safety orientation training that includes, but is not limited to, chemicals, PPE requirements, and electrical safety.

12.3.4 The analyst must read and understand this SOP.

12.3.5 The analyst must read and understand the Method used as reference for this SOP.

12.3.6 The analyst must complete a DOC or successfully analyze PT samples annually.

12.3.7 The analyst must complete the TestAmerica Quality Assurance Training.

13.0 Pollution Control

It is TestAmerica’s policy to evaluate each method and look for opportunities to minimize waste generated (i.e., examine recycling options, ordering chemicals based on quantity needed, preparation of reagents based on anticipated usage and reagent stability). Employees must abide by the policies in Section 13 of the Corporate Environmental Health and Safety Manual (CW-E-M-001) for “Waste Management and Pollution Prevention.”

14.0 Waste Management

Waste management practices are conducted consistent with all applicable rules and regulations.

Excess reagents, samples and method process wastes are disposed of in an accepted manner. Waste description rules and land disposal restrictions are followed. Waste disposal procedures are incorporated by reference to the Corporate Safety Manual. The following waste streams are produced when this method is carried out.

14.1 Waste Streams Produced by the Method

The following waste streams are produced when this method is carried out.

14.1.1 Spill Response: Any spills must be cleaned up immediately and handled correctly. Any wastes that have a pH < 7 must be disposed of in an "A" waste container. Any wastes having a pH > 7 must be disposed of in a "D" waste container.

14.1.2 Aqueous waste generated from analysis: Any wastes that have a pH < 7 must be disposed of in an "A" waste container. Any wastes having a pH > 7 must be disposed of in a "D" waste container.

14.1.3 Solvent waste generated from analysis: Solvent waste is stored in laboratory approved metal waste receptacle and labeled "C" waste. Waste receptacles are then taken to sample control where they are then properly disposed of.

14.1.4 Solid waste generated from analysis: Solid volatile analysis waste consists of soils and glass. The soil is wrapped in tin foil and placed in the solid waste receptacle. Soils used for dry weight measurements are also disposed of in this manner. Glass waste such as pipettes and vials are rinsed and disposed of in approved glass receptacles

14.1.5 Expired Standards: Expired and used standards are stored in a laboratory approved metal waste receptacle labeled “BV”. Waste receptacles are then taken to sample control where they are then properly disposed of.

15.0 References / Cross-References

- Method 8260B, “Test Methods for Evaluating Solid Waste”; SW846, Third Edition, December 1996.

16.0 Method Modifications:

Item	Method	Modification
		N/A

17.0 Attachments

Table 1. Compounds Determined by Method 8260B
Table 2. BFB Key Ions and Ion Abundance Criteria
Table 3. Volume of Medium Level Extracts for Dilution
Table 4. Characteristic Masses (m/z) for Purgeable Organic Compounds
Table 5. Poor Performing Compounds
Table 6. Job Summary Check List (Page 1 & 2)

Tables 7-15. Composition of Stock Standards

18.0 Revision History

- Revision 0, dated 18 July, 2008
    - Quality Director change, signature added
    - Section 6.0: Changed section to reflect current instrumentation and column specifications
    - Section 9: Added Trichloroethene to MSB list, took out duplicate Chlorobenzene
    - Minor grammatical changes

- Revision 01, dated -24 July, 2008
    - Sec 2.4- removed reference to jet separator and added clarification to 5 point curve reference
    - Section 7.5.5.2.1: Changed volumes used to make ICAL
    - Reduced throughout document volumes used from 25ul to 12.5ul. Also changed throughout final concentrations from 50ppb to 25ppb.
    - Sec 9.2.3 and 9.5.5.1- replaced couple with few
    - Sec 9.4.1.2- removed reference to use of mean RSD
    - Sec 9.5.1.1- added text to clarify lab practice
    - Sec 10.1.3- fixed table to reflect concentrations and volumes used.
    - Sec 10.4.3- added temperature range due to variances in instrumentation

- Revision 02, dated – 30 March, 2010
    - SOP revised. All sections.
    - Added table 5 for poor performing compounds and their criteria section 9.2.4.

Table 1: Compounds Determined by Method 8260B

Compound	CAS No.[b]	Appropriate Technique					
		5030/5035	5031	5032	5021	5041	Direct Injection
Acetone	67-64-1	pp	c	c	nd	c	c
Acetonitrile	75-05-8	pp	c	nd	nd	nd	c
Acrolein	107-02-8	pp	c	c	nd	nd	c
Acrylonitrile	107-13-1	pp	c	c	nd	c	c
Allyl alcohol	107-18-6	ht	c	nd	nd	nd	c
Allyl chloride	107-05-1	c	nd	nd	nd	nd	c
Benzene	71-43-2	c	nd	c	c	c	c
Benzyl chloride	100-44-7	c	nd	nd	nd	nd	c
Bis(2-chloroethyl)sulfide	505-60-2	pp	nd	nd	nd	nd	c
Bromoacetone	598-31-2	pp	nd	nd	nd	nd	c
Bromochloromethane	74-97-5	c	nd	c	c	c	c
Bromodichloromethane	75-27-4	c	nd	c	c	c	c
4-Bromofluorobenzene (surr)	460-00-4	c	nd	c	c	c	c
Bromoform	75-25-2	c	nd	c	c	c	c
Bromomethane	74-83-9	c	nd	c	c	c	c
n-Butanol	71-36-3	ht	c	nd	nd	nd	c
2-Butanone (MEK)	78-93-3	pp	c	c	nd	nd	c
t-Butyl alcohol	75-65-0	pp	c	nd	nd	nd	c
Carbon disulfide	75-15-0	pp	nd	c	nd	c	c
Carbon tetrachloride	56-23-5	c	nd	c	c	c	c
Chloral hydrate	302-17-0	pp	nd	nd	nd	nd	c
Chlorobenzene	108-90-7	c	nd	c	c	c	c
Chlorobenzene-d5 (IS)		c	nd	c	c	c	c
Chlorodibromomethane	124-48-1	c	nd	c	nd	c	c
Chloroethane	75-00-3	c	nd	c	c	c	c
2-Chloroethanol	107-03-3	pp	nd	nd	nd	nd	c
2-Chloroethyl vinyl ether	110-75-8	c	nd	c	nd	nd	c
Chloroform	67-66-3	c	nd	c	c	c	c
Chloromethane	74-87-3	c	nd	c	c	c	c
Chloroprene	126-99-8	c	nd	nd	nd	nd	c
3-Chloropropionitrile	542-76-7	I	nd	nd	nd	nd	pc
Crotonaldehyde	4170-30-3	pp	c	nd	nd	nd	c
1,2-Dibromo-3-chloropropane	96-12-8	pp	nd	nd	c	nd	c
1,2-Dibromoethane	106-93-4	c	nd	nd	c	nd	c
Dibromomethane	74-95-3	c	nd	c	c	c	c
1,2-Dichlorobenzene	95-50-1	c	nd	nd	c	nd	c

Compound	CAS No.[b]	Appropriate Technique					
		5030/5035	5031	5032	5021	5041	Direct Injection
1,3-Dichlorobenzene	541-73-1	c	nd	nd	c	nd	c
1,4-Dichlorobenzene	106-46-7	c	nd	nd	c	nd	c
1,4-Dichlorobenzene-d4 (IS)		c	nd	nd	c	nd	c
cis-1,4-Dichloro-2-butene	1476-11-5	c	nd	c	nd	nd	c
trans-1,4-Dichloro-2-butene	110-57-6	pp	nd	c	nd	nd	c
Dichlorodifluoromethane	75-71-8	c	nd	c	c	nd	c
1,1-Dichloroethane	75-34-3	c	nd	c	c	c	c
1,2-Dichloroethane	107-06-2	c	nd	c	c	c	c
1,2-Dichloroethane-d4 (surr)		c	nd	c	c	c	c
1,1-Dichloroethene	75-35-4	c	nd	c	c	c	c
trans-1,2-Dichloroethene	156-60-5	c	nd	c	c	c	c
1,2-Dichloropropane	78-87-5	c	nd	c	c	c	c
1,3-Dichloro-2-propanol	96-23-1	pp	nd	nd	nd	nd	c
cis-1,3-Dichloropropene	10061-01-5	c	nd	c	nd	c	c
trans-1,3-Dichloropropene	10061-02-6	c	nd	c	nd	c	c
1,2,3,4-Diepoxybutane	1464-53-5	c	nd	nd	nd	nd	c
Diethyl ether	60-29-7	c	nd	nd	nd	nd	c
1,4-Difluorobenzene (I.S.)	540-36-3	nd	nd	nd	nd	c	c
1,4-Dioxane	123-91-1	pp	c	c	nd	nd	c
Epichlorohydrin	106-89-8	I	nd	nd	nd	nd	c
Ethanol	64-17-5	I	c	c	nd	nd	c
Ethyl acetate	141-78-6	I	c	nd	nd	nd	c
Ethylbenzene	100-41-4	c	nd	c	c	c	c
Ethylene oxide	75-21-8	pp	c	nd	nd	nd	c
Ethyl methacrylate	97-63-2	c	nd	c	nd	nd	c
Fluorobenzene (IS)	462-06-6	c	nd	nd	nd	nd	nd
Hexachlorobutadiene	87-68-3	c	nd	nd	c	nd	c
Hexachloroethane	67-72-1	I	nd	nd	nd	nd	c
2-Hexanone	591-78-6	pp	nd	c	nd	nd	c
2-Hydroxypropionitrile	78-97-7	I	nd	nd	nd	nd	pc
Iodomethane	74-88-4	c	nd	c	nd	c	c
Isobutyl alcohol	78-83-1	pp	c	nd	nd	nd	c
Isopropylbenzene	98-82-8	c	nd	nd	c	nd	c
Malononitrile	109-77-3	pp	nd	nd	nd	nd	c
Methacrylonitrile	126-98-7	pp	I	nd	nd	nd	c
Methanol	67-56-1	I	c	nd	nd	nd	c
Methylene chloride	75-09-2	c	nd	c	c	c	c

Compound	CAS No.[b]	Appropriate Technique					
		5030/5035	5031	5032	5021	5041	Direct Injection
Methyl methacrylate	80-62-6	c	nd	nd	nd	nd	c
4-Methyl-2-pentanone (MIBK)	108-10-1	pp	c	c	nd	nd	c
Naphthalene	91-20-3	c	nd	nd	c	nd	c
Nitrobenzene	98-95-3	c	nd	nd	nd	nd	c
2-Nitropropane	79-46-9	c	nd	nd	nd	nd	c
N-Nitroso-di-n-butylamine	924-16-3	pp	c	nd	nd	nd	c
Paraldehyde	123-63-7	pp	c	nd	nd	nd	c
Pentachloroethane	76-01-7	I	nd	nd	nd	nd	c
2-Pentanone	107-87-9	pp	c	nd	nd	nd	c
2-Picoline	109-06-8	pp	c	nd	nd	nd	c
1-Propanol	71-23-8	pp	c	nd	nd	nd	c
2-Propanol	67-63-0	pp	c	nd	nd	nd	c
Propargyl alcohol	107-19-7	pp	I	nd	nd	nd	c
B-Propiolactone	57-57-8	pp	nd	nd	nd	nd	c
Propionitrile (ethyl cyanide)	107-12-0	ht	c	nd	nd	nd	c
n-Propylamine	107-10-8	c	nd	nd	nd	nd	c
Pyridine	110-86-1	I	c	nd	nd	nd	c
Styrene	100-42-5	c	nd	c	c	c	c
1,1,1,2-Tetrachloroethane	630-20-6	c	nd	nd	c	c	c
1,1,2,2-Tetrachloroethane	79-34-5	c	nd	c	c	c	c
Tetrachloroethene	127-18-4	c	nd	c	c	c	c
Toluene	108-88-33	c	nd	c	c	c	c
Toluene-d8 (surr)	2037-26-5	c	nd	c	c	c	c
o-Toluene	95-53-4	pp	c	nd	nd	nd	c
1,2,4-Trichlorobenzene	120-82-1	c	nd	nd	c	nd	c
1,1,1-Trichloroethane	71-55-6	c	nd	c	c	c	c
1,1,2-Trichloroethane	79-00-5	c	nd	c	c	c	c
Trichloroethane	79-01-6	c	nd	c	c	c	c
Trichlorofluoromethane	75-69-4	c	nd	c	c	c	c
1,2,3-Trichloropropane	96-18-4	c	nd	c	c	c	c
Vinyl acetate	108-05-4	c	nd	c	nd	nd	c
Vinyl chloride	75-01-4	c	nd	c	c	c	c
Xylene (Total)	1330-20-7	c	nd	c	c	c	c

c= Adequate response by this technique
b= Chemical Abstract Services Registry Number
pp= Poor purging efficiency resulting in high EQLs
I= Inappropriate technique for this analyte

nd= Not determined
surr= Surrogate
IS= Internal Standard
ht= Method analyte only when purged at 80 C

pc= Poor chromatographic behavior

The following compounds are also amenable to analysis by Method 8260:

Bromombenzene
n-Butylbenzene
sec-Butlybenzene
tert-Butylbenzene
Chloroacetonitrile
1-Chlorobutane
1-Chlorohexane
2-Chlorotoluene
4-Chlorotoluene
Dibromofluoromethane
Cis-1,2-Dichloroethene
1,3-Dichloropropane
2,2-Dichloropropane
1,1-Dichloropropene
p-Isopropyltoluene
Methyl acrylate
Methyl-t-butyl ether
Pentafluorobenzene
n-Propylbenzene
1,2,3-Trichlorobenzene
1,2,4-Trimethylbenzene
1,3,5-Trimethylbenzene

Table 2. BFB Key Ions and Ion Abundance Criteria

mz	**Required Intensity (relative abundance)**
50	15 to 40% of m/z 95
75	30 to 60% of m/z 95
95	Base peak, 100% relative abundance
96	5 to 9% of m/z 95
173	less than 2% of m/z 174
174	Greater than 50% of m/z 95
175	5 to 9% of m/z 174
176	Greater than 95% but less than 101% of m/z 174
177	5 to9% of m/z 176

*Alternate tuning criteria may be used, (e.g. CLP, Method 524.2, or manufacturers' instructions), provided that method performance is not adversely affected.

Table 3. Volume of Medium Level Extracts for Dilution

Dilution Factor	Volume of Extract
1	100ul
2	50ul
5	20ul
10	10ul
20	5ul
25	4ul
40	2.5ul
50	2ul
100	1ul
200	50ul of a 1/10 Dilution

Table 4. Characteristic Masses (m/z) for Purgeable Organic Compounds

Analyte	Primary Characteristic Ion	Secondary Characteristic Ion(s)
Acetone	58	43
Acetonitrile	41	40,39
Acrolein	56	55,58
Acrylonitrile	53	52,51
Allyl alcohol	57	58,39
Allyl chloride	76	41,39,78
Benzene	78	-
Benzyl chloride	91	126,65,128
Bromoacetone	136	43,138,93,95
Bromobenzene	156	77,158
Bromochloromethane	128	49,130
Bromodichloromethane	83	85,127
Bromoform	173	175,254
Bromomethane	94	96
iso-Butanol	74	43
n-Butanol	56	41
2-Butanone	72	43
n-Butylbenzene	91	92,134
sec-Butylbenzene	105	134
tert-Butylbenzene	119	91,134
Carbon disulfide	76	78
Carbon tetrachloride	117	119
Chloral hydrate	82	44,84,86,111
Chloroacetonitrile	48	75
Chlorobenzene	112	77,114
1-Chlorobutane	56	49
Chlorodibromomethane	129	208,206
Chloroethane	64 (49*)	66 (51*)
2-Chloroethanol	49	44,43,51,80
bis-(2-Chloroethyl) sulfide	109	111,158,160
2-Chloroethyl vinyl ether	63	65,106
Chloroform	83	85
Chloromethane	50 (49*)	52 (51*)
Chloroprene	53	88,90,51
3-Chloropropionitrile	54	49,89,91
3-Chlorotoluene	91	126
4-Chlorotoluene	91	126
1,2-Dibromo-3-chloropropane	75	155,157
Dibromochloromethane	129	127
1,2-Dibromoethane	107	109,188
Dibromomethane	93	95,174
1,2-Dichlorobenzene	146	111,148
1,2-Dichlorobenzene-d_4	152	115,150
1,3-Dichlorobenzene	146	111,148
1,4-Dichlorobenzene	146	111,148
cis-1,4-Dichloro-2-butene	75	53,77,124,89
trans-1,4-Dichloro-2-butene	53	88,75

Analyte	**Primary Characteristic Ion**	**Secondary Characteristic Ion(s)**
Dichlorodifluoromethane	85	87
1,1-Dichlorothane	63	65,83
1,2-Dichloroethane	62	98
1,1-Dichlorothene	96	61,63
cis-1,2-Dichloroethene	96	61,98
trans-1,2-Dichloroethene	96	61,98
1,2-Dichloropropane	63	112
1,3-Dichloropropane	76	78
2,2-Dichloropropane	77	97
1,3-Dichloro-2-propanol	79	43,81,49
1,1-Dichloropropene	75	110,77
cis-1,3-Dichloropropene	75	77,39
trans-1,3-Dichloropropene	75	77,39
1,2,3,4-Diepoxybutane	55	57,56
Diethyl ether	74	45,59
1,4-Dioxane	88	58,43,57
Epichlorohydrin	57	49,62,51
Ethanol	31	45,27,46
Ethyl acetate	88	43,45,61
Ethylbenzene	91	106
Ethylene oxide	44	43,42
Ethyl methacrylate	69	41,99,86,114
Hexachlorobutadiene	225	223,227
Hexachloroethane	201	166,199,203
2-Hexanone	43	58,57,100
2-Hydroxypropionitrile	44	43,42,53
Iodomethane	142	127,141
Isobutyl alcohol	43	41,42,74
Isopropylbenzene	105	120
p-Isopropyl toluene	119	134,91
Malonitrile	66	39,65,38
Methacrylonitrile	41	67,39,52,66
Methyl acrylate	55	85
Methyl-t-butyl ether	73	57
Methylene chloride	84	86,49
Methyl ethyl ketone	72	43
Methyl iodide	142	127,141
Methyl methacrylate	69	41,100,39
4-Methyl-2-pentanone	100	43,58,85
Naphthalene	128	-
Nitrobenzene	123	51,77
2-Nitropropane	46	-
2-Picoline	93	66,92,78
Pentachloroethane	167	130,132,165,169
Propargyl alcohol	55	39,38,53
B-Propiolactone	42	43,44
Propionitrile (ethyl cyanide)	54	52,55,40
n-Propylamine	59	41,39
n-Propylbenzene	91	120

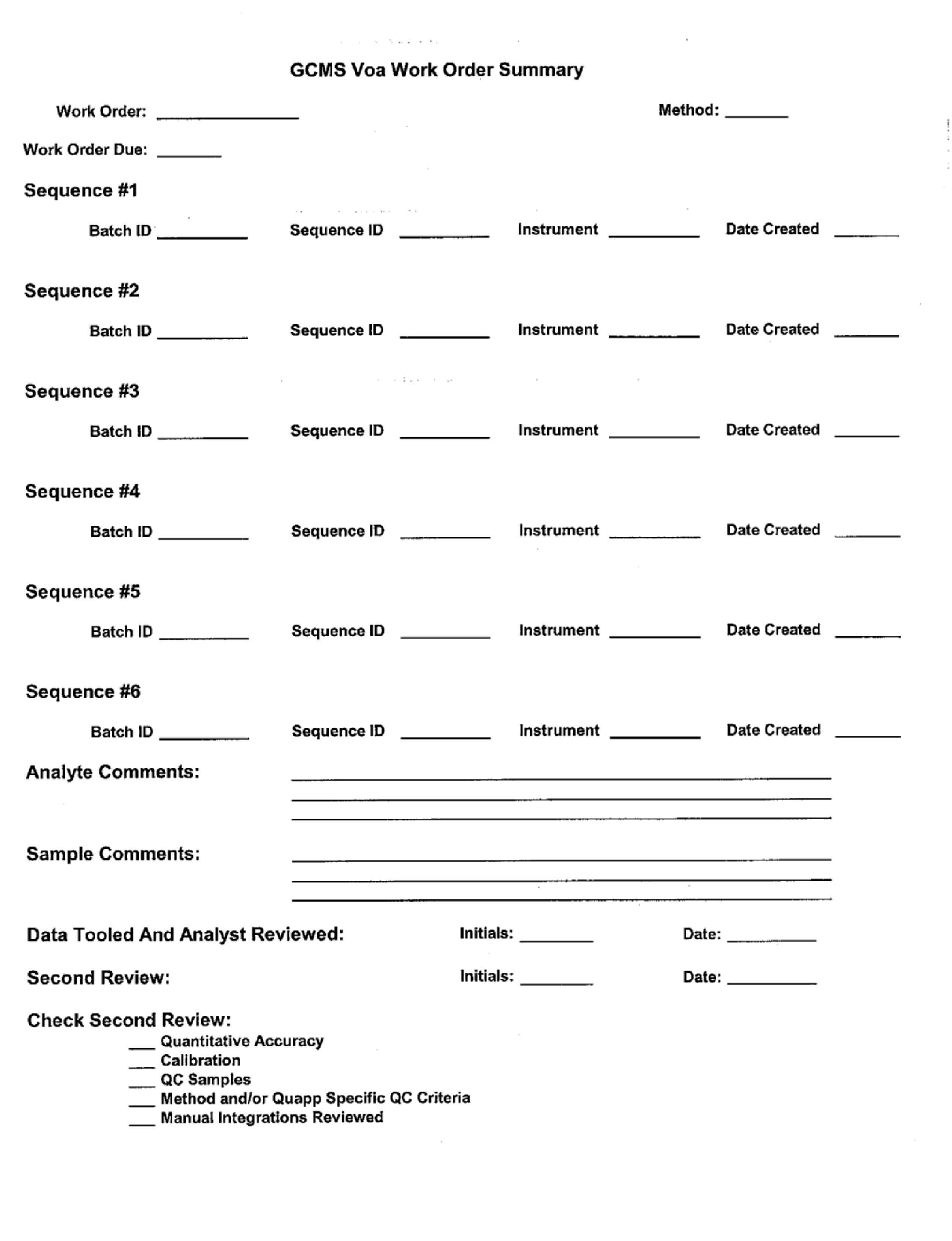
Analyte	Primary Characteristic Ion	Secondary Characteristic Ion(s)
Pyridine	79	52
Styrene	104	78
1,2,3-Trichlorobenzene	180	182,145
1,2,4-Trichlorobenzene	180	182,145
1,1,1,2-Tetrachloroethane	131	133,119
1,1,2,2-Tetrachloroethane	83	131,85
Tetrachloroethene	164	129,131,166
Toluene	92	91
1,1,1-Trichloroethane	97	99,61
1,1,2-Trichloroethane	83	97,85
Trichloroethene	95	97,130,132
Trichlorofluoromethane	151	101,153
1,2,3-Trichloropropane	75	77
1,2,4-Trimethylbenzene	105	120
1,3,5-Trimethylbenzene	105	120
Vinyl acetate	43	86
Vinyl chloride	62	64
o-Xylene	106	91
m-Xylene	106	91
p-Xylene	106	91
INTERNAL STANDARDS/SURROGATES		
Benzene-d6	84	83
Bromobenzene-d5	82	162
Bromochloromethane-d2	51	131
1,4-Difluorobenzene	114	
Chlorobenzene-d5	117	
1,4-Dichlorobenzene-d4	152	115,150
1,1,2-Trichloroethane-d3	100	
4-Bromofluorobenzene	95	174,176
Chloroform-d1	84	
Dibromofluoromethane	113	

Table 5. Poor Performing Compounds

1,1-Dimethoxyethane*	Bromomethane
1,2-Dibromo-3-chloropropane (DBCP)	Carbon Disulfide
1,4-Dioxane*	Chloroethane
2-Butanone (MEK)	Cyclohexanone*
2-Chloroethylvinyl ether	Dichlorodifluoromethane
2-Nitropropane*	Iodomethane
4-Methyl-2-pentanone (MIBK)	Methyl Acetate
Acetone	Propylene Oxide*
Acrolein	trans-1,4-Dichloro-2-butene

***** Indicates “Add” compounds that are not routinely spiked for in LCS/MS/SD

Table 6. Job Summary Check List



GCMS Voa Work Order Summary

Work Order: ______________ **Method:** ______

Work Order Due: ______

Sequence #1

Batch ID _________ **Sequence ID** _________ **Instrument** _________ **Date Created** ______

Sequence #2

Batch ID _________ **Sequence ID** _________ **Instrument** _________ **Date Created** ______

Sequence #3

Batch ID _________ **Sequence ID** _________ **Instrument** _________ **Date Created** ______

Sequence #4

Batch ID _________ **Sequence ID** _________ **Instrument** _________ **Date Created** ______

Sequence #5

Batch ID _________ **Sequence ID** _________ **Instrument** _________ **Date Created** ______

Sequence #6

Batch ID _________ **Sequence ID** _________ **Instrument** _________ **Date Created** ______

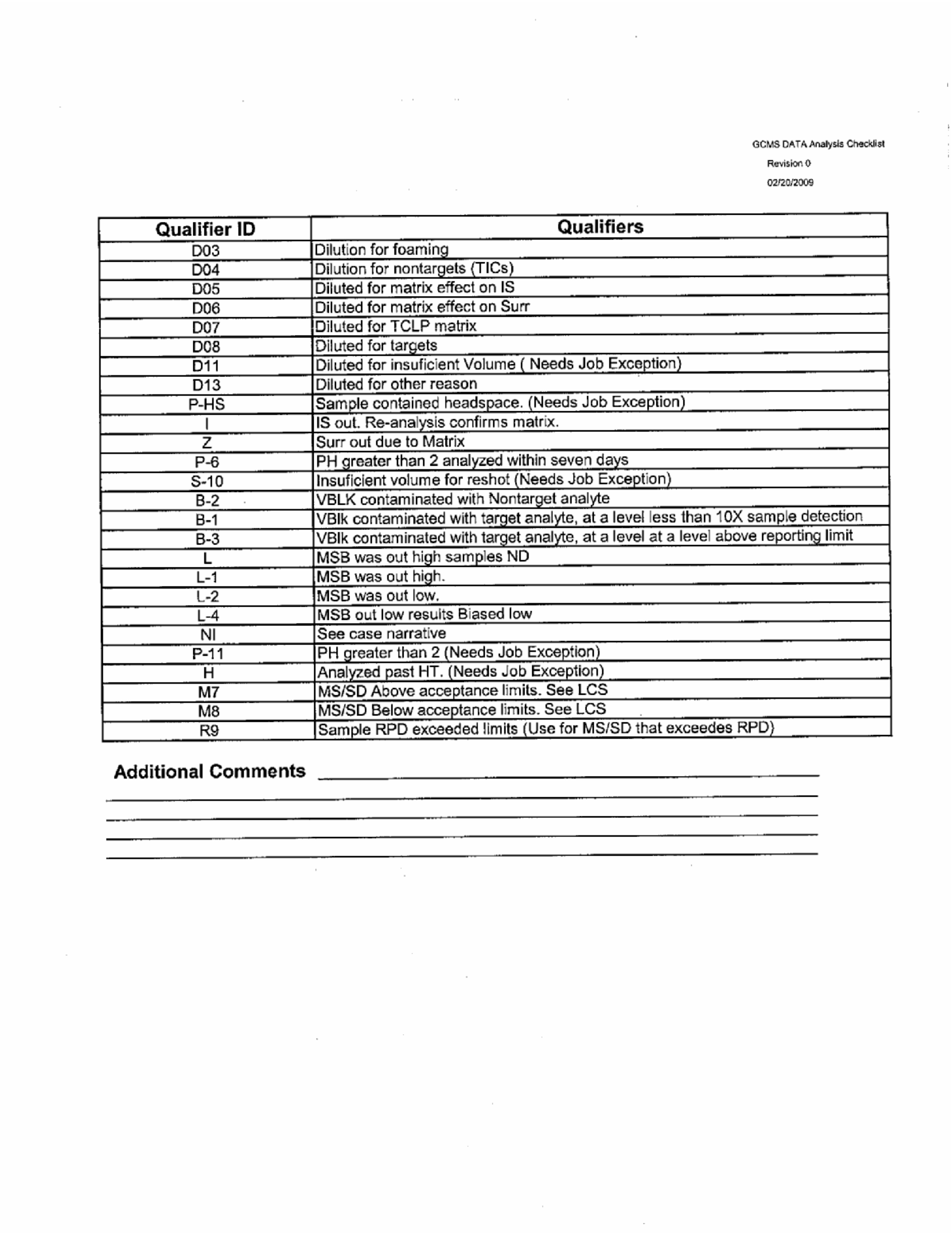
Analyte Comments: ____
____
____

Sample Comments: ____
____
____

Data Tooled And Analyst Reviewed: **Initials:** _______ **Date:** _________

Second Review: **Initials:** _______ **Date:** _________

Check Second Review:
___ Quantitative Accuracy
___ Calibration
___ QC Samples
___ Method and/or Quapp Specific QC Criteria
___ Manual Integrations Reviewed

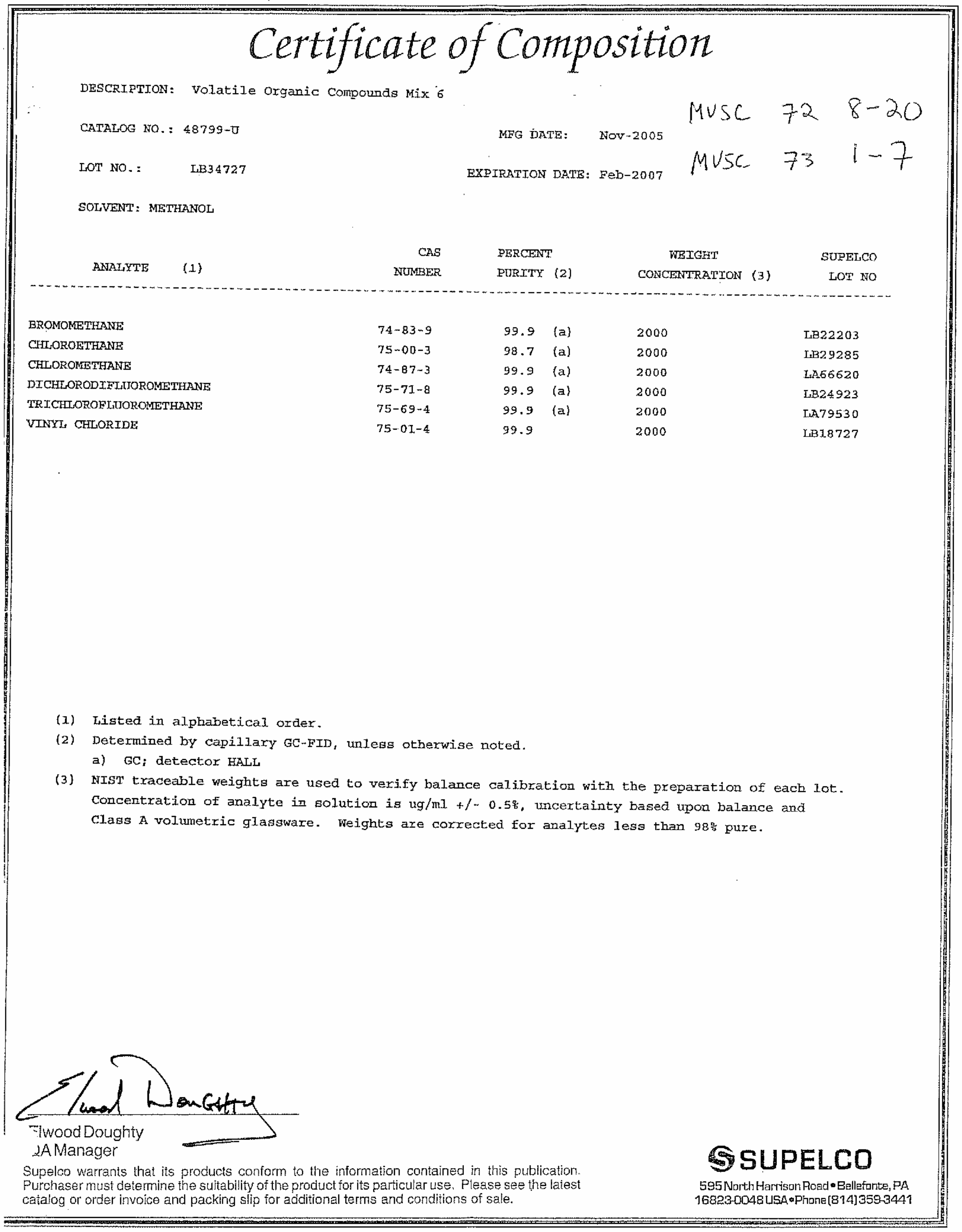


GCMS DATA Analysis Checklist
Revision 0
02/20/2009

Qualifier ID	Qualifiers
D03	Dilution for foaming
D04	Dilution for nontargets (TICs)
D05	Diluted for matrix effect on IS
D06	Diluted for matrix effect on Surr
D07	Diluted for TCLP matrix
D08	Diluted for targets
D11	Diluted for insuficient Volume (Needs Job Exception)
D13	Diluted for other reason
P-HS	Sample contained headspace. (Needs Job Exception)
I	IS out. Re-analysis confirms matrix.
Z	Surr out due to Matrix
P-6	PH greater than 2 analyzed within seven days
S-10	Insuficient volume for reshot (Needs Job Exception)
B-2	VBLK contaminated with Nontarget analyte
B-1	VBlk contaminated with target analyte, at a level less than 10X sample detection
B-3	VBlk contaminated with target analyte, at a level at a level above reporting limit
L	MSB was out high samples ND
L-1	MSB was out high.
L-2	MSB was out low.
L-4	MSB out low results Biased low
NI	See case narrative
P-11	PH greater than 2 (Needs Job Exception)
H	Analyzed past HT. (Needs Job Exception)
M7	MS/SD Above acceptance limits. See LCS
M8	MS/SD Below acceptance limits. See LCS
R9	Sample RPD exceeded limits (Use for MS/SD that exceedes RPD)

Additional Comments

Table 7. Gas Mixture



Certificate of Composition

DESCRIPTION: Volatile Organic Compounds Mix 6

CATALOG NO.: 48799-U

MFG DATE: Nov-2005

LOT NO.: LB34727

EXPIRATION DATE: Feb-2007

SOLVENT: METHANOL

MVSC 72 8-20
MVSC 73 1-7

ANALYTE (1)	CAS NUMBER	PERCENT PURITY (2)		WEIGHT CONCENTRATION (3)	SUPELCO LOT NO
BROMOMETHANE	74-83-9	99.9	(a)	2000	LB22203
CHLOROETHANE	75-00-3	98.7	(a)	2000	LB29285
CHLOROMETHANE	74-87-3	99.9	(a)	2000	LA66620
DICHLORODIFLUOROMETHANE	75-71-8	99.9	(a)	2000	LB24923
TRICHLOROFLUOROMETHANE	75-69-4	99.9	(a)	2000	LA79530
VINYL CHLORIDE	75-01-4	99.9		2000	LB18727

(1) Listed in alphabetical order.

(2) Determined by capillary GC-FID, unless otherwise noted.
a) GC; detector HALL

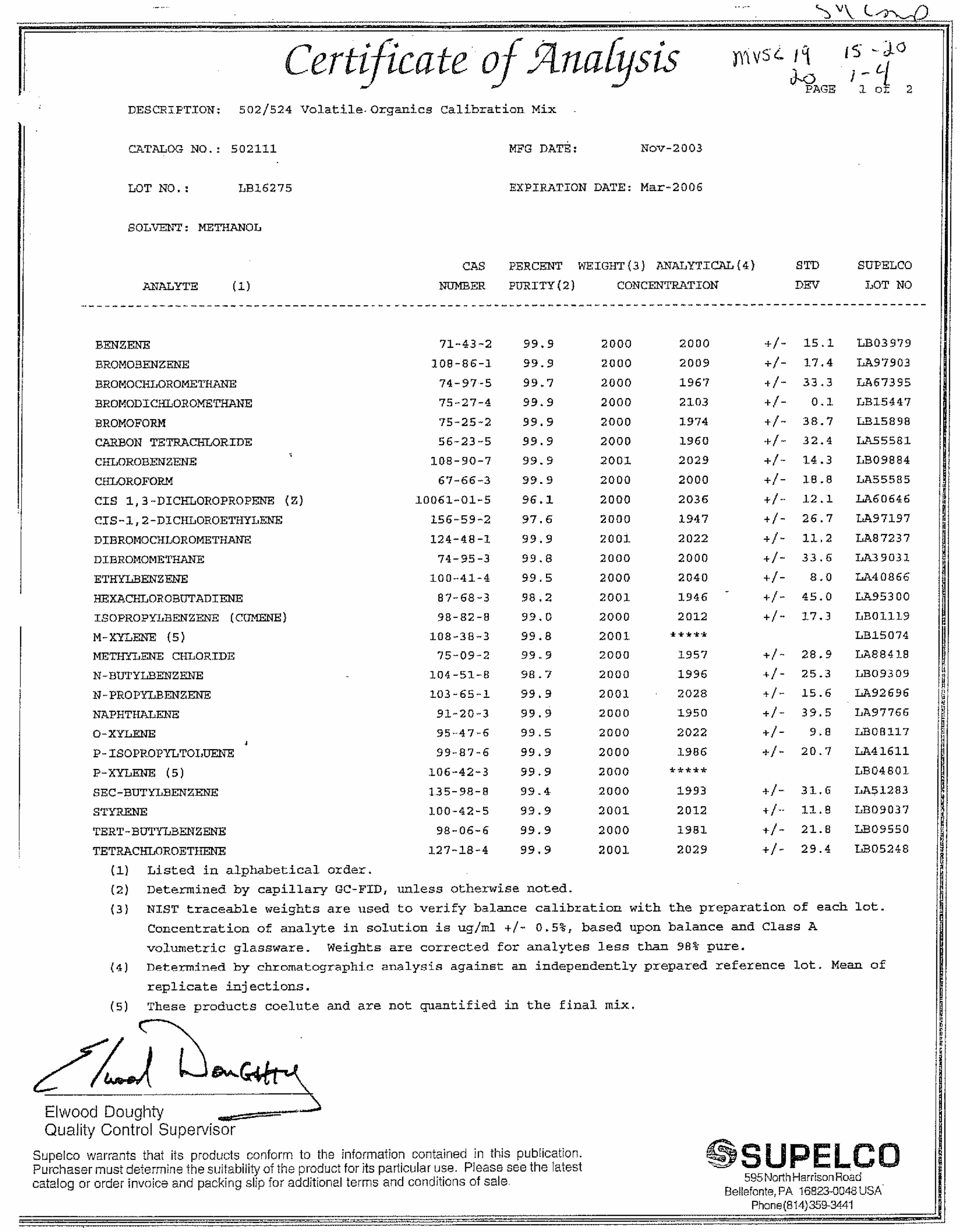
(3) NIST traceable weights are used to verify balance calibration with the preparation of each lot. Concentration of analyte in solution is ug/ml +/- 0.5%, uncertainty based upon balance and Class A volumetric glassware. Weights are corrected for analytes less than 98% pure.

Elwood Doughty
Elwood Doughty
QA Manager

Supelco warrants that its products conform to the information contained in this publication. Purchaser must determine the suitability of the product for its particular use. Please see the latest catalog or order invoice and packing slip for additional terms and conditions of sale.

SUPELCO
595 North Harrison Road • Bellefonte, PA
16823-0048 USA • Phone (814) 359-3441

Table 8. 54 Component Mixture



54 Comp

Certificate of Analysis

MVS2 19 15-20
20 1-4

PAGE 1 of 2

DESCRIPTION: 502/524 Volatile Organics Calibration Mix

CATALOG NO.: 502111 MFG DATE: Nov-2003

LOT NO.: LB16275 EXPIRATION DATE: Mar-2006

SOLVENT: METHANOL

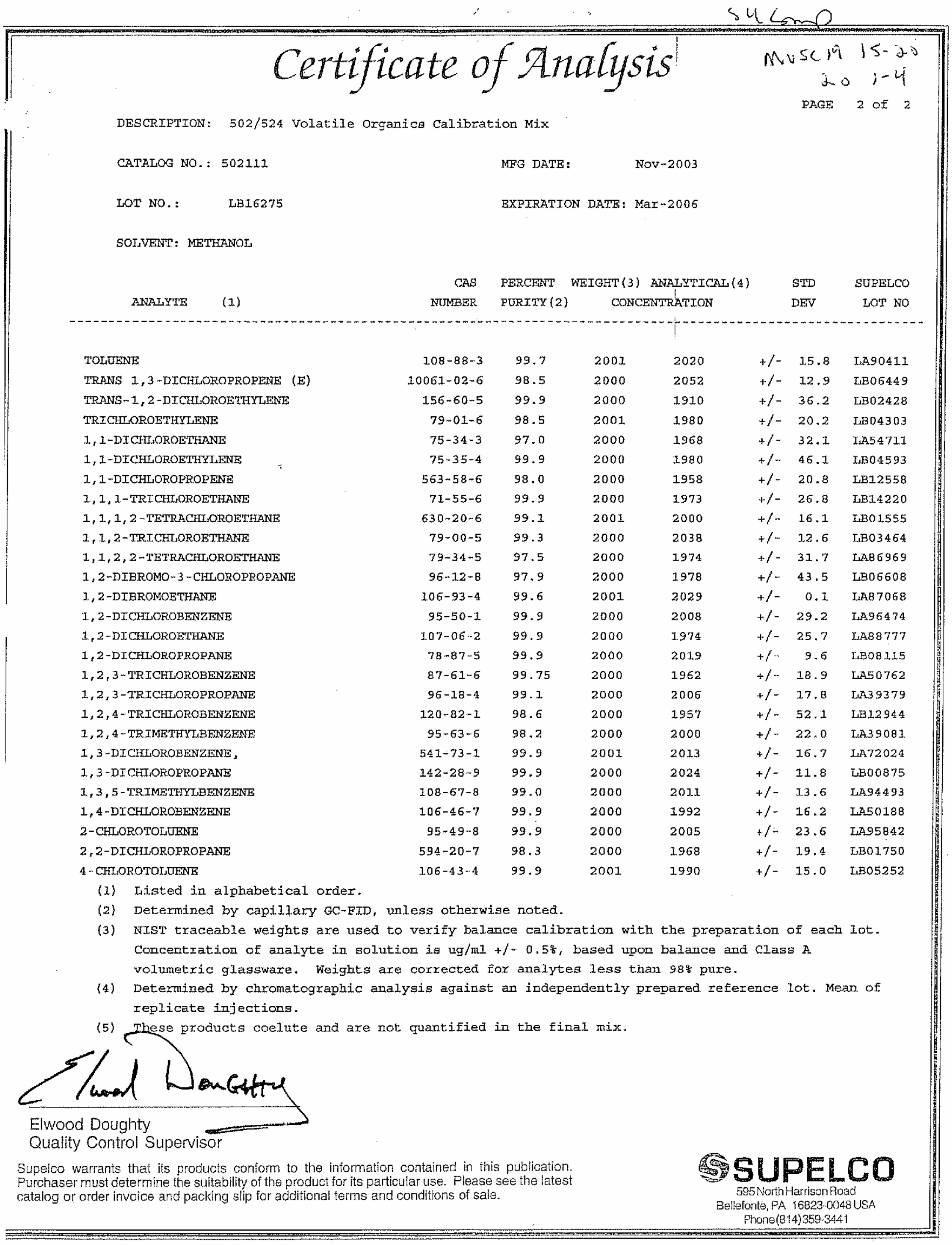
ANALYTE (1)	CAS NUMBER	PERCENT PURITY(2)	WEIGHT(3)	ANALYTICAL(4) CONCENTRATION		STD DEV	SUPELCO LOT NO
BENZENE	71-43-2	99.9	2000	2000	+/-	15.1	LB03979
BROMOBENZENE	108-86-1	99.9	2000	2009	+/-	17.4	LA97903
BROMOCHLOROMETHANE	74-97-5	99.7	2000	1967	+/-	33.3	LA67395
BROMODICHLOROMETHANE	75-27-4	99.9	2000	2103	+/-	0.1	LB15447
BROMOFORM	75-25-2	99.9	2000	1974	+/-	38.7	LB15898
CARBON TETRACHLORIDE	56-23-5	99.9	2000	1960	+/-	32.4	LA55581
CHLOROBENZENE	108-90-7	99.9	2001	2029	+/-	14.3	LB09884
CHLOROFORM	67-66-3	99.9	2000	2000	+/-	18.8	LA55585
CIS 1,3-DICHLOROPROPENE (Z)	10061-01-5	96.1	2000	2036	+/-	12.1	LA60646
CIS-1,2-DICHLOROETHYLENE	156-59-2	97.6	2000	1947	+/-	26.7	LA97197
DIBROMOCHLOROMETHANE	124-48-1	99.9	2001	2022	+/-	11.2	LA87237
DIBROMOMETHANE	74-95-3	99.8	2000	2000	+/-	33.6	LA39031
ETHYLBENZENE	100-41-4	99.5	2000	2040	+/-	8.0	LA40866
HEXACHLOROBUTADIENE	87-68-3	98.2	2001	1946	+/-	45.0	LA95300
ISOPROPYLBENZENE (CUMENE)	98-82-8	99.0	2000	2012	+/-	17.3	LB01119
M-XYLENE (5)	108-38-3	99.8	2001	*****			LB15074
METHYLENE CHLORIDE	75-09-2	99.9	2000	1957	+/-	28.9	LA88418
N-BUTYLBENZENE	104-51-8	98.7	2000	1996	+/-	25.3	LB09309
N-PROPYLBENZENE	103-65-1	99.9	2001	2028	+/-	15.6	LA92696
NAPHTHALENE	91-20-3	99.9	2000	1950	+/-	39.5	LA97766
O-XYLENE	95-47-6	99.5	2000	2022	+/-	9.8	LB08117
P-ISOPROPYLTOLUENE	99-87-6	99.9	2000	1986	+/-	20.7	LA41611
P-XYLENE (5)	106-42-3	99.9	2000	*****			LB04801
SEC-BUTYLBENZENE	135-98-8	99.4	2000	1993	+/-	31.6	LA51283
STYRENE	100-42-5	99.9	2001	2012	+/-	11.8	LB09037
TERT-BUTYLBENZENE	98-06-6	99.9	2000	1981	+/-	21.8	LB09550
TETRACHLOROETHENE	127-18-4	99.9	2001	2029	+/-	29.4	LB05248

(1) Listed in alphabetical order.
(2) Determined by capillary GC-FID, unless otherwise noted.
(3) NIST traceable weights are used to verify balance calibration with the preparation of each lot. Concentration of analyte in solution is ug/ml +/- 0.5%, based upon balance and Class A volumetric glassware. Weights are corrected for analytes less than 98% pure.
(4) Determined by chromatographic analysis against an independently prepared reference lot. Mean of replicate injections.
(5) These products coelute and are not quantified in the final mix.

Elwood Doughty

Elwood Doughty
Quality Control Supervisor

SUPELCO
595 North Harrison Road
Bellefonte, PA 16823-0048 USA
Phone (814) 359-3441



54 Comp

MVSC19 15-20
20 1-4

Certificate of Analysis

PAGE 2 of 2

DESCRIPTION: 502/524 Volatile Organics Calibration Mix

CATALOG NO.: 502111
MFG DATE: Nov-2003

LOT NO.: LB16275
EXPIRATION DATE: Mar-2006

SOLVENT: METHANOL

ANALYTE (1)	CAS NUMBER	PERCENT PURITY(2)	WEIGHT(3)	ANALYTICAL(4) CONCENTRATION		STD DEV	SUPELCO LOT NO
TOLUENE	108-88-3	99.7	2001	2020	+/-	15.8	LA90411
TRANS 1,3-DICHLOROPROPENE (E)	10061-02-6	98.5	2000	2052	+/-	12.9	LB06449
TRANS-1,2-DICHLOROETHYLENE	156-60-5	99.9	2000	1910	+/-	36.2	LB02428
TRICHLOROETHYLENE	79-01-6	98.5	2001	1980	+/-	20.2	LB04303
1,1-DICHLOROETHANE	75-34-3	97.0	2000	1968	+/-	32.1	LA54711
1,1-DICHLOROETHYLENE	75-35-4	99.9	2000	1980	+/-	46.1	LB04593
1,1-DICHLOROPROPENE	563-58-6	98.0	2000	1958	+/-	20.8	LB12558
1,1,1-TRICHLOROETHANE	71-55-6	99.9	2000	1973	+/-	26.8	LB14220
1,1,1,2-TETRACHLOROETHANE	630-20-6	99.1	2001	2000	+/-	16.1	LB01555
1,1,2-TRICHLOROETHANE	79-00-5	99.3	2000	2038	+/-	12.6	LB03464
1,1,2,2-TETRACHLOROETHANE	79-34-5	97.5	2000	1974	+/-	31.7	LA86969
1,2-DIBROMO-3-CHLOROPROPANE	96-12-8	97.9	2000	1978	+/-	43.5	LB06608
1,2-DIBROMOETHANE	106-93-4	99.6	2001	2029	+/-	0.1	LA87068
1,2-DICHLOROBENZENE	95-50-1	99.9	2000	2008	+/-	29.2	LA96474
1,2-DICHLOROETHANE	107-06-2	99.9	2000	1974	+/-	25.7	LA88777
1,2-DICHLOROPROPANE	78-87-5	99.9	2000	2019	+/-	9.6	LB08115
1,2,3-TRICHLOROBENZENE	87-61-6	99.75	2000	1962	+/-	18.9	LA50762
1,2,3-TRICHLOROPROPANE	96-18-4	99.1	2000	2006	+/-	17.8	LA39379
1,2,4-TRICHLOROBENZENE	120-82-1	98.6	2000	1957	+/-	52.1	LB12944
1,2,4-TRIMETHYLBENZENE	95-63-6	98.2	2000	2000	+/-	22.0	LA39081
1,3-DICHLOROBENZENE,	541-73-1	99.9	2001	2013	+/-	16.7	LA72024
1,3-DICHLOROPROPANE	142-28-9	99.9	2000	2024	+/-	11.8	LB00875
1,3,5-TRIMETHYLBENZENE	108-67-8	99.0	2000	2011	+/-	13.6	LA94493
1,4-DICHLOROBENZENE	106-46-7	99.9	2000	1992	+/-	16.2	LA50188
2-CHLOROTOLUENE	95-49-8	99.9	2000	2005	+/-	23.6	LA95842
2,2-DICHLOROPROPANE	594-20-7	98.3	2000	1968	+/-	19.4	LB01750
4-CHLOROTOLUENE	106-43-4	99.9	2001	1990	+/-	15.0	LB05252

(1) Listed in alphabetical order.
(2) Determined by capillary GC-FID, unless otherwise noted.
(3) NIST traceable weights are used to verify balance calibration with the preparation of each lot. Concentration of analyte in solution is ug/ml +/- 0.5%, based upon balance and Class A volumetric glassware. Weights are corrected for analytes less than 98% pure.
(4) Determined by chromatographic analysis against an independently prepared reference lot. Mean of replicate injections.
(5) These products coelute and are not quantified in the final mix.

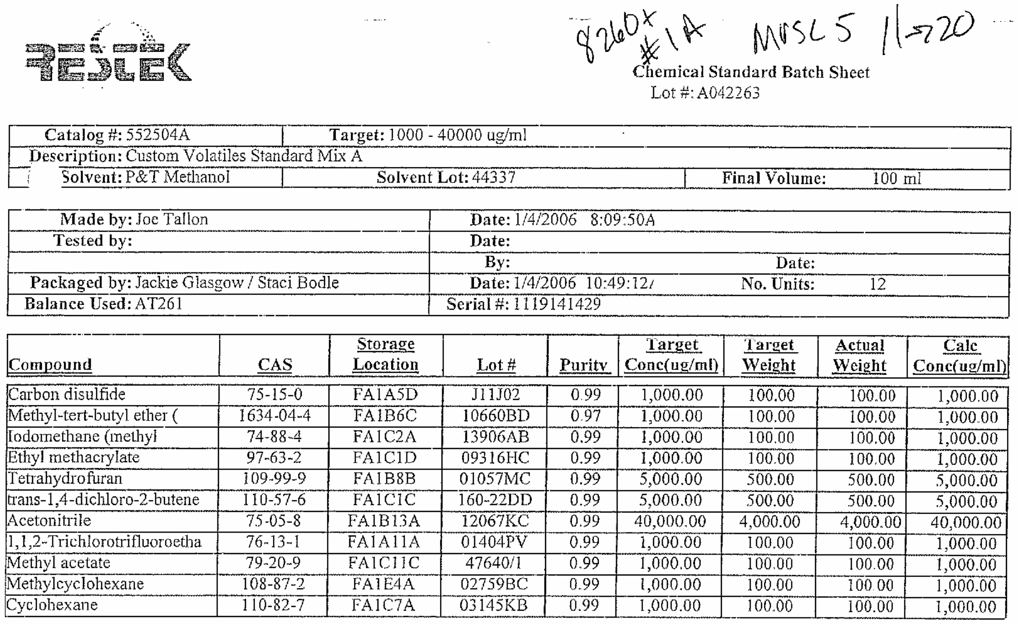
Elwood Doughty

Elwood Doughty
Quality Control Supervisor

Supelco warrants that its products conform to the information contained in this publication. Purchaser must determine the suitability of the product for its particular use. Please see the latest catalog or order invoice and packing slip for additional terms and conditions of sale.

SUPELCO
595 North Harrison Road
Bellefonte, PA 16823-0048 USA
Phone (814) 359-3441

Table 9. 8260 + Mix



RESTEK

8260x #1A MVSC5 11→20

Chemical Standard Batch Sheet

Lot #: A042263

Catalog #: 552504A	**Target:** 1000 - 40000 ug/ml		
Description: Custom Volatiles Standard Mix A			
Solvent: P&T Methanol	**Solvent Lot:** 44337	**Final Volume:**	100 ml

Made by: Joe Tallon	**Date:** 1/4/2006 8:09:50A	
Tested by:	**Date:**	
	By:	**Date:**
Packaged by: Jackie Glasgow / Staci Bodle	**Date:** 1/4/2006 10:49:12/	**No. Units:** 12
Balance Used: AT261	**Serial #:** 1119141429	

Compound	CAS	Storage Location	Lot #	Purity	Target Conc(ug/ml)	Target Weight	Actual Weight	Calc Conc(ug/ml)
Carbon disulfide	75-15-0	FA1A5D	J11J02	0.99	1,000.00	100.00	100.00	1,000.00
Methyl-tert-butyl ether (	1634-04-4	FA1B6C	10660BD	0.97	1,000.00	100.00	100.00	1,000.00
Iodomethane (methyl	74-88-4	FA1C2A	13906AB	0.99	1,000.00	100.00	100.00	1,000.00
Ethyl methacrylate	97-63-2	FA1C1D	09316HC	0.99	1,000.00	100.00	100.00	1,000.00
Tetrahydrofuran	109-99-9	FA1B8B	01057MC	0.99	5,000.00	500.00	500.00	5,000.00
trans-1,4-dichloro-2-butene	110-57-6	FA1C1C	160-22DD	0.99	5,000.00	500.00	500.00	5,000.00
Acetonitrile	75-05-8	FA1B13A	12067KC	0.99	40,000.00	4,000.00	4,000.00	40,000.00
1,1,2-Trichlorotrifluoroetha	76-13-1	FA1A11A	01404PV	0.99	1,000.00	100.00	100.00	1,000.00
Methyl acetate	79-20-9	FA1C11C	47640/1	0.99	1,000.00	100.00	100.00	1,000.00
Methylcyclohexane	108-87-2	FA1E4A	02759BC	0.99	1,000.00	100.00	100.00	1,000.00
Cyclohexane	110-82-7	FA1C7A	03145KB	0.99	1,000.00	100.00	100.00	1,000.00

RESTEK

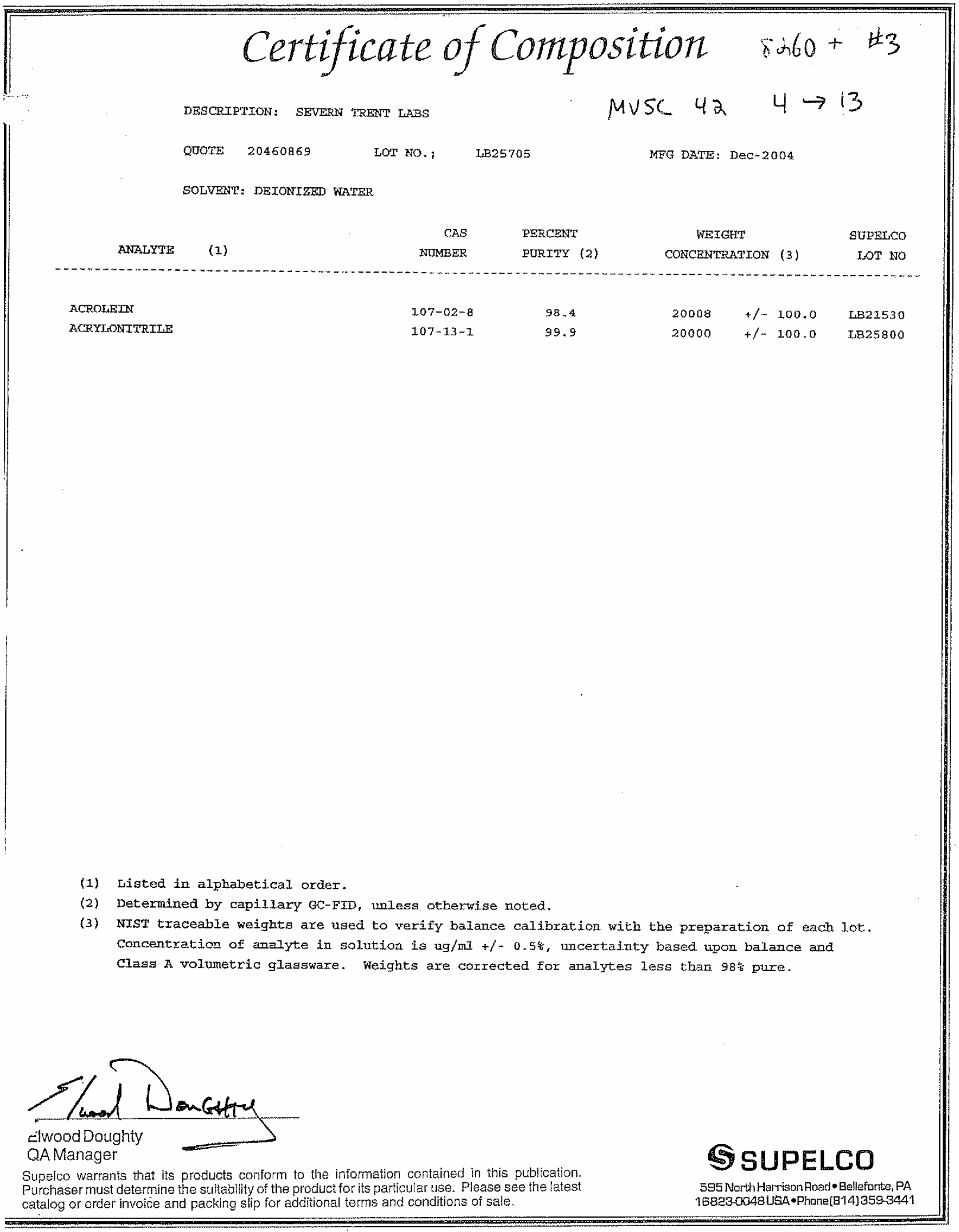
8260+ #1B MVSC5 11-720

Chemical Standard Batch Sheet
Lot #: A042264

Catalog #: 552504B	**Target:** 5000 ug/ml	
Description: Custom Volatiles Standard Mix B		
Solvent: P&T Methanol	**Solvent Lot:** A041266	**Final Volume:** 50 ml

Made by: Joe Tallon	**Date:** 1/4/2006 8:30:59A	
Tested by:	**Date:**	
	By:	**Date:**
Packaged by: Jackie Glasgow / Staci Bodle	**Date:** 1/4/2006 10:54:16/	**No. Units:** 12
Balance Used: AT261	**Serial #:** 1119141429	

Compound	CAS	Storage Location	Lot #	Purity	Target Conc(ug/ml)	Target Weight	Actual Weight	Calc Conc(ug/ml)
2-Chloroethyl vinyl ether	110-75-8	FA1A11D	03206CI	0.99	5,000.00	250.00	250.00	5,000.00



Certificate of Composition

8260 + #3

MVSC 42 4 → 13

DESCRIPTION: SEVERN TRENT LABS

QUOTE 20460869 LOT NO.: LB25705 MFG DATE: Dec-2004

SOLVENT: DEIONIZED WATER

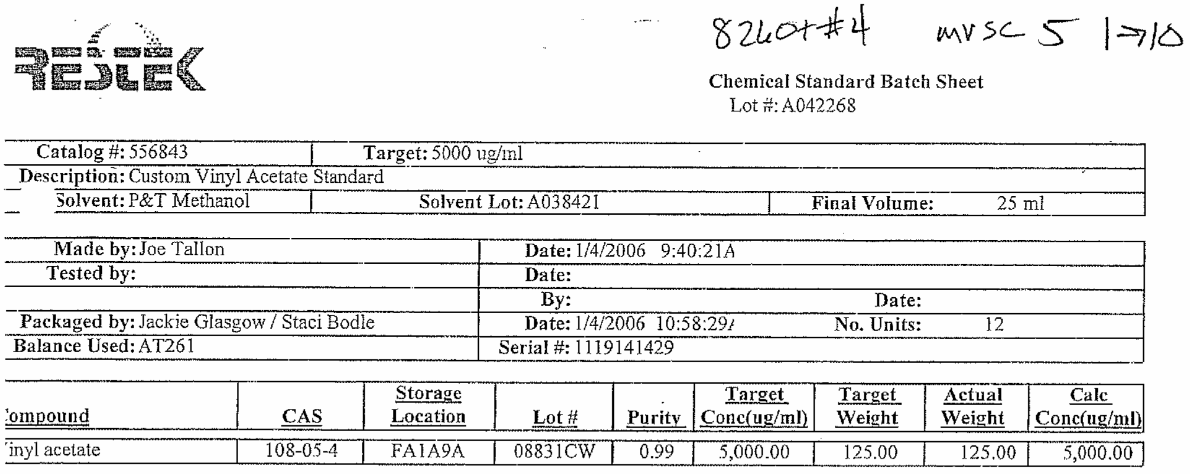
ANALYTE (1)	CAS NUMBER	PERCENT PURITY (2)	WEIGHT CONCENTRATION (3)	SUPELCO LOT NO
ACROLEIN	107-02-8	98.4	20008 +/- 100.0	LB21530
ACRYLONITRILE	107-13-1	99.9	20000 +/- 100.0	LB25800

(1) Listed in alphabetical order.
(2) Determined by capillary GC-FID, unless otherwise noted.
(3) NIST traceable weights are used to verify balance calibration with the preparation of each lot. Concentration of analyte in solution is ug/ml +/- 0.5%, uncertainty based upon balance and Class A volumetric glassware. Weights are corrected for analytes less than 98% pure.

Elwood Doughty
QA Manager

Supelco warrants that its products conform to the information contained in this publication. Purchaser must determine the suitability of the product for its particular use. Please see the latest catalog or order invoice and packing slip for additional terms and conditions of sale.

SUPELCO
595 North Harrison Road • Bellefonte, PA
16823-0048 USA • Phone (814) 359-3441



8260r#4 mvsc 5 1→10

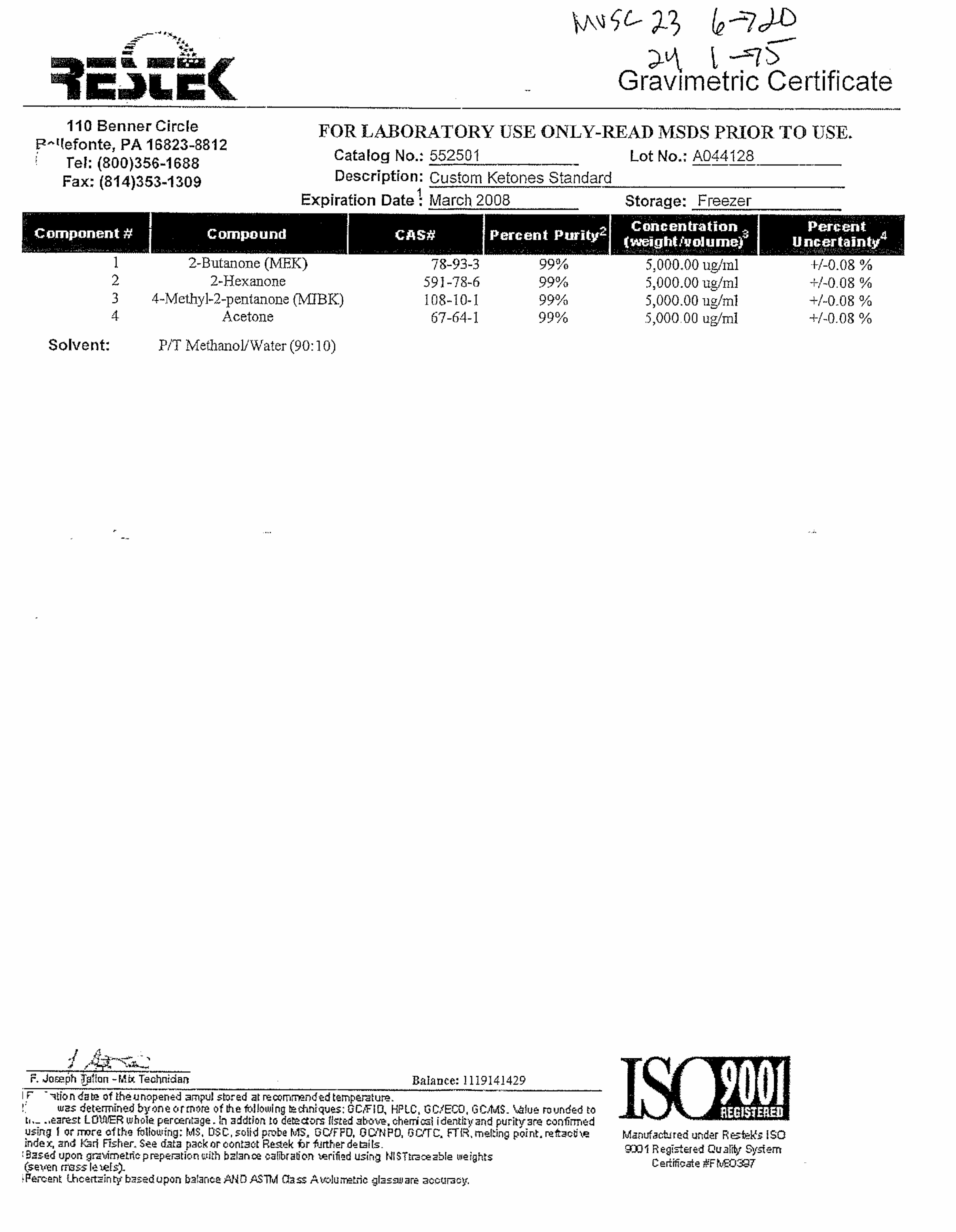
RESTEK

Chemical Standard Batch Sheet
Lot #: A042268

Catalog #: 556843
Target: 5000 ug/ml
Description: Custom Vinyl Acetate Standard
Solvent: P&T Methanol
Solvent Lot: A038421
Final Volume: 25 ml

Made by: Joe Tallon
Date: 1/4/2006 9:40:21A
Tested by:
Date:
By:
Date:
Packaged by: Jackie Glasgow / Staci Bodle
Date: 1/4/2006 10:58:29
No. Units: 12
Balance Used: AT261
Serial #: 1119141429

ompound	CAS	Storage Location	Lot #	Purity	Target Conc(ug/ml)	Target Weight	Actual Weight	Calc Conc(ug/ml)
inyl acetate	108-05-4	FA1A9A	08831CW	0.99	5,000.00	125.00	125.00	5,000.00



MVSC 23 6→20
24 1→75

RESTEK

Gravimetric Certificate

110 Benner Circle
Bellefonte, PA 16823-8812
Tel: (800)356-1688
Fax: (814)353-1309

FOR LABORATORY USE ONLY-READ MSDS PRIOR TO USE.

Catalog No.: 552501
Lot No.: A044128
Description: Custom Ketones Standard
Expiration Date[1]: March 2008
Storage: Freezer

Component #	Compound	CAS#	Percent Purity[2]	Concentration (weight/volume)[3]	Percent Uncertainty[4]
1	2-Butanone (MEK)	78-93-3	99%	5,000.00 ug/ml	+/-0.08 %
2	2-Hexanone	591-78-6	99%	5,000.00 ug/ml	+/-0.08 %
3	4-Methyl-2-pentanone (MIBK)	108-10-1	99%	5,000.00 ug/ml	+/-0.08 %
4	Acetone	67-64-1	99%	5,000.00 ug/ml	+/-0.08 %

Solvent: P/T Methanol/Water (90:10)

F. Joseph Tallon - Mix Technician

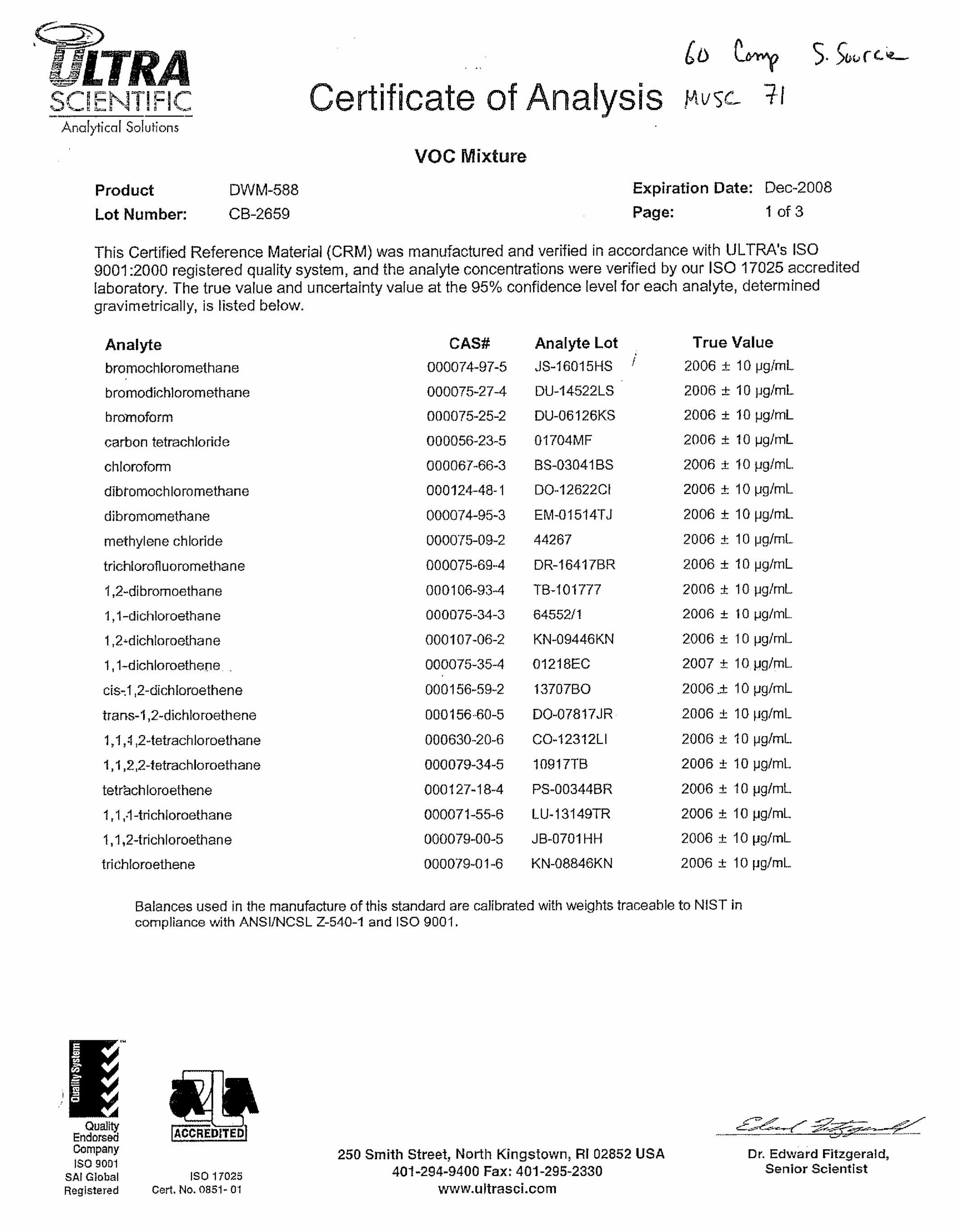
Balance: 1119141429

[1] F ation date of the unopened ampul stored at recommended temperature.
[2] was determined by one or more of the following techniques: GC/FID, HPLC, GC/ECD, GC/MS. Value rounded to the nearest LOWER whole percentage. In addition to detectors listed above, chemical identity and purity are confirmed using 1 or more of the following: MS, DSC, solid probe MS, GC/FPD, GC/NPD, GC/TC, FTIR, melting point, refractive index, and Karl Fisher. See data pack or contact Restek for further details.
[3] Based upon gravimetric preperation with balance calibration verified using NIST traceable weights (seven mass levels).
[4] Percent Uncertainty based upon balance AND ASTM Class A volumetric glassware accuracy.

ISO 9001 REGISTERED

Manufactured under Restek's ISO 9001 Registered Quality System Certificate #FM80397

Table 10. Second Source 60 Component Mixture



ULTRA SCIENTIFIC
Analytical Solutions

60 Comp S. Source

Certificate of Analysis

MUSC 71

VOC Mixture

Product	DWM-588	**Expiration Date:**	Dec-2008
Lot Number:	CB-2659	**Page:**	1 of 3

This Certified Reference Material (CRM) was manufactured and verified in accordance with ULTRA's ISO 9001:2000 registered quality system, and the analyte concentrations were verified by our ISO 17025 accredited laboratory. The true value and uncertainty value at the 95% confidence level for each analyte, determined gravimetrically, is listed below.

Analyte	CAS#	Analyte Lot	True Value
bromochloromethane	000074-97-5	JS-16015HS	2006 ± 10 µg/mL
bromodichloromethane	000075-27-4	DU-14522LS	2006 ± 10 µg/mL
bromoform	000075-25-2	DU-06126KS	2006 ± 10 µg/mL
carbon tetrachloride	000056-23-5	01704MF	2006 ± 10 µg/mL
chloroform	000067-66-3	BS-03041BS	2006 ± 10 µg/mL
dibromochloromethane	000124-48-1	DO-12622CI	2006 ± 10 µg/mL
dibromomethane	000074-95-3	EM-01514TJ	2006 ± 10 µg/mL
methylene chloride	000075-09-2	44267	2006 ± 10 µg/mL
trichlorofluoromethane	000075-69-4	DR-16417BR	2006 ± 10 µg/mL
1,2-dibromoethane	000106-93-4	TB-101777	2006 ± 10 µg/mL
1,1-dichloroethane	000075-34-3	64552/1	2006 ± 10 µg/mL
1,2-dichloroethane	000107-06-2	KN-09446KN	2006 ± 10 µg/mL
1,1-dichloroethene	000075-35-4	01218EC	2007 ± 10 µg/mL
cis-1,2-dichloroethene	000156-59-2	13707BO	2006 ± 10 µg/mL
trans-1,2-dichloroethene	000156-60-5	DO-07817JR	2006 ± 10 µg/mL
1,1,1,2-tetrachloroethane	000630-20-6	CO-12312LI	2006 ± 10 µg/mL
1,1,2,2-tetrachloroethane	000079-34-5	10917TB	2006 ± 10 µg/mL
tetrachloroethene	000127-18-4	PS-00344BR	2006 ± 10 µg/mL
1,1,1-trichloroethane	000071-55-6	LU-13149TR	2006 ± 10 µg/mL
1,1,2-trichloroethane	000079-00-5	JB-0701HH	2006 ± 10 µg/mL
trichloroethene	000079-01-6	KN-08846KN	2006 ± 10 µg/mL

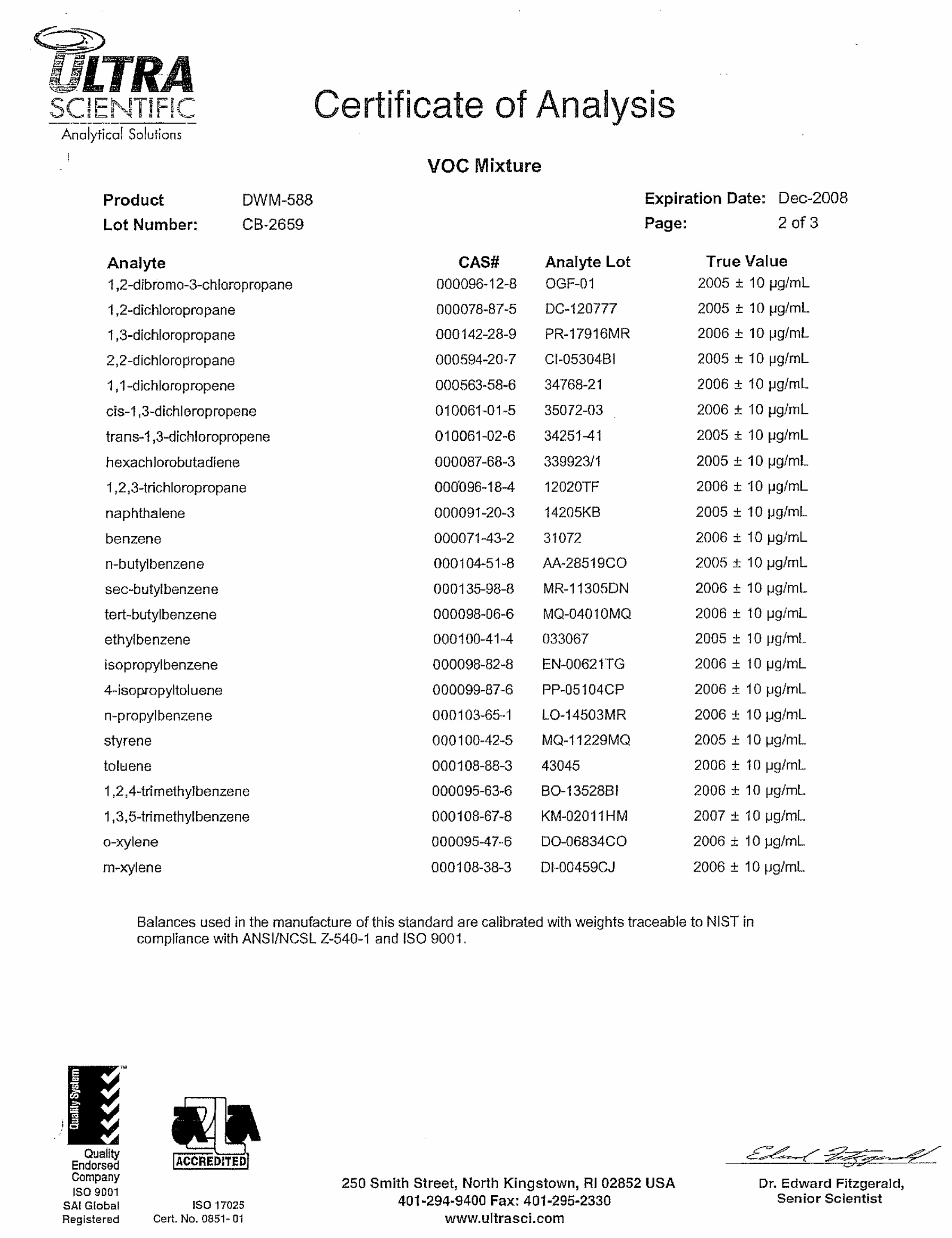
Balances used in the manufacture of this standard are calibrated with weights traceable to NIST in compliance with ANSI/NCSL Z-540-1 and ISO 9001.

Quality System
Quality Endorsed Company
ISO 9001
SAI Global
Registered

ACCREDITED
ISO 17025
Cert. No. 0851- 01

250 Smith Street, North Kingstown, RI 02852 USA
401-294-9400 Fax: 401-295-2330
www.ultrasci.com

Dr. Edward Fitzgerald,
Senior Scientist



ULTRA
SCIENTIFIC
Analytical Solutions

Certificate of Analysis

VOC Mixture

Product	DWM-588	**Expiration Date:**	Dec-2008
Lot Number:	CB-2659	**Page:**	2 of 3

Analyte	CAS#	Analyte Lot	True Value
1,2-dibromo-3-chloropropane	000096-12-8	OGF-01	2005 ± 10 µg/mL
1,2-dichloropropane	000078-87-5	DC-120777	2005 ± 10 µg/mL
1,3-dichloropropane	000142-28-9	PR-17916MR	2006 ± 10 µg/mL
2,2-dichloropropane	000594-20-7	CI-05304BI	2005 ± 10 µg/mL
1,1-dichloropropene	000563-58-6	34768-21	2006 ± 10 µg/mL
cis-1,3-dichloropropene	010061-01-5	35072-03	2006 ± 10 µg/mL
trans-1,3-dichloropropene	010061-02-6	34251-41	2005 ± 10 µg/mL
hexachlorobutadiene	000087-68-3	339923/1	2005 ± 10 µg/mL
1,2,3-trichloropropane	000096-18-4	12020TF	2006 ± 10 µg/mL
naphthalene	000091-20-3	14205KB	2005 ± 10 µg/mL
benzene	000071-43-2	31072	2006 ± 10 µg/mL
n-butylbenzene	000104-51-8	AA-28519CO	2005 ± 10 µg/mL
sec-butylbenzene	000135-98-8	MR-11305DN	2006 ± 10 µg/mL
tert-butylbenzene	000098-06-6	MQ-04010MQ	2006 ± 10 µg/mL
ethylbenzene	000100-41-4	033067	2005 ± 10 µg/mL
isopropylbenzene	000098-82-8	EN-00621TG	2006 ± 10 µg/mL
4-isopropyltoluene	000099-87-6	PP-05104CP	2006 ± 10 µg/mL
n-propylbenzene	000103-65-1	LO-14503MR	2006 ± 10 µg/mL
styrene	000100-42-5	MQ-11229MQ	2005 ± 10 µg/mL
toluene	000108-88-3	43045	2006 ± 10 µg/mL
1,2,4-trimethylbenzene	000095-63-6	BO-13528BI	2006 ± 10 µg/mL
1,3,5-trimethylbenzene	000108-67-8	KM-02011HM	2007 ± 10 µg/mL
o-xylene	000095-47-6	DO-06834CO	2006 ± 10 µg/mL
m-xylene	000108-38-3	DI-00459CJ	2006 ± 10 µg/mL

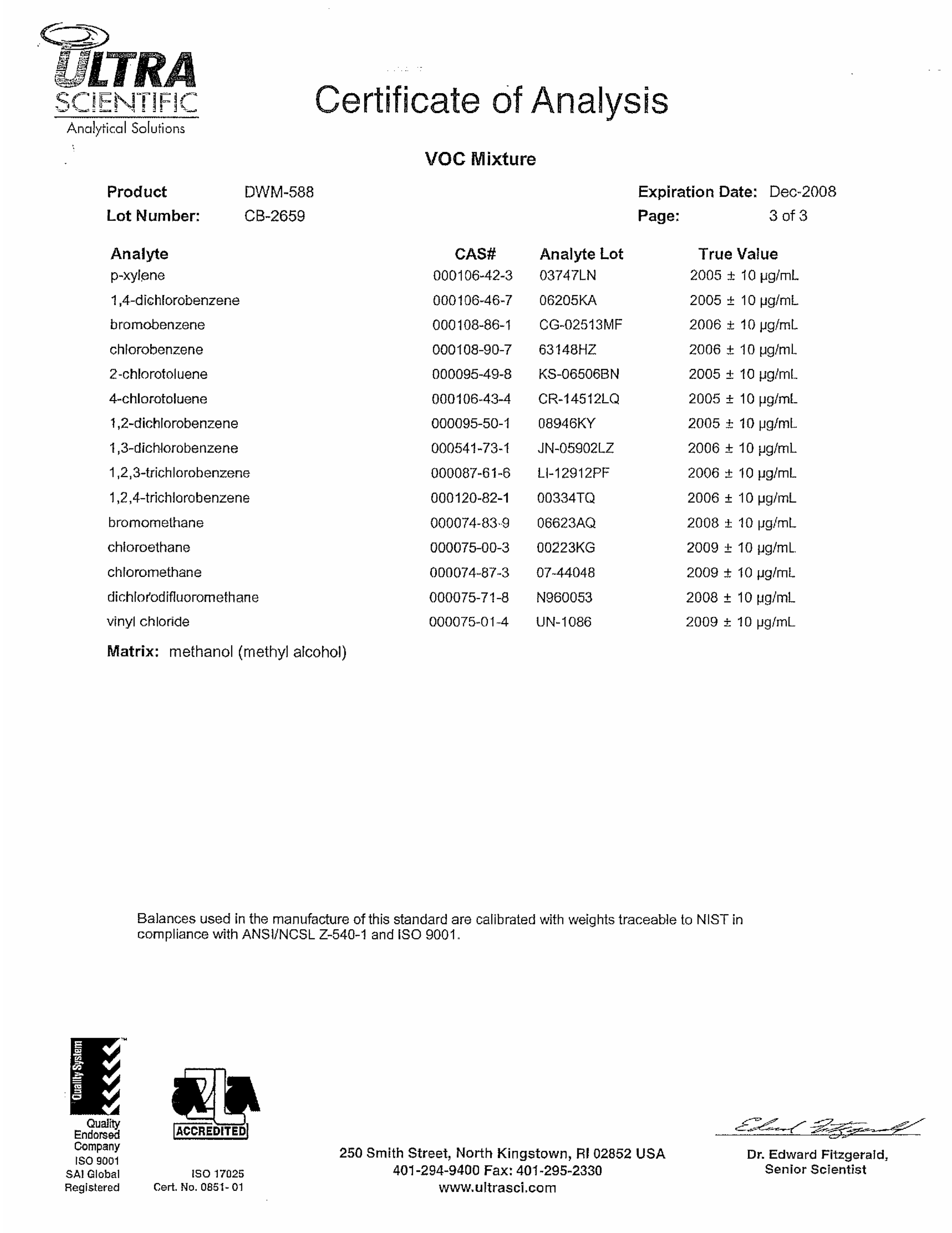
Balances used in the manufacture of this standard are calibrated with weights traceable to NIST in compliance with ANSI/NCSL Z-540-1 and ISO 9001.

Quality System
Quality Endorsed Company
ISO 9001
SAI Global Registered

ACCREDITED
ISO 17025
Cert. No. 0851-01

250 Smith Street, North Kingstown, RI 02852 USA
401-294-9400 Fax: 401-295-2330
www.ultrasci.com

Edward Fitzgerald
Dr. Edward Fitzgerald,
Senior Scientist



ULTRA SCIENTIFIC
Analytical Solutions

Certificate of Analysis

VOC Mixture

Product	DWM-588	**Expiration Date:**	Dec-2008
Lot Number:	CB-2659	**Page:**	3 of 3

Analyte	CAS#	Analyte Lot	True Value
p-xylene	000106-42-3	03747LN	2005 ± 10 µg/mL
1,4-dichlorobenzene	000106-46-7	06205KA	2005 ± 10 µg/mL
bromobenzene	000108-86-1	CG-02513MF	2006 ± 10 µg/mL
chlorobenzene	000108-90-7	63148HZ	2006 ± 10 µg/mL
2-chlorotoluene	000095-49-8	KS-06506BN	2005 ± 10 µg/mL
4-chlorotoluene	000106-43-4	CR-14512LQ	2005 ± 10 µg/mL
1,2-dichlorobenzene	000095-50-1	08946KY	2005 ± 10 µg/mL
1,3-dichlorobenzene	000541-73-1	JN-05902LZ	2006 ± 10 µg/mL
1,2,3-trichlorobenzene	000087-61-6	LI-12912PF	2006 ± 10 µg/mL
1,2,4-trichlorobenzene	000120-82-1	00334TQ	2006 ± 10 µg/mL
bromomethane	000074-83-9	06623AQ	2008 ± 10 µg/mL
chloroethane	000075-00-3	00223KG	2009 ± 10 µg/mL
chloromethane	000074-87-3	07-44048	2009 ± 10 µg/mL
dichlorodifluoromethane	000075-71-8	N960053	2008 ± 10 µg/mL
vinyl chloride	000075-01-4	UN-1086	2009 ± 10 µg/mL

Matrix: methanol (methyl alcohol)

Balances used in the manufacture of this standard are calibrated with weights traceable to NIST in compliance with ANSI/NCSL Z-540-1 and ISO 9001.

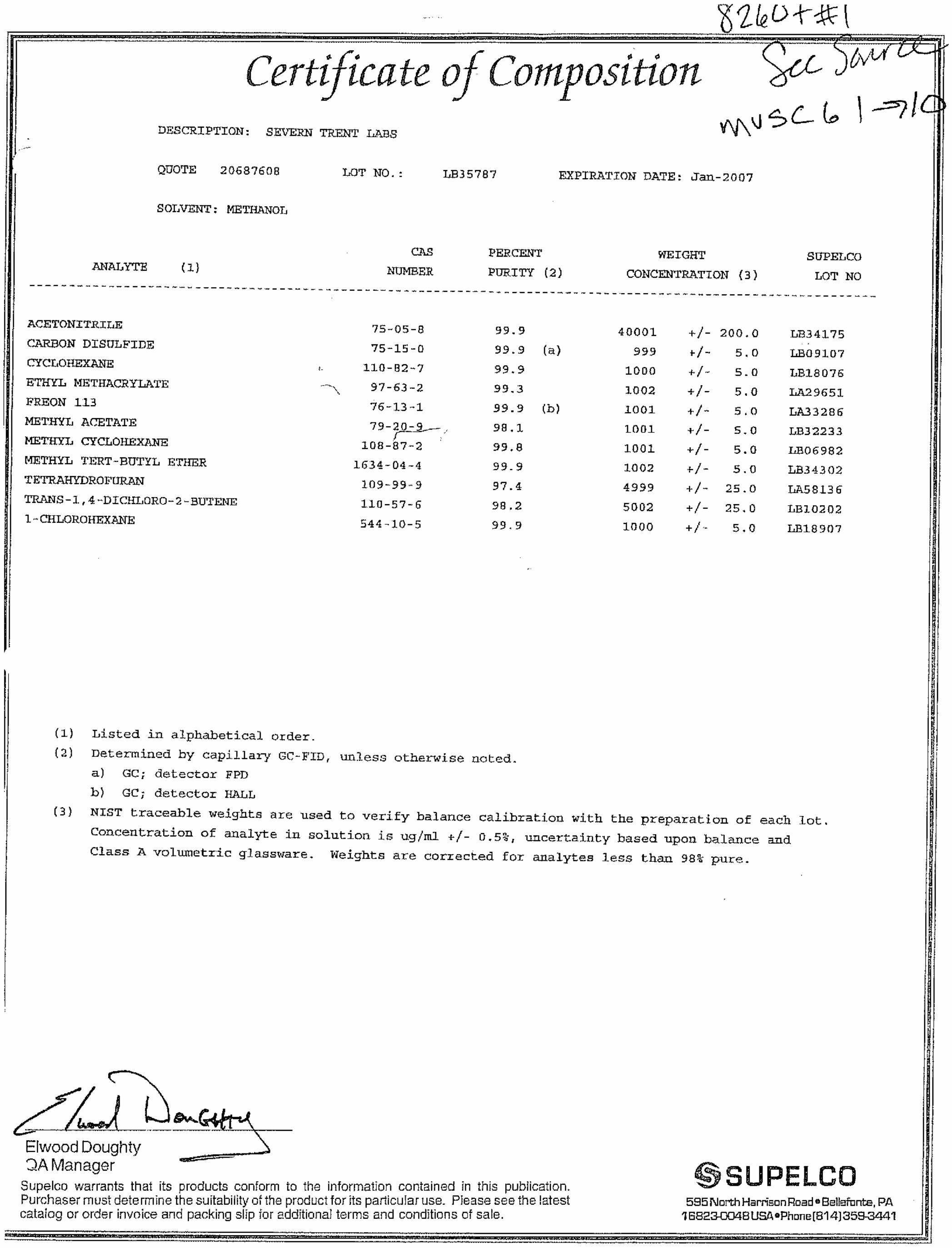
Quality System
Quality Endorsed Company
ISO 9001
SAI Global
Registered

ACCREDITED
ISO 17025
Cert. No. 0851- 01

250 Smith Street, North Kingstown, RI 02852 USA
401-294-9400 Fax: 401-295-2330
www.ultrasci.com

Dr. Edward Fitzgerald,
Senior Scientist

Table11. Second Source 8260 + Mixture



8260+#1

Sec Source

MVSC 6 1→10

Certificate of Composition

DESCRIPTION: SEVERN TRENT LABS

QUOTE 20687608 LOT NO.: LB35787 EXPIRATION DATE: Jan-2007

SOLVENT: METHANOL

ANALYTE (1)	CAS NUMBER	PERCENT PURITY (2)		WEIGHT CONCENTRATION (3)			SUPELCO LOT NO
ACETONITRILE	75-05-8	99.9		40001	+/-	200.0	LB34175
CARBON DISULFIDE	75-15-0	99.9	(a)	999	+/-	5.0	LB09107
CYCLOHEXANE	110-82-7	99.9		1000	+/-	5.0	LB18076
ETHYL METHACRYLATE	97-63-2	99.3		1002	+/-	5.0	LA29651
FREON 113	76-13-1	99.9	(b)	1001	+/-	5.0	LA33286
METHYL ACETATE	79-20-9	98.1		1001	+/-	5.0	LB32233
METHYL CYCLOHEXANE	108-87-2	99.8		1001	+/-	5.0	LB06982
METHYL TERT-BUTYL ETHER	1634-04-4	99.9		1002	+/-	5.0	LB34302
TETRAHYDROFURAN	109-99-9	97.4		4999	+/-	25.0	LA58136
TRANS-1,4-DICHLORO-2-BUTENE	110-57-6	98.2		5002	+/-	25.0	LB10202
1-CHLOROHEXANE	544-10-5	99.9		1000	+/-	5.0	LB18907

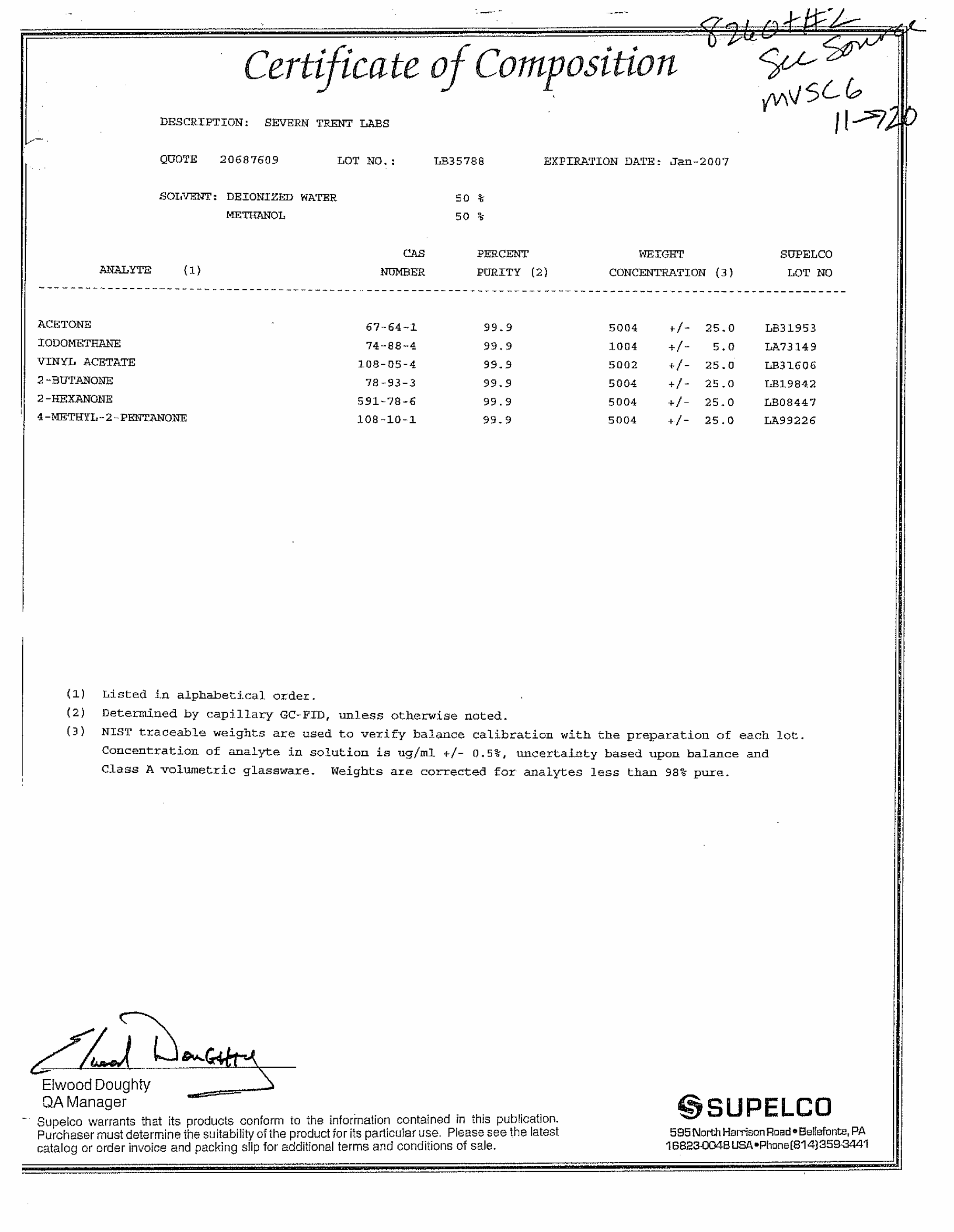
(1) Listed in alphabetical order.

(2) Determined by capillary GC-FID, unless otherwise noted.
  a) GC; detector FPD
  b) GC; detector HALL

(3) NIST traceable weights are used to verify balance calibration with the preparation of each lot. Concentration of analyte in solution is ug/ml +/- 0.5%, uncertainty based upon balance and Class A volumetric glassware. Weights are corrected for analytes less than 98% pure.

Elwood Doughty
QA Manager

SUPELCO
595 North Harrison Road • Bellefonte, PA
16823-0048 USA • Phone (814) 359-3441



8260+#2
Sec Source
MVSC6
11→20

Certificate of Composition

DESCRIPTION: SEVERN TRENT LABS

QUOTE 20687609 LOT NO.: LB35788 EXPIRATION DATE: Jan-2007

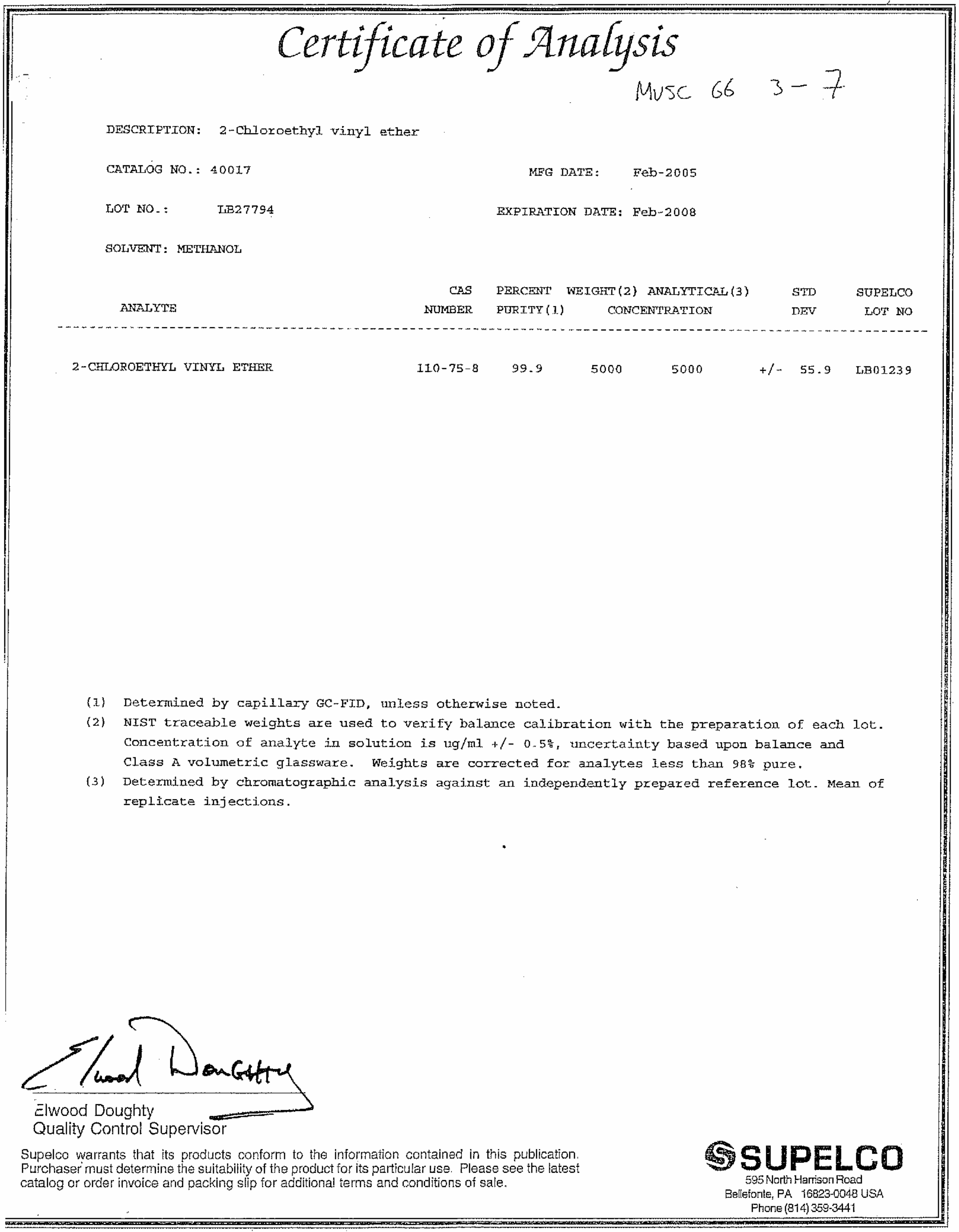
SOLVENT: DEIONIZED WATER 50 %
METHANOL 50 %

ANALYTE (1)	CAS NUMBER	PERCENT PURITY (2)	WEIGHT CONCENTRATION (3)	SUPELCO LOT NO
ACETONE	67-64-1	99.9	5004 +/- 25.0	LB31953
IODOMETHANE	74-88-4	99.9	1004 +/- 5.0	LA73149
VINYL ACETATE	108-05-4	99.9	5002 +/- 25.0	LB31606
2-BUTANONE	78-93-3	99.9	5004 +/- 25.0	LB19842
2-HEXANONE	591-78-6	99.9	5004 +/- 25.0	LB08447
4-METHYL-2-PENTANONE	108-10-1	99.9	5004 +/- 25.0	LA99226

(1) Listed in alphabetical order.
(2) Determined by capillary GC-FID, unless otherwise noted.
(3) NIST traceable weights are used to verify balance calibration with the preparation of each lot. Concentration of analyte in solution is ug/ml +/- 0.5%, uncertainty based upon balance and Class A volumetric glassware. Weights are corrected for analytes less than 98% pure.

Elwood Doughty
Elwood Doughty
QA Manager

SUPELCO
595 North Harrison Road • Bellefonte, PA
16823-0048 USA • Phone (814) 359-3441



Certificate of Analysis

MVSC 66 3-7

DESCRIPTION: 2-Chloroethyl vinyl ether

CATALOG NO.: 40017

MFG DATE: Feb-2005

LOT NO.: LB27794

EXPIRATION DATE: Feb-2008

SOLVENT: METHANOL

ANALYTE	CAS NUMBER	PERCENT PURITY(1)	WEIGHT(2) CONCENTRATION	ANALYTICAL(3) CONCENTRATION	STD DEV	SUPELCO LOT NO
2-CHLOROETHYL VINYL ETHER	110-75-8	99.9	5000	5000	+/- 55.9	LB01239

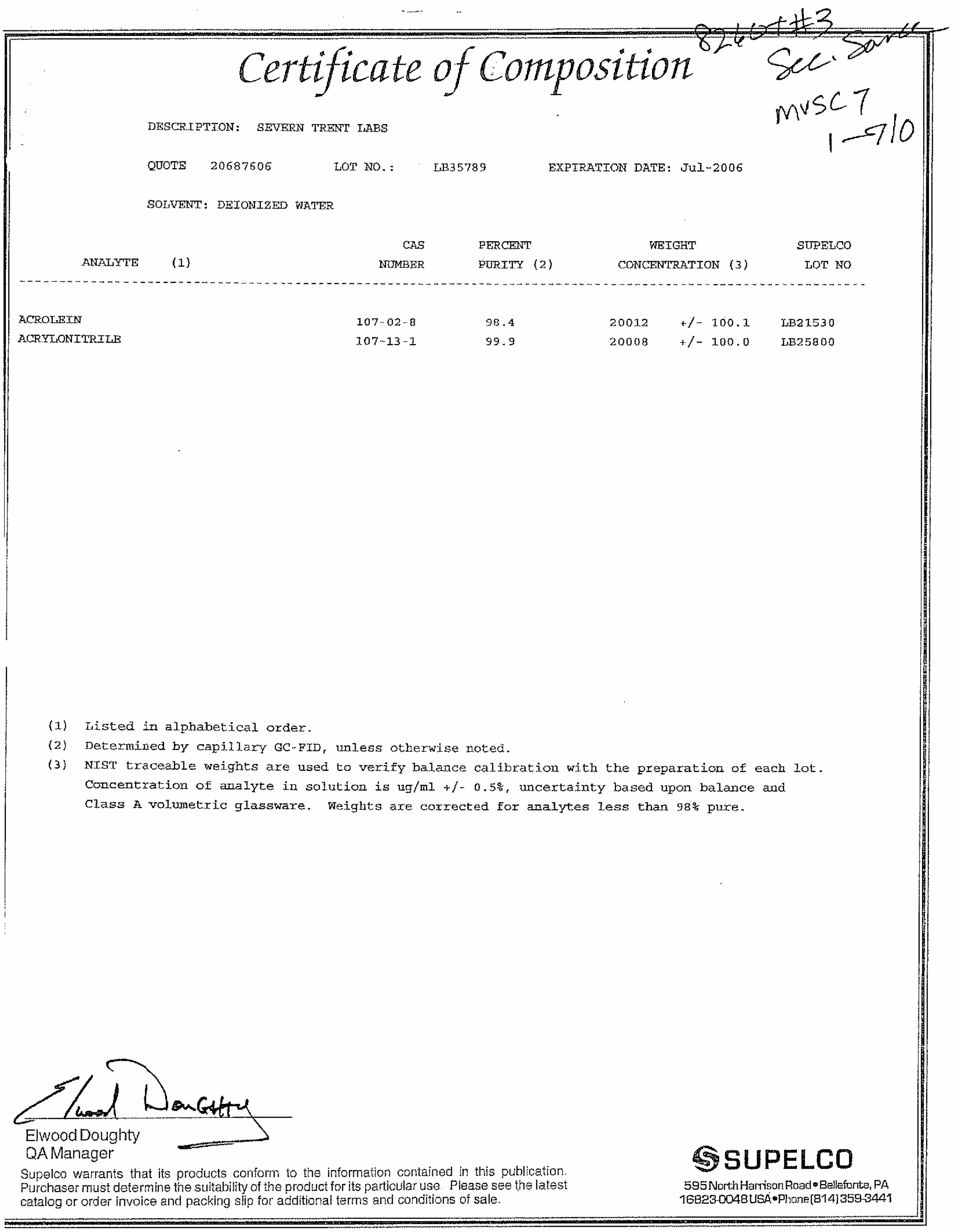
(1) Determined by capillary GC-FID, unless otherwise noted.

(2) NIST traceable weights are used to verify balance calibration with the preparation of each lot. Concentration of analyte in solution is ug/ml +/- 0.5%, uncertainty based upon balance and Class A volumetric glassware. Weights are corrected for analytes less than 98% pure.

(3) Determined by chromatographic analysis against an independently prepared reference lot. Mean of replicate injections.

Elwood Doughty
Quality Control Supervisor

SUPELCO
595 North Harrison Road
Bellefonte, PA 16823-0048 USA
Phone (814) 359-3441



8260 #3 Sec. Source

Certificate of Composition

MVSC7
1-910

DESCRIPTION: SEVERN TRENT LABS

QUOTE 20687606 LOT NO.: LB35789 EXPIRATION DATE: Jul-2006

SOLVENT: DEIONIZED WATER

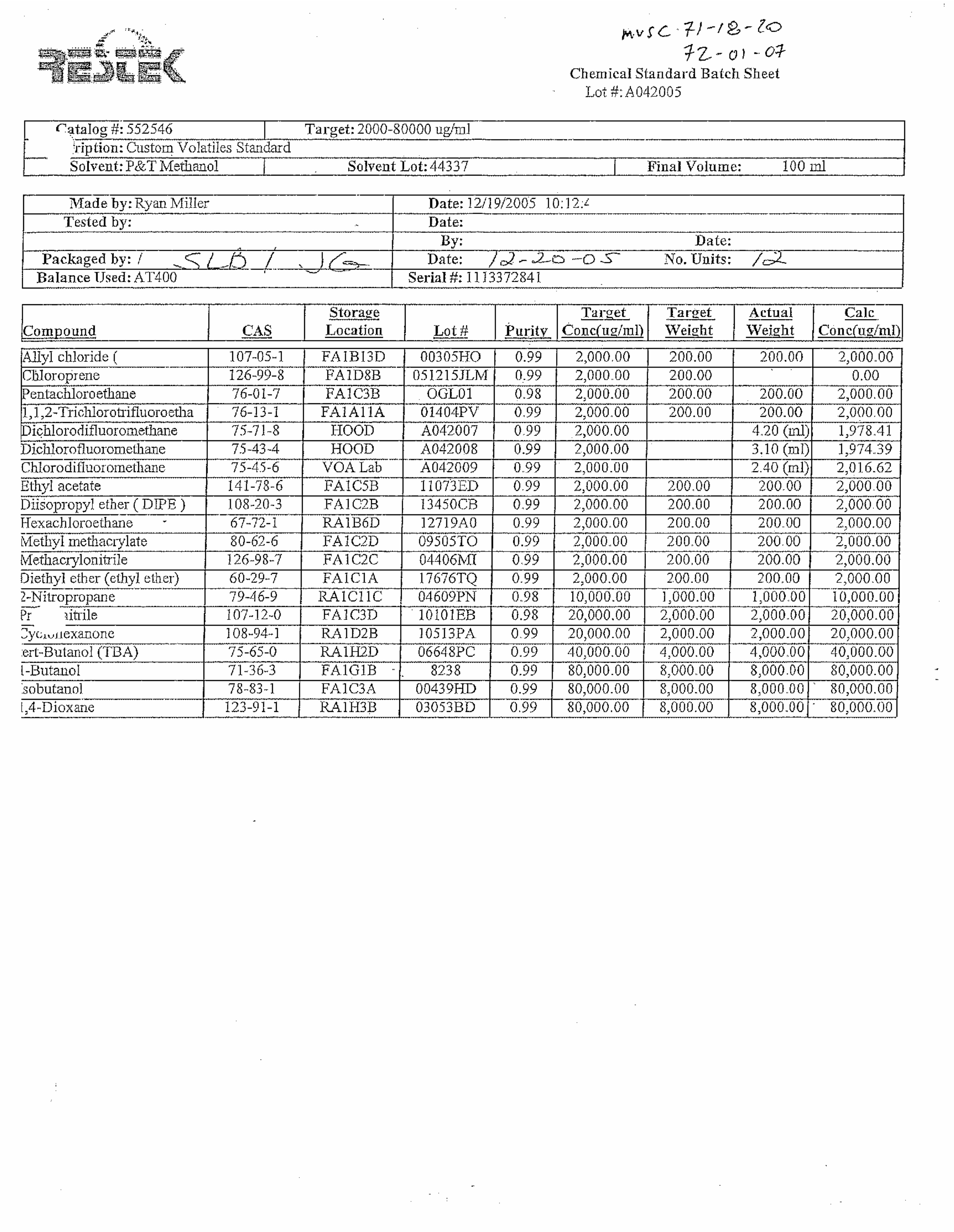
ANALYTE (1)	CAS NUMBER	PERCENT PURITY (2)	WEIGHT CONCENTRATION (3)	SUPELCO LOT NO
ACROLEIN	107-02-8	98.4	20012 +/- 100.1	LB21530
ACRYLONITRILE	107-13-1	99.9	20008 +/- 100.0	LB25800

(1) Listed in alphabetical order.
(2) Determined by capillary GC-FID, unless otherwise noted.
(3) NIST traceable weights are used to verify balance calibration with the preparation of each lot. Concentration of analyte in solution is ug/ml +/- 0.5%, uncertainty based upon balance and Class A volumetric glassware. Weights are corrected for analytes less than 98% pure.

Elwood Doughty
Elwood Doughty
QA Manager

SUPELCO
595 North Harrison Road • Bellefonte, PA
16823-0048 USA • Phone (814) 359-3441

Table 12. 8260 Add Mixture



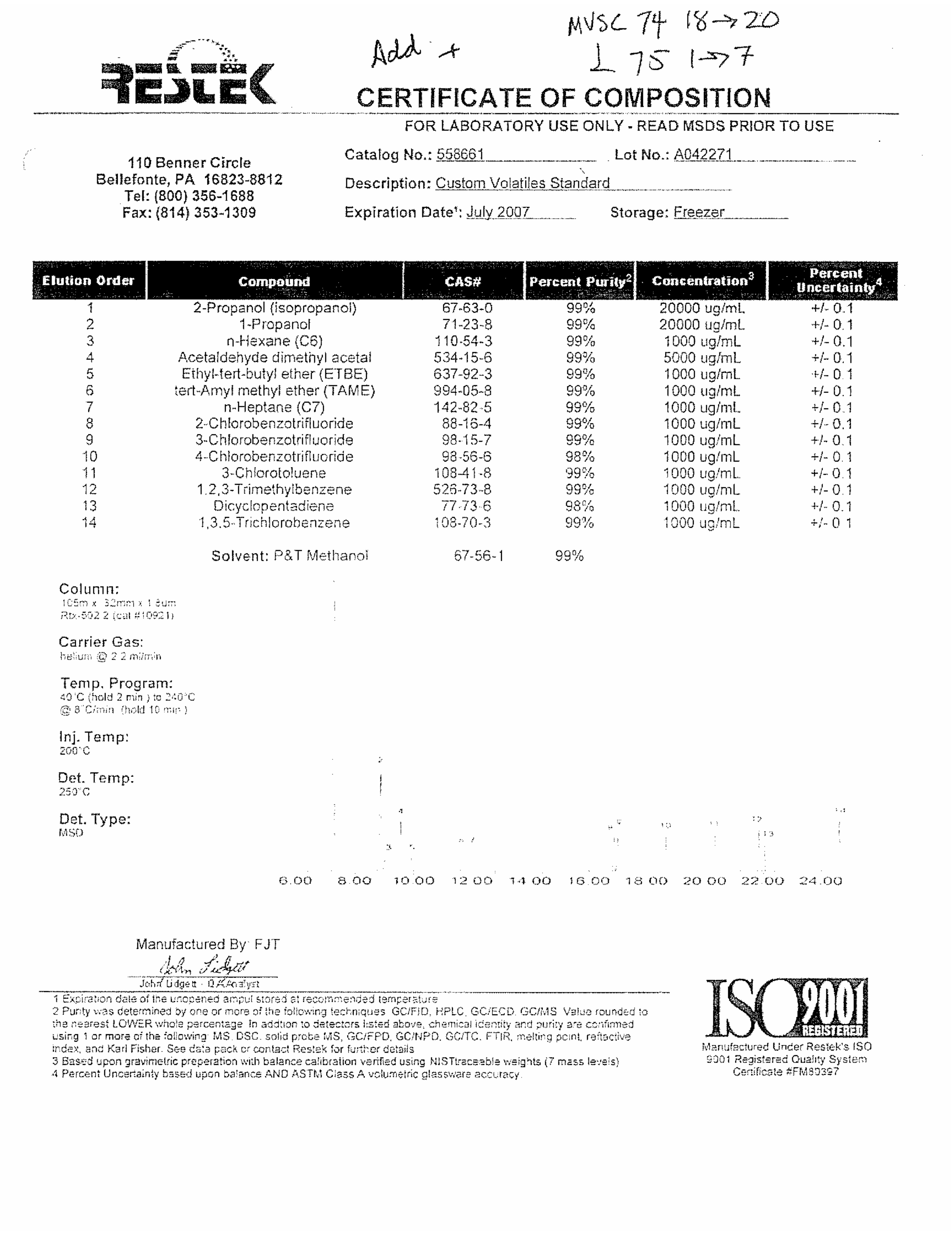
mvsc 71-18-20
72-01-07

Chemical Standard Batch Sheet
Lot #: A042005

Catalog #: 552546	Target: 2000-80000 ug/ml	
ription: Custom Volatiles Standard		
Solvent: P&T Methanol	Solvent Lot: 44337	Final Volume: 100 ml

Made by: Ryan Miller	Date: 12/19/2005 10:12:4	
Tested by:	Date:	
	By:	Date:
Packaged by: / SLB / JG	Date: 12-20-05	No. Units: 12
Balance Used: AT400	Serial #: 1113372841	

Compound	CAS	Storage Location	Lot #	Purity	Target Conc(ug/ml)	Target Weight	Actual Weight	Calc Conc(ug/ml)
Allyl chloride (	107-05-1	FA1B13D	00305HO	0.99	2,000.00	200.00	200.00	2,000.00
Chloroprene	126-99-8	FA1D8B	051215JLM	0.99	2,000.00	200.00		0.00
Pentachloroethane	76-01-7	FA1C3B	OGL01	0.98	2,000.00	200.00	200.00	2,000.00
1,1,2-Trichlorotrifluoroetha	76-13-1	FA1A11A	01404PV	0.99	2,000.00	200.00	200.00	2,000.00
Dichlorodifluoromethane	75-71-8	HOOD	A042007	0.99	2,000.00		4.20 (ml)	1,978.41
Dichlorofluoromethane	75-43-4	HOOD	A042008	0.99	2,000.00		3.10 (ml)	1,974.39
Chlorodifluoromethane	75-45-6	VOA Lab	A042009	0.99	2,000.00		2.40 (ml)	2,016.62
Ethyl acetate	141-78-6	FA1C5B	11073ED	0.99	2,000.00	200.00	200.00	2,000.00
Diisopropyl ether (DIPE)	108-20-3	FA1C2B	13450CB	0.99	2,000.00	200.00	200.00	2,000.00
Hexachloroethane	67-72-1	RA1B6D	12719A0	0.99	2,000.00	200.00	200.00	2,000.00
Methyl methacrylate	80-62-6	FA1C2D	09505TO	0.99	2,000.00	200.00	200.00	2,000.00
Methacrylonitrile	126-98-7	FA1C2C	04406MI	0.99	2,000.00	200.00	200.00	2,000.00
Diethyl ether (ethyl ether)	60-29-7	FA1C1A	17676TQ	0.99	2,000.00	200.00	200.00	2,000.00
2-Nitropropane	79-46-9	RA1C11C	04609PN	0.98	10,000.00	1,000.00	1,000.00	10,000.00
Pr itrile	107-12-0	FA1C3D	10101EB	0.98	20,000.00	2,000.00	2,000.00	20,000.00
Cyclohexanone	108-94-1	RA1D2B	10513PA	0.99	20,000.00	2,000.00	2,000.00	20,000.00
ert-Butanol (TBA)	75-65-0	RA1H2D	06648PC	0.99	40,000.00	4,000.00	4,000.00	40,000.00
1-Butanol	71-36-3	FA1G1B	8238	0.99	80,000.00	8,000.00	8,000.00	80,000.00
sobutanol	78-83-1	FA1C3A	00439HD	0.99	80,000.00	8,000.00	8,000.00	80,000.00
1,4-Dioxane	123-91-1	RA1H3B	03053BD	0.99	80,000.00	8,000.00	8,000.00	80,000.00



Add + MVSC 74 18→20
L 75 1→7

CERTIFICATE OF COMPOSITION

FOR LABORATORY USE ONLY - READ MSDS PRIOR TO USE

110 Benner Circle
Bellefonte, PA 16823-8812
Tel: (800) 356-1688
Fax: (814) 353-1309

Catalog No.: 558661 Lot No.: A042271

Description: Custom Volatiles Standard

Expiration Date[1]: July 2007 Storage: Freezer

Elution Order	Compound	CAS#	Percent Purity[2]	Concentration[3]	Percent Uncertainty[4]
1	2-Propanol (isopropanol)	67-63-0	99%	20000 ug/mL	+/- 0.1
2	1-Propanol	71-23-8	99%	20000 ug/mL	+/- 0.1
3	n-Hexane (C6)	110-54-3	99%	1000 ug/mL	+/- 0.1
4	Acetaldehyde dimethyl acetal	534-15-6	99%	5000 ug/mL	+/- 0.1
5	Ethyl-tert-butyl ether (ETBE)	637-92-3	99%	1000 ug/mL	+/- 0.1
6	tert-Amyl methyl ether (TAME)	994-05-8	99%	1000 ug/mL	+/- 0.1
7	n-Heptane (C7)	142-82-5	99%	1000 ug/mL	+/- 0.1
8	2-Chlorobenzotrifluoride	88-16-4	99%	1000 ug/mL	+/- 0.1
9	3-Chlorobenzotrifluoride	98-15-7	99%	1000 ug/mL	+/- 0.1
10	4-Chlorobenzotrifluoride	98-56-6	98%	1000 ug/mL	+/- 0.1
11	3-Chlorotoluene	108-41-8	99%	1000 ug/mL	+/- 0.1
12	1,2,3-Trimethylbenzene	526-73-8	99%	1000 ug/mL	+/- 0.1
13	Dicyclopentadiene	77-73-6	98%	1000 ug/mL	+/- 0.1
14	1,3,5-Trichlorobenzene	108-70-3	99%	1000 ug/mL	+/- 0.1
	Solvent: P&T Methanol	67-56-1	99%		

Column:
105m x .32mm x 1.8um
Rtx-502.2 (cat #10921)

Carrier Gas:
helium @ 2.2 ml/min

Temp. Program:
40°C (hold 2 min.) to 240°C
@ 8°C/min. (hold 10 min.)

Inj. Temp:
200°C

Det. Temp:
250°C

Det. Type:
MSD

Manufactured By: FJT

John Lidgett
John Lidgett - QA Analyst

---

[1] Expiration date of the unopened ampul stored at recommended temperature

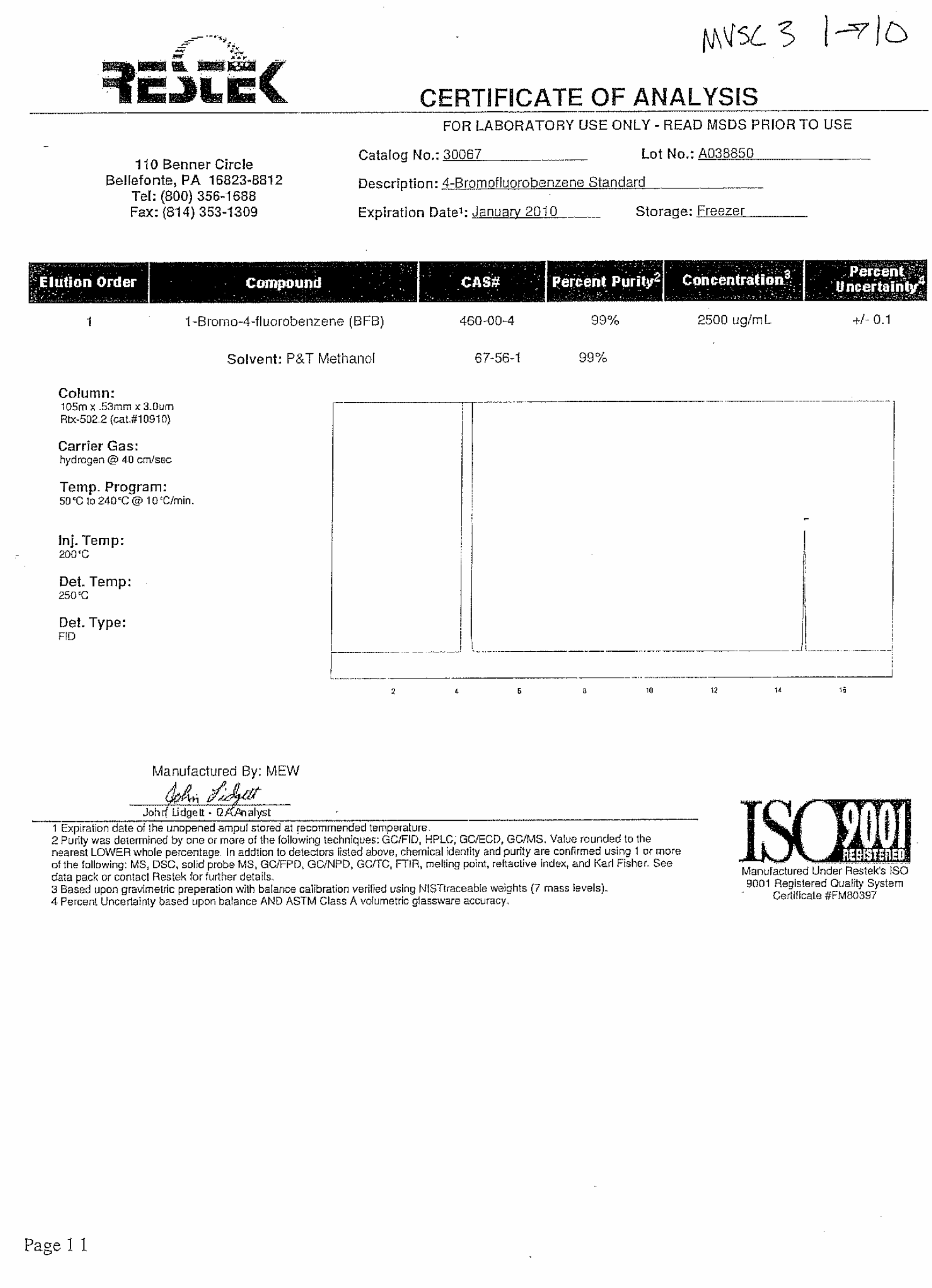
[2] Purity was determined by one or more of the following techniques: GC/FID, HPLC, GC/ECD, GC/MS. Value rounded to the nearest LOWER whole percentage. In addtion to detectors listed above, chemical identity and purity are confirmed using 1 or more of the following: MS, DSC, solid probe MS, GC/FPD, GC/NPD, GC/TC, FTIR, melting point, reftactive index, and Karl Fisher. See data pack or contact Restek for further details

[3] Based upon gravimetric preperation with balance calibration verified using NISTtraceable weights (7 mass levels)

[4] Percent Uncertainty based upon balance AND ASTM Class A volumetric glassware accuracy.

ISO 9001 REGISTERED
Manufactured Under Restek's ISO 9001 Registered Quality System Certificate #FM80397

Table 13. BFB Standard



MVSC 3 1-7-10

RESTEK

CERTIFICATE OF ANALYSIS

FOR LABORATORY USE ONLY - READ MSDS PRIOR TO USE

110 Benner Circle
Bellefonte, PA 16823-8812
Tel: (800) 356-1688
Fax: (814) 353-1309

Catalog No.: 30067 **Lot No.:** A038850

Description: 4-Bromofluorobenzene Standard

Expiration Date[1]**:** January 2010 **Storage:** Freezer

Elution Order	Compound	CAS#	Percent Purity[2]	Concentration[3]	Percent Uncertainty[4]
1	1-Bromo-4-fluorobenzene (BFB)	460-00-4	99%	2500 ug/mL	+/- 0.1
	Solvent: P&T Methanol	67-56-1	99%		

Column:
105m x .53mm x 3.0um
Rtx-502.2 (cat.#10910)

Carrier Gas:
hydrogen @ 40 cm/sec

Temp. Program:
50°C to 240°C @ 10°C/min.

Inj. Temp:
200°C

Det. Temp:
250°C

Det. Type:
FID

Manufactured By: MEW

John Lidgett
John Lidgett - QA Analyst

---

[1] Expiration date of the unopened ampul stored at recommended temperature.

[2] Purity was determined by one or more of the following techniques: GC/FID, HPLC, GC/ECD, GC/MS. Value rounded to the nearest LOWER whole percentage. In addtion to detectors listed above, chemical identity and purity are confirmed using 1 or more of the following: MS, DSC, solid probe MS, GC/FPD, GC/NPD, GC/TC, FTIR, melting point, reftactive index, and Karl Fisher. See data pack or contact Restek for further details.

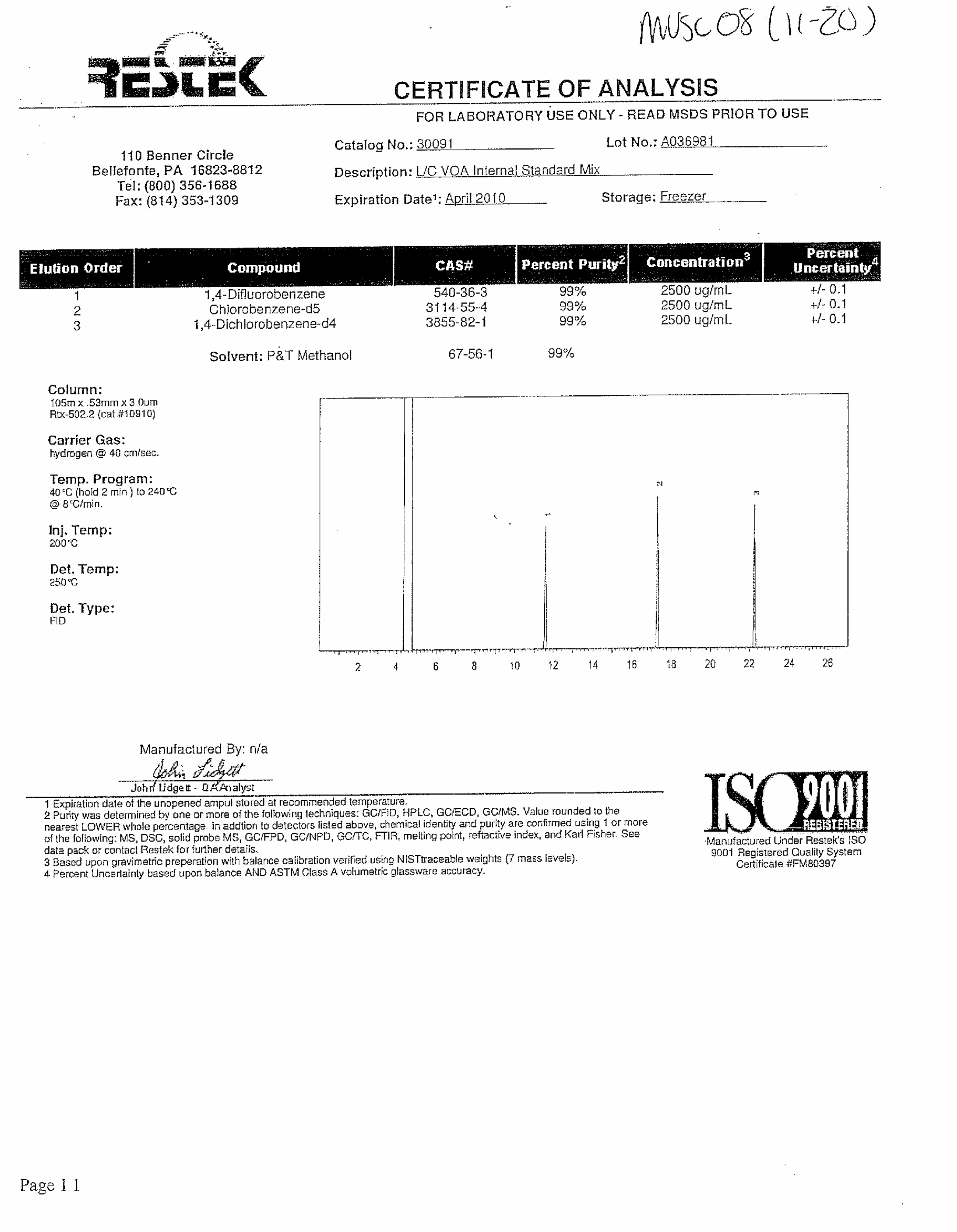
[3] Based upon gravimetric preperation with balance calibration verified using NISTtraceable weights (7 mass levels).

[4] Percent Uncertainty based upon balance AND ASTM Class A volumetric glassware accuracy.

ISO 9001 REGISTERED
Manufactured Under Restek's ISO 9001 Registered Quality System
Certificate #FM80397

Page 1 1

Table 14. Internal Standard Mixture



MUSC08 (11-20)

RESTEK

CERTIFICATE OF ANALYSIS

FOR LABORATORY USE ONLY - READ MSDS PRIOR TO USE

110 Benner Circle
Bellefonte, PA 16823-8812
Tel: (800) 356-1688
Fax: (814) 353-1309

Catalog No.: 30091
Lot No.: A036981
Description: L/C VOA Internal Standard Mix
Expiration Date[1]: April 2010
Storage: Freezer

Elution Order	Compound	CAS#	Percent Purity[2]	Concentration[3]	Percent Uncertainty[4]
1	1,4-Difluorobenzene	540-36-3	99%	2500 ug/mL	+/- 0.1
2	Chlorobenzene-d5	3114-55-4	99%	2500 ug/mL	+/- 0.1
3	1,4-Dichlorobenzene-d4	3855-82-1	99%	2500 ug/mL	+/- 0.1
	Solvent: P&T Methanol	67-56-1	99%		

Column:
105m x .53mm x 3.0um
Rtx-502.2 (cat #10910)

Carrier Gas:
hydrogen @ 40 cm/sec.

Temp. Program:
40°C (hold 2 min) to 240°C
@ 8°C/min.

Inj. Temp:
200°C

Det. Temp:
250°C

Det. Type:
FID

Manufactured By: n/a

John Lidgett
John Lidgett - QA Analyst

---

[1] Expiration date of the unopened ampul stored at recommended temperature.

[2] Purity was determined by one or more of the following techniques: GC/FID, HPLC, GC/ECD, GC/MS. Value rounded to the nearest LOWER whole percentage. In addtion to detectors listed above, chemical identity and purity are confirmed using 1 or more of the following: MS, DSC, solid probe MS, GC/FPD, GC/NPD, GC/TC, FTIR, melting point, reftactive index, and Karl Fisher. See data pack or contact Restek for further details.

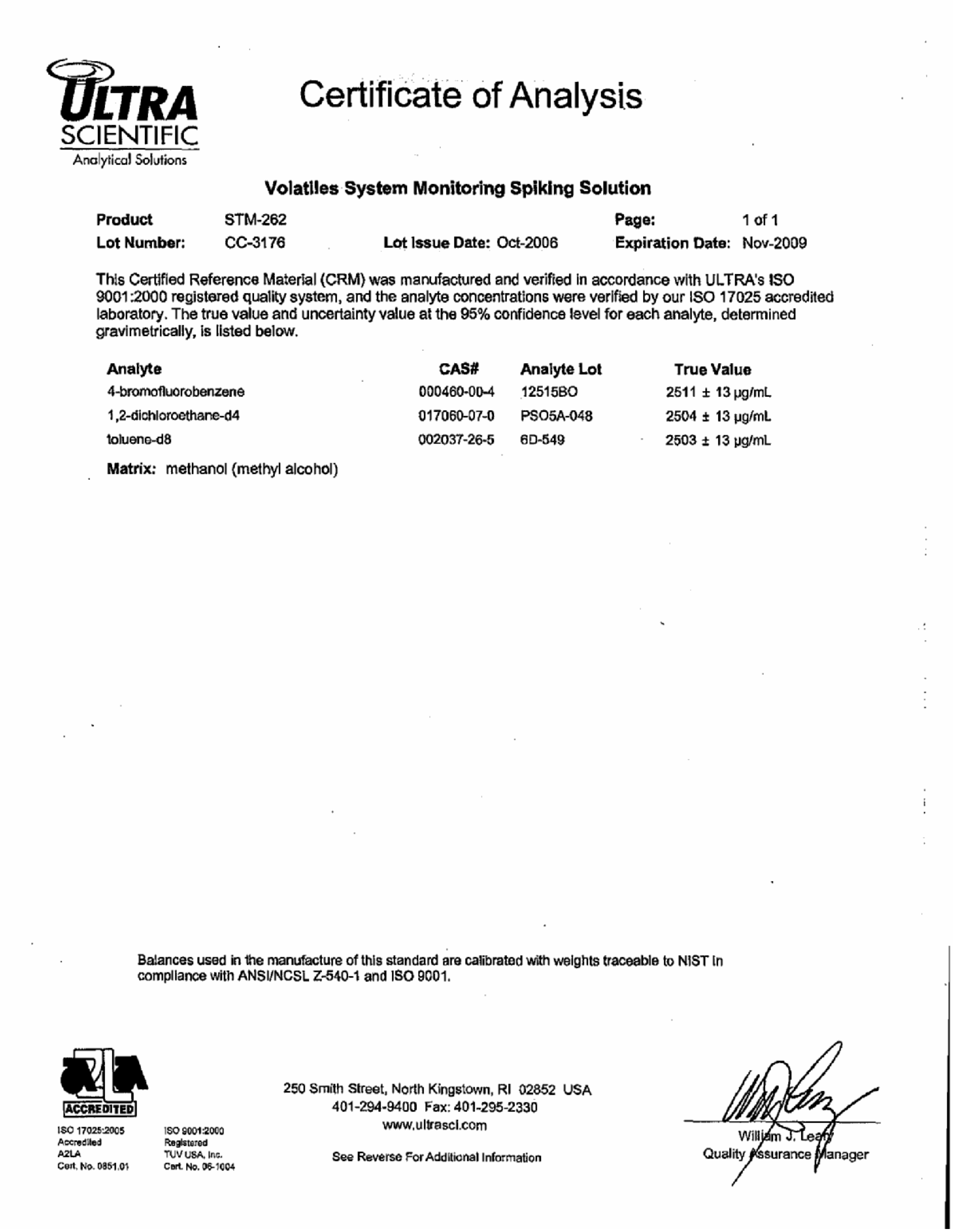
[3] Based upon gravimetric preperation with balance calibration verified using NISTtraceable weights (7 mass levels).

[4] Percent Uncertainty based upon balance AND ASTM Class A volumetric glassware accuracy.

ISO 9001 REGISTERED
Manufactured Under Restek's ISO 9001 Registered Quality System Certificate #FM80397

Page 1 1

Table 15. Surrogate Mixture



ULTRA SCIENTIFIC
Analytical Solutions

Certificate of Analysis

Volatiles System Monitoring Spiking Solution

Product	STM-262			**Page:**	1 of 1
Lot Number:	CC-3176	**Lot Issue Date:**	Oct-2006	**Expiration Date:**	Nov-2009

This Certified Reference Material (CRM) was manufactured and verified in accordance with ULTRA's ISO 9001:2000 registered quality system, and the analyte concentrations were verified by our ISO 17025 accredited laboratory. The true value and uncertainty value at the 95% confidence level for each analyte, determined gravimetrically, is listed below.

Analyte	CAS#	Analyte Lot	True Value
4-bromofluorobenzene	000460-00-4	12515BO	2511 ± 13 µg/mL
1,2-dichloroethane-d4	017060-07-0	PSO5A-048	2504 ± 13 µg/mL
toluene-d8	002037-26-5	6D-549	2503 ± 13 µg/mL

Matrix: methanol (methyl alcohol)

Balances used in the manufacture of this standard are calibrated with weights traceable to NIST in compliance with ANSI/NCSL Z-540-1 and ISO 9001.

ACCREDITED

ISO 17025:2005
Accredited
A2LA
Cert. No. 0851.01

ISO 9001:2000
Registered
TUV USA, Inc.
Cert. No. 06-1004

250 Smith Street, North Kingstown, RI 02852 USA
401-294-9400 Fax: 401-295-2330
www.ultrasci.com

See Reverse For Additional Information

William J. Leahy
Quality Assurance Manager

Nitrate+Nitrite Nitrogen, Nitrite Nitrogen and Nitrate Nitrogen Method 353.2, Standard Method 4500-NO_3, Lachat Method 10-107-04-1-C

Once printed, this is considered an uncontrolled document

Approvals (Signature/Date):

Lisa R Matecki 01/27/10
Lisa Matecki Date
Department Manager

Kenneth E. Kasperek 01/27/10
Kenneth Kasperek Date
Health & Safety Manager / Coordinator

Peggy Gray-Erdmann 01/27/10
Peggy Gray-Erdmann Date
Quality Assurance Manager

01/27/10
Christopher Spencer Date
Laboratory Director

Facility Distribution No. ___________ **Distributed To:**________________________

1.0 Scope and Application

1.1 Analytes, Matrix(s), and Reporting Limits

This method is used for the determination of nitrate and nitrite (singly or total) in drinking, surface and saline waters and domestic and industrial wastes. Soils can be analyzed using ASTM Method D 3987, “Shake Extraction of Solid Waste with Water”. The reporting limit has been determined to be 0.05 mg/l for aqueous samples and 1.0 mg/kg for soils subjected to the ASTM leaching procedure prior to final analysis.

2.0 Summary of Method

Nitrate is reduced quantitatively to nitrite in the presence of cadmium. The nitrite thus formed plus any originally present in the sample is determined as an azo dye at 520 nm following its diazotization with sulfanilamide and subsequent coupling with N(-1-naphthyl)ethylenediamine dihydrochloride. Without the introduction of the sample to the cadmium column, nitrite singly is determined. A nitrate only value may be calculated by subtracting the nitrite from the Total nitrite/nitrate value.

3.0 Definitions

Standard definitions are used in this document as defined by the TestAmerica Corporate Quality Assurance Plan.

4.0 Interferences

4.1 Sample color, particulates or turbidity may interfere. Turbid samples or samples with suspended solids must be filtered prior to analysis through a 0.2 pore diameter filter.

4.2 Some metals in high concentration will cause various interferences. EDTA is added to eliminate these interferences.

4.3 Acidic samples are to be adjusted to a pH of 5 to 9 with a dilute solution of ammonium hydroxide.

4.4 Residual Chlorine can interfere by oxidizing the cadmium column and must be removed.

5.0 Safety

Employees must abide by the policies and procedures in the Corporate Safety Manual, Radiation Safety Manual and this document.

This procedure may involve hazardous material, operations and equipment. This SOP does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of the method to follow appropriate safety, waste disposal and health practices under the assumption that all samples and reagents are potentially hazardous. Safety glasses, gloves, lab coats and closed-toe, nonabsorbent shoes are a minimum.

5.1 Specific Safety Concerns or Requirements

None

5.2 Primary Materials Used

The following is a list of the materials used in this method, which have a serious or significant hazard rating. **Note: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the MSDS for each of the materials listed in the table.** A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the MSDS for each material before using it for the first time or when there are major changes to the MSDS.

Material [1]	**Hazards**	**Exposure Limit [2]**	**Signs and symptoms of exposure**
Ammonium Hydroxide	Corrosive Poison	50 ppm-TWA	Vapors and mists cause irritation to the respiratory tract. Causes irritation and burns to the skin and eyes.
Phosphoric Acid	Corrosive	1 Mg/M3 TWA	Inhalation is not an expected hazard unless misted or heated to high temperatures. May cause redness, pain, and severe skin burns. May cause redness, pain, blurred vision, eye burns, and permanent eye damage.
1 – Always add acid to water to prevent violent reactions.			
2 – Exposure limit refers to the OSHA regulatory exposure limit.			

6.0 Equipment and Supplies

6.1 Instrumentation

- LACHAT autoanalyzer equipped with nitrate/nitrite manifold and cadmium column. The cadmium column is a disposable acrylic column packed with cadmium and properly sealed on either end to prevent leaks and water evaporation during storage.

6.2 Supplies

- Class A volumetric and graduated glassware
- Calibrated eppendorfs.
- Miscellaneous disposable supplies, such as culture tubes, pipets, parafilm, etc.

7.0 Reagents and Standards

7.1 All chemicals shall conform to American Chemical Society specifications or equivalent.

7.2 Reagent Water

7.3 Concentrated Ammonium Hydroxide

7.4 1:4 Ammonium Hydroxide Solution (200 ml): Add 50 ml of concentrated ammonium hydroxide to 150 ml of reagent water and mix well. This has a shelf life of six months.

7.5 Stock Ammonium Chloride-EDTA Buffer Solution (2 L): Dissolve 170 g of ammonium chloride and 2.0 g of disodium EDTA in about 1800 ml of reagent water. Adjust the pH to 8.5 with Concentrated NH_4OH. Dilute to 2000 ml with reagent water and mix well. This has a shelf life of one month.

7.6 Concentrated Phosphoric Acid

7.7 Color Reagent (1 L): While stirring, add 100 mL of conc. H_3PO_4 to about 700 mL of reagent water. Dissolve 40 g of sulfanilamide and 1 g of N(-1-naphthyl)ethylenediamine dihydrochloride in the acid solution. Dilute to 1 L with reagent water and mix well. Store reagent in an amber bottle and keep in the dark when not in use. This reagent is stable for one month.

7.8 Sampler Wash Solution: Reagent water.

7.9 Stock 1000 mg/L Nitrate Nitrogen Standard purchased from two separate vendors.

7.9.1. Intermediate 100 mg/L Nitrate Nitrogen Solution (100 ml): Add 5.0 ml of the 1st source stock nitrate nitrogen solution (1000 mg/L) to about 30 ml of reagent water. Dilute to 50 ml with reagent water and mix well. This solution is to be prepared weekly.

7.9.1.1 The calibration curve and matrix spike solution will be prepared from the primary source

7.9.2 Nitrate ICV/CCV 1.5 mg/L: Add 0.15 ml of the 2nd source stock nitrate nitrogen solution (100 mg/L) to about 80 ml of reagent water. Dilute to 100 ml with reagent water and mix well. This solution is to be prepared daily. This solution is used to check the reduction efficiency of the cadmium column and as the continuing quality control check for Nitrates.

7.9.3 Prepare Nitrate calibration standards by adding the appropriate amount of intermediate standard nitrate nitrogen solution (100 mg/L) (see table below for recommended calibrants) to about 80 ml of reagent water. Dilute to 100 ml with reagent water and mix well. These solutions are to be prepared once a month.

7.10 Stock 1000 mg/L Nitrite Nitrogen Standard purchased from two separate vendors.

7.10.2 Intermediate 100 mg/L Nitrite Nitrogen Solution (100 ml): Add 5.0 ml of the 1st source stock nitrite nitrogen solution (1000 mg/L) to about 30 ml of reagent water. Dilute to 50 ml with reagent water and mix well. This solution is to be prepared weekly.

7.10.2.1 The calibration curve and matrix spike solution will be prepared from the primary source.

7.10.3 Nitrite ICV/CCV 1.5 mg/L: Add 0.15 ml of the 2nd source stock nitrite nitrogen solution (100 mg/L) to about 80 ml of reagent water. Dilute to 100 ml with reagent water and mix well. This solution is to be prepared daily. This solution is used to check the reduction efficiency of the cadmium column on the NO2-NO3 analytical batch.

7.10.4 Prepare Nitrite calibration standards by adding the appropriate amount of intermediate standard nitrite nitrogen solution (100 mg/L) (see table below for recommended calibrants) to about 80 ml of reagent water. Dilute to 100 ml with reagent water and mix well. These solutions are to be prepared monthly.

CALIBRANTS

Nitrate or Nitrite Calibrant Concentration (mg/L)	Volume of Intermediate Solution (uL)
0 (blank)	0
0.050	5
0.20	20
0.50	50
1.0	100
2.0	200
3.0	300

7.11 Sodium Thiosulfate solution (Dechlorinating solution): Dissolve 3.5 g sodium thiosulfate (Na2S2O3·5H2O) in water and dilute to 1 Liter. Prepare fresh weekly. Use 1ml reagent to remove 1mg/l residual chlorine in 500 ml sample.

8.0 Sample Collection, Preservation, Shipment and Storage

Sample container, preservation techniques and holding times may vary and are dependent on sample matrix, method of choice, regulatory compliance, and/or specific contract or client requests. Listed below are the holding times and the references that include preservation requirements.

Matrix	Sample Container	Min. Sample Size	Preservation	Holding Time	Reference
Waters	Glass or Plastic	200mL	Cool 4 $\pm$ 2^{o}C	2 Days	40 CFR Part 136.3 SM 4500-NO_3
Waters	Glass or Plastic	200mL	H_2SO_4 <2 Cool 4 $\pm$ 2^{o}C	28 Days	40 CFR Part 136.3 SM 4500-NO_3
Soils	Glass or Plastic	50g	Cool 4 $\pm$ 2^{o}C	14 Days	N/A

9.0 Quality Control

9.1 Initial Calibration Verification (ICV) and Continuing Calibration Verification (CCV): The ICV is prepared from a source other than that used to prepare the calibration curve and is analyzed at start of run. The CCVs are analyzed after every 10 samples and at the end of the analytical sequence. The ICV/CCV is equivalent to an LCS for this method.

Quality Controls	Frequency	Control Limit
Method Blank (MBLK)	1 in 10 or fewer samples	< Rpt. Limit
Laboratory Control Sample (LCS)	1 in 10 or fewer samples	Statistical Limits [3]
Matrix Duplicate (MD)[1]	1 in 20 or fewer samples	Statistical Limits [3]

Quality Controls	Frequency	Control Limit
Matrix Spike (MS)[1]	1 in 20 or fewer samples	Statistical Limits [3]

[1] The sample selection for MD/MS is randomly selected, unless specifically requested by a client.
[2] Analytical and QC samples (MB, LCS, MD/MS)
[3] Statistical control limits are updated annually and are updated into LIMS.

9.1.1 The results of the analyses of the ICV and CCVs must be within +/-10% of the true value. If unacceptable results are obtained, all samples analyzed since the last acceptable CCV must be re-analyzed.

9.2 An Initial Calibration Blank (ICB) is analyzed after the ICV and a Continuing Calibration Blank (CCB) is analyzed with each analytical batch of 10 samples or less and is carried through the entire analytical procedure. These blanks must not exhibit Nitrate (or Nitrite) at concentrations greater than the TestAmerica Buffalo quantitation limit. If unacceptable results are achieved, all samples associated with that blank must be re-analyzed.

9.3 Matrix Spike (MS) and Matrix Duplicate (MD): A MS/MD set is performed for each sample batch or once every 20 samples, whichever is more frequent. One of the samples in the batch is prepared in triplicate, with the second and third aliquots being the MS and MD. The MS is fortified with Intermediate Nitrate Nitrogen Solution or Intermediate Nitrite Nitrogen Solution, depending on analyses performing. The MD is analyzed neat.

9.3.1 The calculated RPD of the sample and MD should be < 20%. If results fall outside of the QC limits but all other QC criteria for the analytical batch have been achieved, re-analysis may not be required.

9.3.2 Results of the MS analysis should be compared to the in-house % recovery limits. These limits are statistically derived based upon historical data and are updated annually. If the lab calculated limits are wider than the method limits, the method limits of 90-110% are used to evaluate matrix spike acceptance. The sample matrix may affect accuracy, therefore if results fall outside QC limits but all other QC criteria for the analytical batch have been achieved, re-analysis may not be required.

9.4 The acceptance criteria for the reduction efficiency for the cadmium column are 90-110%. If criteria are not met, a new column must be used. See Section 11.2 for calculation.

10.0 Procedure

10.1 Prepare reagents and standards as described in Section 7.0.

10.2 Set up manifold as shown in Attachment 17.1.

10.3 Pump DI water through all reagent lines and check for leaks and smooth flow. Switch to reagents and allow system to equilibrate.

10.4 Input the sample identification required by the data system. Before placing samples in the auto sampler, check all samples for residual chlorine. If samples test positive for residual chlorine remove by adding sodium thiosulfate solution (dechlorinating reagent 7.11). Use

1ml reagent to remove 1mg/l residual chlorine in 500 ml sample.

10.5 If samples are preserved, add 1:4 NH_4OH drop wise until a pH of 5-9 is achieved.

10.6 **Total Nitrate + Nitrite:** Connect cadmium column to manifold. The nitrate in the sample is reduced to nitrite as it passes through the cadmium column. Start instrument analysis, checking to be certain that baseline is steady, checking standards are compliant, and no further dilutions are required.

10.7 **Nitrite singly**: Disconnect cadmium column from manifold. The nitrite method is identical in setup to the nitrate/nitrite method, with the exception of the cadmium column. By not allowing the sample to pass through the cadmium column only nitrite values will be acquired.

10.8 **Nitrate singly:** Nitrate only values are calculated by subtracting the nitrite only value from the combined Nitrite + Nitrate value.

11.0 Calculations / Data Reduction

11.1 The concentration of each sample will be calculated by the program based on peak area from the calibration curve.

11.1.1 If nitrate nitrogen singly is to be determined, using the calculated concentrations of the total nitrate/nitrite nitrogen and nitrite nitrogen, then use the following formula:

$$NO_3^- \text{ singly} = [\text{Total } NO_3^- + NO_2^-] - [NO_2^- \text{ singly}]$$

11.2 The reduction efficiency of the cadmium column can be calculated by the following formula and should be within 90-100%, if not a new column must be used:

$$\% \text{ Reduction Efficiency} = \frac{NO_3^- \text{ peak height}}{NO_2^- \text{ peak height}} \text{ X } 100$$

11.3 Results must also be printed off showing area counts. To do this, the current curve must be deleted in the Review screen. After deleting the curve, hit the Analyze button. **Do not at any point save the method**. This will completely delete the current curve, and the instrument will need to be re-calibrated before the next run. Once you have printed area counts, exit out of the Omnion program, being sure to hit ***No***, when asked if you want to save any changes to the method.

12.0 Method Performance

12.1 The applicable range of the Nitrate and Nitrate+Nitrite method is 0.05 - 3.0 mg/L. The applicable range of the Nitrite method is 0.05 - 3.0 mg/L. The range can be extended by sample dilution.

12.2 As specified in EPA Method 353.2, Revision 2.0, method detection limit studies are performed every 6 months. The MDL determination process is performed in accordance with 40 CFR, part 136, Appendix B and must demonstrate the ability to report a detection limit of 0.05 mg/L.

13.0 Pollution Control

13.1 Waste Streams Produced by the Method: The following waste streams are produced when this method is carried out.

13.1.1 Acidic waste generated by the Lachat auto-analyzer. Dispose of this waste in the “A” waste container.

13.1.2 Acidic sample waste generated by sample preparation. Dispose of this waste in the “A” waste container.

13.1.3 Contaminated disposable glassware utilized for the analysis. Empty the contests of the glassware into the “A” waste and dispose of the glassware in the recycling bins located throughout the lab.

14.0 Waste Management

All waste will be disposed of in accordance with Federal, State and Local regulations. Where reasonably feasible, technological changes have been implemented to minimize the potential for pollution of the environment. Employees will abide by this method and the policies in section 13 of the Corporate Safety Manual for “Waste Management and Pollution Prevention.”

15.0 References / Cross-References

15.1 Lachat QuikChem® Method 10-107-04-1-C, Determination of Nitrate/Nitrite in surface and wastewaters by flow injection analysis, 1999.

15.2 Methods for Chemical Analysis of Water and Wastes - Method 353.2.

15.3 Standard Methods, 19th Edition, method 4500-NO3-F.

16.0 Method Modifications:

Item	Method	Modification
N/A	N/A	N/A.

17.0 Attachments

17.1 Nitrate/Nitrite Manifold Diagram
17.2 Data System Parameters for QuickChem 8000
17.3 Analytical Run Sequence
17.4 Analytical Batch

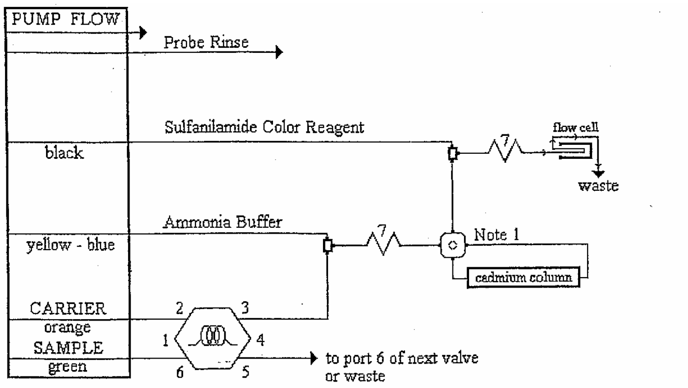
17.5 Wet Chemistry Batch Summary

18.0 Revision History

- Revision 0, dated 30 November 2007
    - Updated Attachments 17.4 and 17.5
    - Update Department manager and QA manager signatures

- Revision 1, January 27, 2010
    - Updated Attachments

Attachment 17.1
NITRATE/NITRITE MANIFOLD DIAGRAM



Sample Loop = 17 cm X 0.8 mm
QC8000 Sample Loop = 22.5 cm X 0.8 mm

Interference Filter = 520 nm

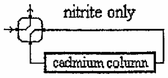
CARRIER is Helium Degassed DI water

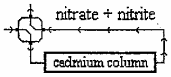
Manifold tubing is **0.8 mm (0.032 in)** i.d. This is 5.2 uL/cm.

7 is **135** cm of tubing on a 7 cm coil support

APPARATUS: An injection valve, a 10 mm path length flow cell , and a colorimetric detector module is required..

Note 1: This is a 2 state switching valve used to place the cadmium column in-line with the manifold.





Attachment 17.2

DATA SYSTEM PARAMETERS FOR QUIKCHEM 8000

The timing values listed below are approximate and will need to be optimized using graphical events programming.

Sample throughput:	55 samples/h, 65 s/sample
Pump speed:	35
Cycle Period:	65

Analyte Data:

Concentration Units:	mg N/L
Peak Base Width:	25 s
% Width Tolerance:	100
Threshold:	5000
Inject to Peak Start:	22 s
Chemistry:	Direct

Calibration Data:

Level	1	2	3	4	5	6	7
Concentration mg/L	2.00	0.80	0.20	0.05	0.02	0.01	0.00

Calibration Fit Type:	1st Order Polynomial
Calibration Rep. Handling:	Average
Weighting Method:	None
Concentration Scaling:	None
Force Through Zero:	No

Sampler Timing:

Min. Probe in Wash Period:	12 s
Probe in Sample Period:	32 s

Valve Timing:

Load Time:	0.0 s
Load Period:	28 s
Inject Period:	37 s

Attachment 17.3
Analytical Run Sequence

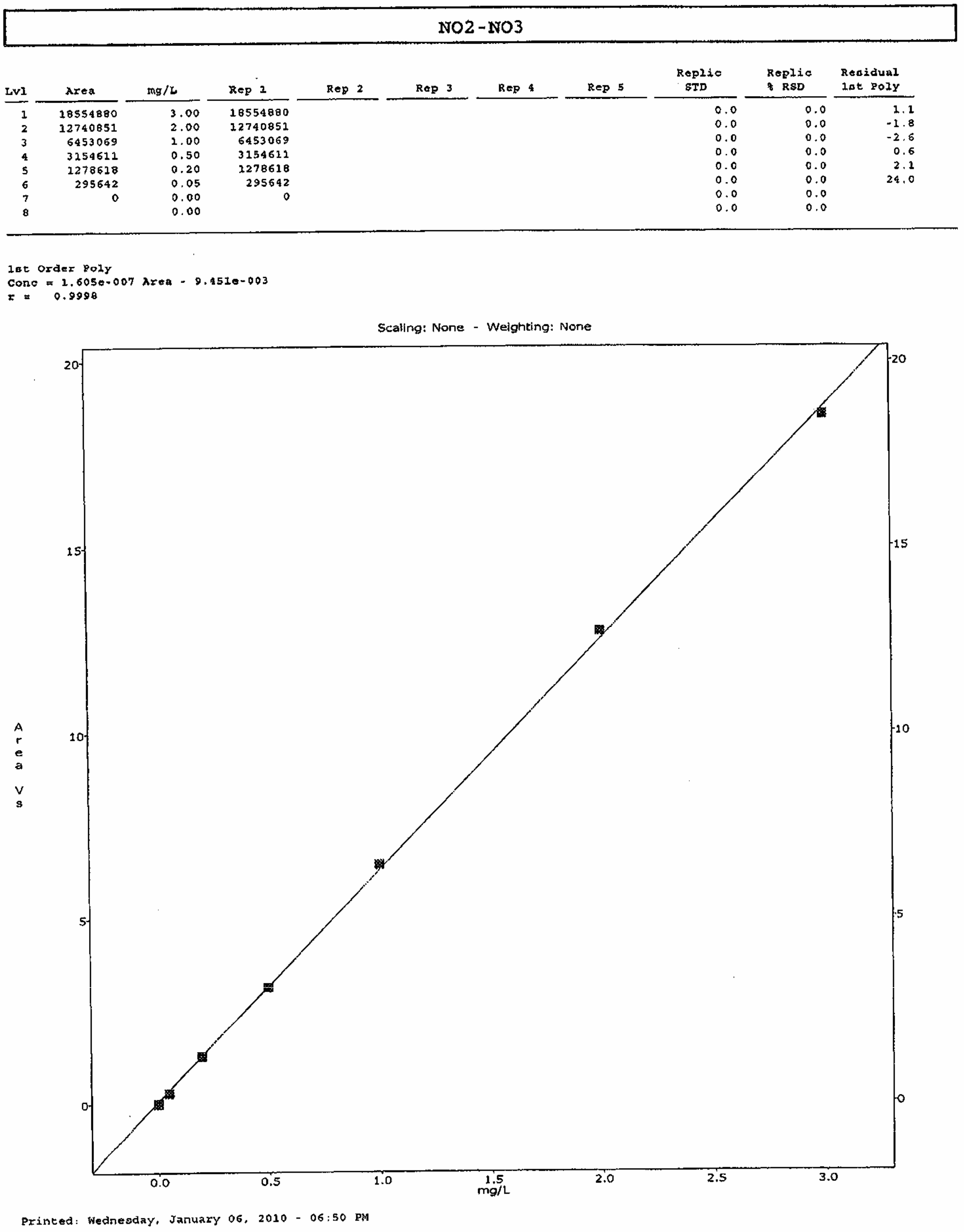
Total Nitrate / Nitrite sequence

LCS/CCV NO3 (1.5 mg/l)
LCS/CCV NO2(1.5 mg/l)
MBLK
Sample
Sample
Sample
Sample
Sample
Sample
Sample
Sample
Sample
Sample
CCV (1.5 mg/l)
CCB
Sample
Sample
Sample
Sample
Sample
Sample
Sample
Sample
Sample duplicate
Sample spike
CCV (1.5 mg/l)
CCB

Total Nitrite sequence

LCS/CCV NO2 (1.5 mg/l)
MBLK
Sample
Sample
Sample
Sample
Sample
Sample
Sample
Sample
Sample
Sample
CCV (1.5 mg/l)
CCB
Sample
Sample
Sample
Sample
Sample
Sample
Sample
Sample
Sample duplicate
Sample spike
CCV (1.5 mg/l)
CCB

Attachment 17.4
Analytical Batch



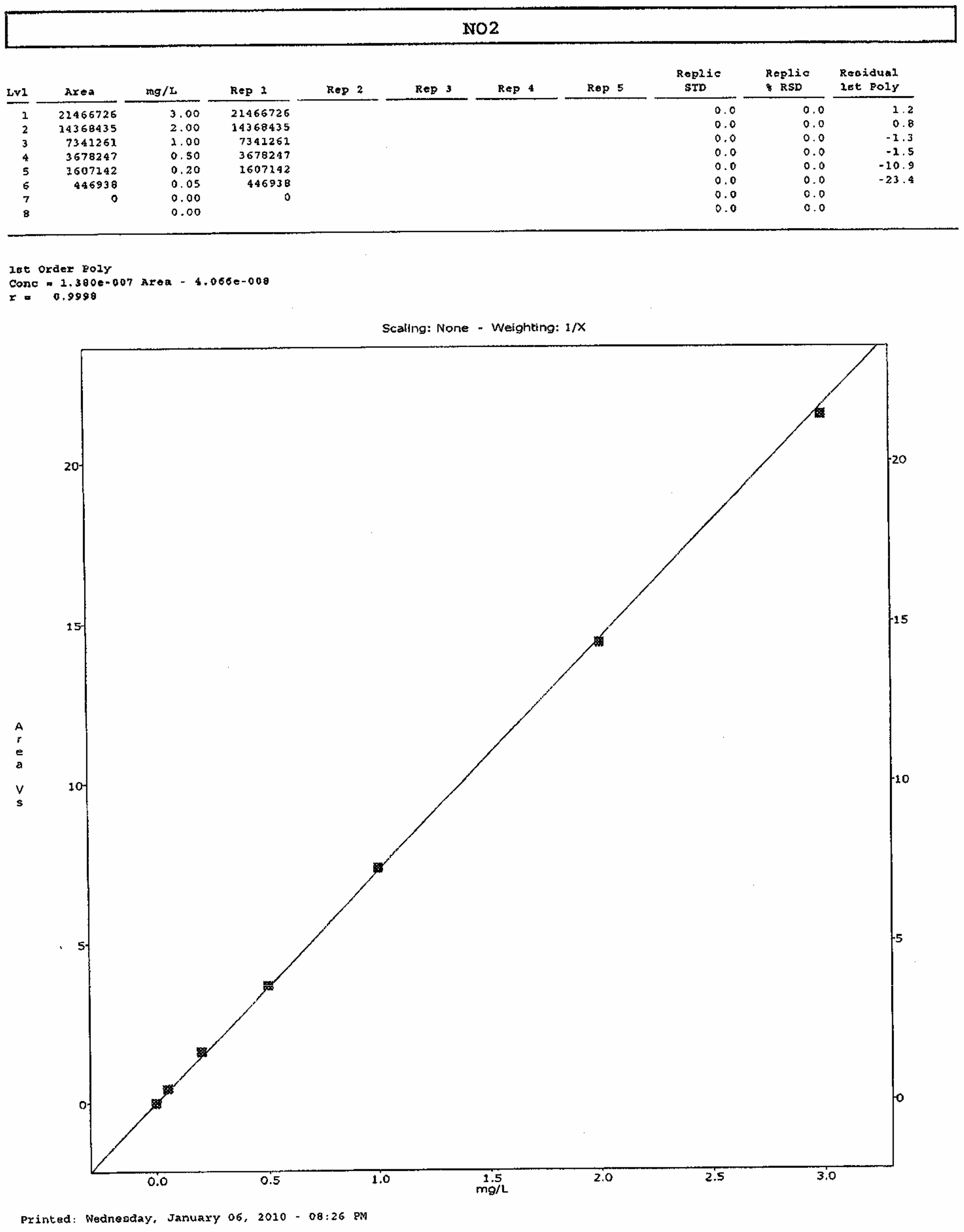
NO2-NO3

Lvl	Area	mg/L	Rep 1	Rep 2	Rep 3	Rep 4	Rep 5	Replic STD	Replic % RSD	Residual 1st Poly
1	18554880	3.00	18554880					0.0	0.0	1.1
2	12740851	2.00	12740851					0.0	0.0	-1.8
3	6453069	1.00	6453069					0.0	0.0	-2.6
4	3154611	0.50	3154611					0.0	0.0	0.6
5	1278618	0.20	1278618					0.0	0.0	2.1
6	295642	0.05	295642					0.0	0.0	24.0
7	0	0.00	0					0.0	0.0	
8		0.00						0.0	0.0	

1st Order Poly
Conc = 1.605e-007 Area - 9.451e-003
r = 0.9998

Scaling: None - Weighting: None

Printed: Wednesday, January 06, 2010 - 06:50 PM



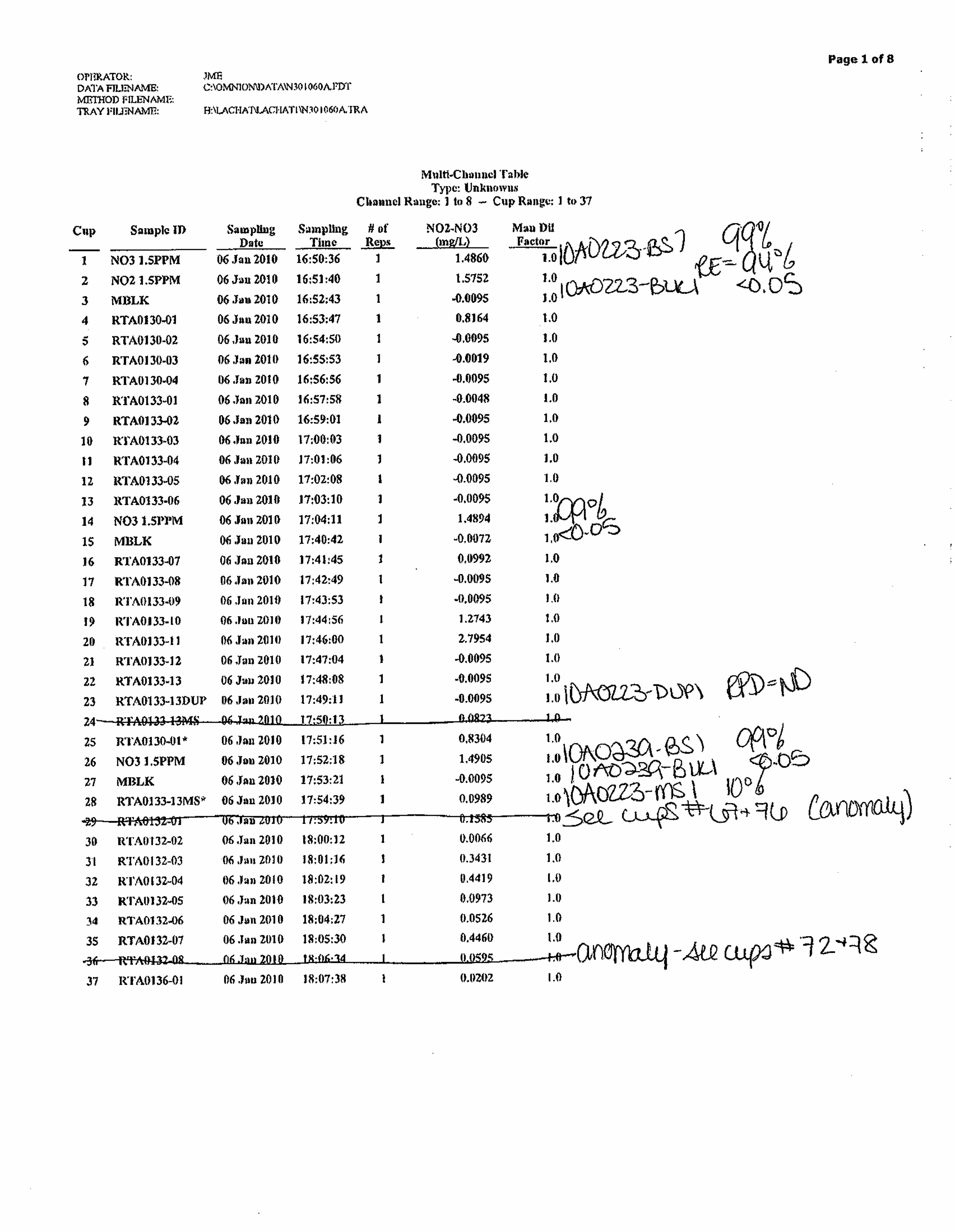
NO2

Lvl	Area	mg/L	Rep 1	Rep 2	Rep 3	Rep 4	Rep 5	Replic STD	Replic % RSD	Residual 1st Poly
1	21466726	3.00	21466726					0.0	0.0	1.2
2	14368435	2.00	14368435					0.0	0.0	0.8
3	7341261	1.00	7341261					0.0	0.0	-1.3
4	3678247	0.50	3678247					0.0	0.0	-1.5
5	1607142	0.20	1607142					0.0	0.0	-10.9
6	446938	0.05	446938					0.0	0.0	-23.4
7	0	0.00	0					0.0	0.0	
8		0.00						0.0	0.0	

1st Order Poly
Conc = 1.380e-007 Area - 4.066e-008
r =   0.9998

Scaling: None - Weighting: 1/X

Printed: Wednesday, January 06, 2010 - 08:26 PM



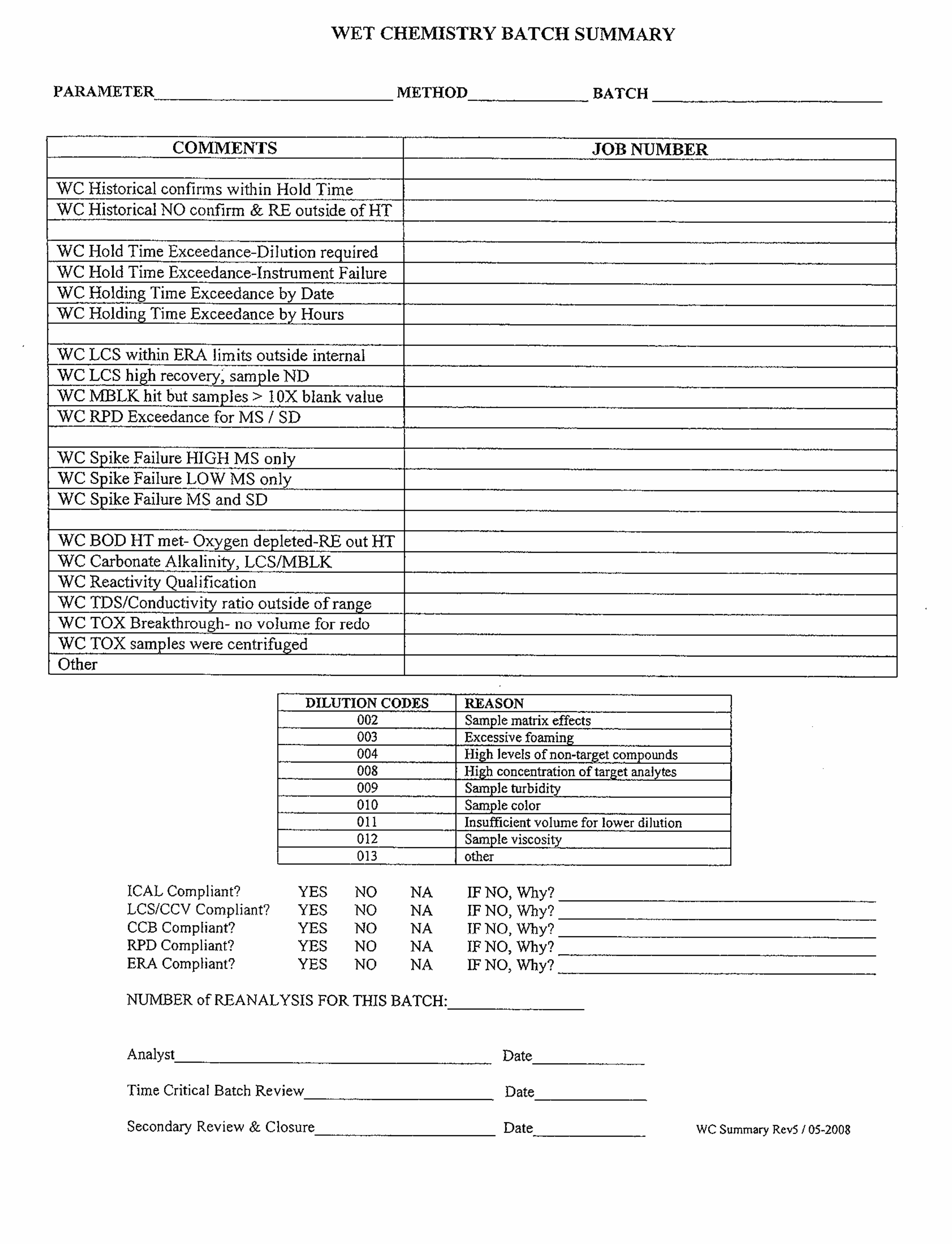
Page 1 of 8

OPERATOR: JME
DATA FILENAME: C:\OMNION\DATA\N301060A.FDT
METHOD FILENAME:
TRAY FILENAME: H:\LACHAT\LACHAT1\N301060A.TRA

Multi-Channel Table
Type: Unknowns
Channel Range: 1 to 8 -- Cup Range: 1 to 37

Cup	Sample ID	Sampling Date	Sampling Time	# of Reps	NO2-NO3 (mg/L)	Man Dil Factor	
1	NO3 1.5PPM	06 Jan 2010	16:50:36	1	1.4860	1.0	10A0223-BS1 99%
2	NO2 1.5PPM	06 Jan 2010	16:51:40	1	1.5752	1.0	RE=94%
3	MBLK	06 Jan 2010	16:52:43	1	-0.0095	1.0	10A0223-BLK1 <0.05
4	RTA0130-01	06 Jan 2010	16:53:47	1	0.8164	1.0	
5	RTA0130-02	06 Jan 2010	16:54:50	1	-0.0095	1.0	
6	RTA0130-03	06 Jan 2010	16:55:53	1	-0.0019	1.0	
7	RTA0130-04	06 Jan 2010	16:56:56	1	-0.0095	1.0	
8	RTA0133-01	06 Jan 2010	16:57:58	1	-0.0048	1.0	
9	RTA0133-02	06 Jan 2010	16:59:01	1	-0.0095	1.0	
10	RTA0133-03	06 Jan 2010	17:00:03	1	-0.0095	1.0	
11	RTA0133-04	06 Jan 2010	17:01:06	1	-0.0095	1.0	
12	RTA0133-05	06 Jan 2010	17:02:08	1	-0.0095	1.0	
13	RTA0133-06	06 Jan 2010	17:03:10	1	-0.0095	1.0	
14	NO3 1.5PPM	06 Jan 2010	17:04:11	1	1.4894	1.0	99%
15	MBLK	06 Jan 2010	17:40:42	1	-0.0072	1.0	<0.05
16	RTA0133-07	06 Jan 2010	17:41:45	1	0.0992	1.0	
17	RTA0133-08	06 Jan 2010	17:42:49	1	-0.0095	1.0	
18	RTA0133-09	06 Jan 2010	17:43:53	1	-0.0095	1.0	
19	RTA0133-10	06 Jan 2010	17:44:56	1	1.2743	1.0	
20	RTA0133-11	06 Jan 2010	17:46:00	1	2.7954	1.0	
21	RTA0133-12	06 Jan 2010	17:47:04	1	-0.0095	1.0	
22	RTA0133-13	06 Jan 2010	17:48:08	1	-0.0095	1.0	
23	RTA0133-13DUP	06 Jan 2010	17:49:11	1	-0.0095	1.0	10A0223-DUP1 RPD=ND
~~24~~	~~RTA0133-13MS~~	~~06 Jan 2010~~	~~17:50:13~~	~~1~~	~~0.0823~~	~~1.0~~	
25	RTA0130-01*	06 Jan 2010	17:51:16	1	0.8304	1.0	
26	NO3 1.5PPM	06 Jan 2010	17:52:18	1	1.4905	1.0	10A0239-BS1 99%
27	MBLK	06 Jan 2010	17:53:21	1	-0.0095	1.0	10A0239-BLK1 <0.05
28	RTA0133-13MS*	06 Jan 2010	17:54:39	1	0.0989	1.0	10A0223-MS1 10%
~~29~~	~~RTA0132-01~~	~~06 Jan 2010~~	~~17:59:10~~	~~1~~	~~0.1585~~	~~1.0~~	See cups #67+76 (anomaly)
30	RTA0132-02	06 Jan 2010	18:00:12	1	0.0066	1.0	
31	RTA0132-03	06 Jan 2010	18:01:16	1	0.3431	1.0	
32	RTA0132-04	06 Jan 2010	18:02:19	1	0.4419	1.0	
33	RTA0132-05	06 Jan 2010	18:03:23	1	0.0973	1.0	
34	RTA0132-06	06 Jan 2010	18:04:27	1	0.0526	1.0	
35	RTA0132-07	06 Jan 2010	18:05:30	1	0.4460	1.0	
~~36~~	~~RTA0132-08~~	~~06 Jan 2010~~	~~18:06:34~~	~~1~~	~~0.0595~~	~~1.0~~	anomaly - see cups #72+78
37	RTA0136-01	06 Jan 2010	18:07:38	1	0.0202	1.0	

Attachment 17.5
Wet Chemistry Batch Summary



WET CHEMISTRY BATCH SUMMARY

PARAMETER______________________ METHOD__________ BATCH ______________________

COMMENTS	JOB NUMBER
WC Historical confirms within Hold Time	
WC Historical NO confirm & RE outside of HT	
WC Hold Time Exceedance-Dilution required	
WC Hold Time Exceedance-Instrument Failure	
WC Holding Time Exceedance by Date	
WC Holding Time Exceedance by Hours	
WC LCS within ERA limits outside internal	
WC LCS high recovery, sample ND	
WC MBLK hit but samples > 10X blank value	
WC RPD Exceedance for MS / SD	
WC Spike Failure HIGH MS only	
WC Spike Failure LOW MS only	
WC Spike Failure MS and SD	
WC BOD HT met- Oxygen depleted-RE out HT	
WC Carbonate Alkalinity, LCS/MBLK	
WC Reactivity Qualification	
WC TDS/Conductivity ratio outside of range	
WC TOX Breakthrough- no volume for redo	
WC TOX samples were centrifuged	
Other	

DILUTION CODES	REASON
002	Sample matrix effects
003	Excessive foaming
004	High levels of non-target compounds
008	High concentration of target analytes
009	Sample turbidity
010	Sample color
011	Insufficient volume for lower dilution
012	Sample viscosity
013	other

ICAL Compliant?	YES	NO	NA	IF NO, Why? ______________________
LCS/CCV Compliant?	YES	NO	NA	IF NO, Why? ______________________
CCB Compliant?	YES	NO	NA	IF NO, Why? ______________________
RPD Compliant?	YES	NO	NA	IF NO, Why? ______________________
ERA Compliant?	YES	NO	NA	IF NO, Why? ______________________

NUMBER of REANALYSIS FOR THIS BATCH:__________

Analyst______________________ Date__________

Time Critical Batch Review______________ Date__________

Secondary Review & Closure______________ Date__________

WC Summary Rev5 / 05-2008

TestAmerica
THE LEADER IN ENVIRONMENTAL TESTING

Title: Dissolved Gases- Modified Method RSK-175

Once printed, this is considered an uncontrolled document.

Approvals (Signature/Date):

12/01/09
Gary S. Rudz — Date
GC Department Manager

Kenneth E. Kasperek 12/01/09
Kene Kasperek — Date
Health & Safety Manager / Coordinator

Peggy Gray-Erdmann 12/01/09
Peggy Gray-Erdmann — Date
Quality Assurance Manager

12/01/09
Chris Spencer — Date
Laboratory Director

Facility Distribution No. ___________ **Distributed To:**______________________

1.0 Scope and Application

This method is used to qualify and quantify aliphatic and olefinic hydrocarbons normally found in the gas phase at room temperature in water. The method is applicable to the preparation of water samples for the analysis of the headspace through introduction into a capillary column equipped with gas chromatography. This method is restricted to use under the supervision of analysts experienced in the use of gas chromatography and the integration of gas chromatography.

1.1 Analytes, Matrix(s), and Reporting Limits

This method is routinely used to determine the amount of Methane, Ethane and Ethene in groundwater, drinking water, surface water and other aqueous samples. The table below shows the reporting limits for each analyte.

Compound	µg/L
Methane	1
Ethane	1.5
Ethene	1.5

Additional compounds can be analyzed for, given by the following reporting Limits:

Compound	µg/L
Acetylene	1.5
Propane	10
Butane	10

On occasion clients may request modifications to this SOP. These modifications are handled following the procedures outlined in the Quality Assurance Manual.

2.0 Summary of Method

A water sample is collected in the field in a 44 ml VOA vial with no headspace. Prior to analysis, the sample is transferred into a 22ml serum vial with a crimp cap. Headspace is generated using UHP helium. The sample is loaded onto the headspace autosampler and analyzed by gas chromatography equipped with an FID detector. The headspace concentration is related to the starting water concentration through the use of Henry's Law.

3.0 Definitions

3.1 *Henry's Law:* Henry's Law states that the ratio of the partial pressure of a gas in a closed system and molar concentration of solution is a constant. This constant varies with temperature and is compound specific. (See attachment 1)
3.2 *LCS/MSB/BS:* all synonyms for laboratory quality control samples.
3.3 *MS/MSD/SD:* all synonyms for client sample spikes
3.4 *DUP/MD:* refers to duplicate samples
3.5 *VBLK/MBLK/BLK:* all refer to method blanks
3.6 *ICV:* initial 2nd source check for calibration in Element (Alt source)
3.7 *CCV:* Continuing Calibration Verification
3.8 *Primer:* old standard/QC/blank analyzed if instrument has been idle

4.0 Interferences

Method interference may be caused by contaminants in solvents, reagents, glassware and other processing apparatus that lead to discreet artifacts. All of these materials must be routinely demonstrated to be free from interference under conditions of the analysis by running laboratory method blanks as described in the Quality Control section. Specific selection of reagents may be required to avoid introduction of contaminants.

Background atmospheric methane is commonly present at levels of 5-10 ppm. This may cause small amounts of methane to be present in laboratory blanks (typically $\leq 0.25\mu g/L$).

Carry-over contamination is not a routine problem with this analysis due to the volatile nature of the gases being tested. The characteristics (heat and run length) of the column and the GC temperature program are sufficient to prevent carryover. Each sample has its own new vile and is directly transferred on to the GC. The analyst must be familiar with the characteristics of the system to determine when carryover may have occurred, reanalyzing any samples suspected of carryover contamination as soon as possible.

5.0 Safety

Employees must abide by the policies and procedures in the Corporate Safety Manual, Radiation Safety Manual and this document.

This procedure may involve hazardous material, operations and equipment. This SOP does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of the method to follow appropriate safety, waste disposal and health practices under the assumption that all samples and reagents are potentially hazardous. Safety glasses, gloves, lab coats and closed-toe, nonabsorbent shoes are a minimum.

The toxicity and carcinogenicity of each reagent used in this SOP has not been precisely defined. Additional health and safety information can be obtained from the applicable Material Safety Data Sheet (MSDS) maintained in the laboratory and on-line in the EH&S section of the Test America intranet.

Each Sample is treated as a potential health hazard; exposure to each sample is reduced to the lowest possible level. Personal protective equipment, including but not necessarily limited to eye protection, lab coats, and gloves must be worn and used as specified in the CSM. This includes all personnel, visitors, and contractors that are in the laboratory area unless that area has been designated by the EH&S Coordinator as an exclusion area.

5.2 Primary Materials Used

The following is a list of the materials used in this method, which have a serious or significant hazard rating. **Note: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the MSDS for each of the materials listed in the table.** A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the MSDS for each material before using it for the first time or when there are major changes to the MSDS.

Material	Hazards	Exposure Limit (1)	Signs and symptoms of exposure
Hydrochloric Acid	Corrosive Poison	5 ppm-celing	Inhalation of vapors can cause coughing, choking, inflammation of the nose, throat, and upper respiratory tract. In severe cases can result in pulmonary edema, circulatory failure, and death. Can cause redness and severe skin burns. Vapors are irritating and may cause severe burns and permanent eye damage.
1 – Exposure limit refers to the OSHA regulatory exposure limit. 2 – Always add acid to water to prevent violent reactions.			

6.0 Equipment and Supplies

Gas Chromatograph – Analytical system, compete with gas chromatograph and a headspace autosampler, as well as all required accessories. This includes, but is not limited to FID, column supplies, recorder, gases and syringes. A data system for measuring peak heights and/or areas is recommended.

6.1 Instrumentation

- Column A: U-Plot
- Column B: Alumina
- headspace autosampler

6.2 Supplies

- Sample containers: 44 ml VOA vials, 22ml crimp cap vials.
- Syringes: various sizes from 10uL to 5 mL gastight syringes.
- Various sample "loops"
- Tedlar bags: 1 Liter

7.0 Reagents and Standards

- Gas Cylinders of ultrahigh purity helium and nitrogen
- Calibration Standards: The standard is composed of 1% (molar basis) or 10,000 ppmv Methane, Ethane, Ethene and Acetylene.
  - A true second source material is not available for these calibration standards. Test America attempts to obtain separate lot numbers to perform calibration verification.
  - The calibration levels are achieved by injecting different amounts of the primary source calibration standard into a 22 ml vial that contains 17 ml of deionized water and 5 ml of headspace.
- Laboratory Control Sample (LCS)- is prepared using the primary source calibration standard and are fortified to the concentration of the middle calibration standard.

8.0 Sample Collection, Preservation, Shipment and Storage

- Sample container, preservation techniques and holding times may vary and are dependent on sample matrix, method of choice, regulatory compliance, and/or specific contract or client requests.
- Samples are collected in the field in a 44mL VOA vial. The samples are preserved with 1:1 HCL to a pH of less than 2.
- Care should be taken that no headspace is present when capping the vials.
- Samples are maintained at a temperature of 4+/-2°C and must be analyzed within 14 days of collection.
- If dissolved CO2 or CO are being determined the sample should be collected in a 44 ml VOA vial with no preservative. If acid is added, dissolved carbonates with be converted to CO2 and bias the results.

9.0 Quality Control

For the standard analyte list, the initial demonstration of capability (IDOC) and method detection limits must be acceptable before analysis of samples may begin.

9.1 Sample QC

Quality Controls	Frequency	Control Limit
Method Blank (MB)	1 in 20 or fewer samples	< Rpt. Limit
Laboratory Control Sample (LCS)[1]	1 in 20 or fewer samples	Statistical Limits [4]
Matrix Spike (MS)[2]	1 in 20 or fewer samples	Statistical Limits [4]
MS Duplicate (MSD)[2]	1 in 20 or fewer samples	Statistical Limits [4]
Surrogates	every sample[3]	Statistical Limits [4]

9.1.1 A batch is set up to 20 samples of the same matrix processed using the same procedures and reagents with the same time period. The batch should contain a method blank, a laboratory control sample (LCS/MSB) and a matrix spike.matrix spike duplicate (MS/MSD). If insufficient sample is available for a MS/MSD, a LCSD (LCSD/MSBD) may be substituted.

9.1.2 A method blank must be analyzed once every 20 samples or 24 hours, whichever comes first. The method blank consists of reagent water containing all reagents specific to the method and is carried through the entire analytical procedure.

- If a method blank exhibits contamination above Laboratory Quantitation Limit, all related sample results must be evaluated.
- Positive method blank results slightly below the reporting limit should be evaluated by the analyst for potential impact on sample results at or near the reporting limit.
- The most common lab contaminant for this is Methane. Methane can be present up to 5 times the reporting limit.
- Samples containing the same analytes found in the method blank must be re-analyzed.

9.1.3 Matrix spike and matrix spike duplicate samples are to prepared at a frequency of at least 5% (1 MS per 20 samples and 1 MSD per 20 samples).

Batch MS/SD is an option of fulfilling the requirements, but at least one set of MS/SD should be run per day of instrument operation. The MS and MSD are fortified at a concentration equal to Level C of the calibration curve.

9.1.4 Laboratory Control Sample/ Matrix Spike Blank - a minimum of one matrix spike blank is required every 20 samples or 24 hour, whichever comes first. The LCS/MSB is prepared using the primary source calibration standard.

- Spiking levels for LCS are the same as for MS/MSD.
- The LCS/MSB must provide a recovery that is within ±50% of its theoretical value, until internal limits are established. In the event of LCS failure, re-analysis is required. If re-analysis continues to indicate LCS failure, all sample analyses completed relative to that LCS are subject to re-analysis.
- If the LCS recoveries are biased high and the associated samples are ND for the parameter of interest, the sample data is acceptable and may be processed for reporting.

9.2 Instrument QC

9.2.1 Initial Calibration Verification (ICV): A check of the initial calibration curve must be made after the initial calibration. The ICV consists of the injection of a second source standard prepared at Level D of the calibration curve. The ICV must meet ±30% of its theoretical value.

9.2.2 Continuing Calibration Verification (CCV): For calibration verification (i.e. continuing calibration) of the analytical curve, a standard prepared at Level D of the curve (20 µL in 16.98 mL of VOA free water) must be analyzed every 20 samples of 24 hours, whichever comes first, and at the end of each analysis sequence. The CCV response factor must be within ±30% of the calibration average response factor.

10.0 Procedure

10.1 Sample Preparation

- Remove samples from the refrigerator and allow them to come to room temperature.
- Transfer the sample into a 22mL vial with a crimp cap
- Place the vial upside down, and insert a 22 gauge needle into the septum.
- Using an additional 5 mL gastight syringe, inject 5 mL of UHP helium into the sample. The helium forces out an equal amount of sample through the 22 gauge needle to create a headspace volume of 5 mL.
- Withdraw the syringes from the vial and load the sample onto the headspace autosampler. The autosampler allows the sample's water and headspace phases to equilibrate.
- The sample's headspace is injected directly onto the GC column where target compounds, if present, are detected by the FID. The instrument operating conditions are outline in section 10.3.

10.2 Calibration

Using the primary source calibration standard, a five-point curve is established for each compound of interest using the concentrations noted in the table below:

Compound	**Cal Level A**	**Cal Level B**	**Cal Level C**	**Cal Level D**	**Cal Level E**
Volume injected (ul)	**2**	**5**	**10**	**20**	**50**
Volume diluent (ml)	**16.998**	**16.995**	**16.99**	**16.98**	**19.95**
Methane (ug/L)	0.772	1.93	3.86	7.72	19.29
Ethane (ug/L)	1.33	3.38	6.76	13.52	33.76
Ethene (ug/L)	1.45	3.62	7.24	14.48	36.17
Add Compounds					
Acetylene (ug/L)	1.25	3.13	6.27	12.5	31.34
Propane (ug/L)	2.12	5.30	10.6	21.20	53.00
Butane (ug/L)	2.80	6.99	13.98	29.97	69.92

10.2.1 For the initial calibration curve to be acceptable, it must meet the appropriate criteria of either average response factor or correlation coefficient curve fit.

- The average calibration factor may be used if the average percent Relative Standard Deviation (%RSD) of the response factors in ≤30%.
- The correlation coefficient may be used if it is ≥0.995.

10.2.2 Removal or replacement of levels from the middle of a calibration is not permitted unless an injection or instrument problem confined to that point can be clearly documented. Removal of points for individual analytes from levels other than the highest and lowest is not permitted in any event.

10.3 Instrument Operating Conditions

10.3.1 Instrument Performance Specifications-The gas chromatograph is set up to reflect the following operating conditions:

Injection B Temperature	200°C
Detector B	250°C
Oven Maximum	250°C
Range 2	0
Signal 1	Att. 0
Signal 2	Att. 0

10.3.2 Column Conditions reflect the compounds of interest in an analysis.

Initial Temperature	45°C
Initial Time	2 min.
Rate	20°C/min.
Final Temperature	220°C

Final Time	0 min.

10.3.3 Tekmar® 7000 autosampler conditions:

Platen	65°C
Platen Equilibrium Time	1
Sample Equilibrium Time	3
Vial Size	20 ml
Mix	on
Mix Power	3
Stabilize	2
Pressure	0.5
Pressure Equilibrium Time	0.2
Loop	0.3
Loop Equilibrium	0.05
Injection	0.5
Valve	120°C
Line	120°C
Cycle Time	12

10.4 Instrument Maintenance Procedures

- Analyze blanks before samples and determine system to be clean and free of interference.
- If interference is present, the column may be baked out at 190°C, and/or the capillary column may be cut at the injector end at a length of 6-12 inches.
- Maintenance activities must be logged into the instrument maintenance logbook.

10.5 Analytical Documentation

- Record all analytical information in the analytical logbook, including the analytical data from standards, blanks, LCS/LCDSs, MS/MSDs and any corrective actions or modifications to the method.
- All standards are logged into the department standard logbook. All standards are assigned a unique number for identification.
- Documentation such as all associated instrument printouts and daily calibration data corresponding to all final runs is available for each data file.
- Sample results and associated QC are reviewed by the primary analyst and entered into Element. A secondary technical review and evaluation of the Element data is performed and documented prior to release of data for reporting.

11.0 Calculations / Data Reduction

11.1 Calibration Factor for GC-FID

$$\mathbf{CF\chi = A\chi/C\chi}$$

$CF\chi$= Calibration factor of compound χ
$A\chi$= Peak height or area
$C\chi$= Calibration amount

11.2 Percent Difference for Calibration Factors

$$\mathbf{\%D=(CF[ave]-CF[c]/CF[ave])*100}$$

CF[ave]=Average Calibration Factor for an analyte from the intial calibration
CF[c]= Calibration Factor for an analyte from current check standard

11.3 Relative Standard Deviations

$$\mathbf{\%RSD=(SD/CF[ave])*DF}$$

CF[ave]=Average Calibration Factor for an analyte for the initial calibration
SD=Standard Deviation of average calibration factors for a compound
DF= dilution factor

11.4 Sample Concentration in water

$$\mathbf{C\chi=(A\chi/CF[ave])*DF}$$

$C\chi$= Concentration of target analyte χ in sample (ug/L)
$A\chi$= peak area of analyte χ
CF[ave]= Average calibration factor for an analyte for the calibration
DF=dilution factor

12.0 Method Performance

12.1 Method Detection Limit Study (MDL)

The method detection limit (MDL) is the lowest concentration that can be detected for a given analytical method and sample matrix with 99% confidence that the analyte is present. A method detection limit study is performed on an annual basis in accordance with the current specifications described in 40CFR part 136, Appendix B. The final MDL for each analyte should be at least less that half of the laboratory quantitation limit. MDLs reflect a calculated (statistical) value determined under ideal laboratory conditions in a clean matrix, and may not be achievable in all environmental matrices. The laboratory maintains MDL studies for analyses performed; these are verified at least annually unless method requirements require a greater frequency.

12.2 Demonstration of Capabilities

Each analyst, prior to sample analysis, must analyze an Initial Demonstration of Capability. This consists of four replicate QC check standards composed of 20ul of specialty gases at 1% (mole basis) analyzed with a mean recovery of 70-130%.

13.0 Pollution Control

It is TestAmerica's policy to evaluate each method and look for opportunities to minimize waste generated. This includes, but is not limited to, examining recycling options, ordering chemicals based on quantity needs, as well as preparation of reagents based on anticipated usage and stability. Employees must abide by the policies in Section 13 of the Corporate Environmental Health and Safety Manual (CW-E-M-001) for "Waste Management and Pollution Prevention.

14.0 Waste Management

All waste will be disposed of in accordance with Federal, State, and Local regulations. Where reasonably feasible, technological changes have been implemented to minimize the potential for pollution of the environment.

Disposal of liquid volatile waste is broken down into two categories: Aqueous waste and solvent waste. Aqueous waste is temporarily stored in a laboratory approved waste receptacle and labeled "A" waste. Solvent waste is stored in laboratory approved metal waste receptacle and labeled "C" waste.

Waste receptacles are taken to sample control where they are disposed of.

Glass waste such as pipettes and vials are rinsed and disposed of in approved glass receptacles.

15.0 Attachments

Attachment 1: Example Calculation
Attachment 2: Table of Constants

16.0 Revision History

CHANGES LISTED IN PREVIOUS FORMAT

Laboratory Director change, updated signature
Section 8.1: Included Corporate EH&S statement.
Section 10.2.1: Removed reference to two separate vendors. Stated that 2nd lot# was used for calibration verification.
Section 18.0: Specific Corrective Action information included for out-of-control QC indicators. Included dual CCV decision tree.
Section 20.1: Included Corporate EH&S statement related to waste disposal regulations.

Revision 0, dated December 1, 2009

- Transferred into updated format, changed multiple section headers numbers, rearranged to match requirements (Removed sections 17-23)
- Updated TCL and RL to reflect add compounds – section 1.0
- Updated Calibration to reflect curve levels for add compounds – section 10.0
- Updated columns used – section 6.1
- Removed CCV decision tree, dual CCVs no longer used.

Attachment 1:

Example Calculation

(Based on 22°C at 754 mmHg – Molar Equivalent 0.04099)

(Level D) 20ul of 10,000 ppmv – 17 ml H2O

$$\frac{10{,}000\ ulCH_4}{LN_2} \times \frac{1LCH_4}{1{,}000{,}000\ ulCH_4} \times \frac{0.04099\ molesCH_4}{1LCH_4} \times \frac{16\ gCH_4}{1moleCH_4} \times \frac{0.00002\ LN_2}{0.017\ LH_2} \times$$

$$\frac{1{,}000{,}000\ ugCH_4}{1gCH_4} = \frac{0.1312}{0.017} = \frac{7.72\ ugCH_4}{LH_2O}$$

Attachment 2:

Table of Compound Constants

Compound	Molecular Weight (g)
Methane	16
Ethane	30
Ethene	28
Propane	44
Propene	42

Title: Sulfate - Turbidimetric
EPA 375.4 & SW846 9038 & ASTM D-516-90

Once printed, this is considered an uncontrolled document

Approvals (Signature/Date):

Lisa R Matecki 03/12/10
Lisa R. Matecki Date
Department Manager

Kenneth E. Kasperek 03/12/10
Kenneth E. Kasperek Date
Technical Director

Peggy Gray-Erdmann 03/11/10
Peggy Gray-Erdmann Date
Quality Assurance Manager

03/11/10
Christopher A. Spencer Date
Laboratory Director

Facility Distribution No. ___________ **Distributed To:**________________________

1.0 Scope and Application

1.1 Analytes, Matrix(s), and Reporting Limits

This method is applicable to drinking and surface waters and domestic and industrial wastes for sulfate concentrations in the range of 5 to 40 mg/L.

On occasion clients may request modifications to this SOP. These modifications are handled following the procedures outlined in the Quality Assurance Manual.

2.0 Summary of Method

Sulfate ion (SO_4) reacts with barium chloride in an acid medium to form a barium sulfate suspension, the absorbance of which is measured by a spectrophotometer and compared to a standard curve. Solid samples are extracted into water, filtered and then analyzed according to this SOP.

3.0 Definitions

3.1 Standard definitions are in section 3.0 of the LQM

3.2 Konelab: Automated multi-Chemistry Analyzer Model Aqua20 or 20XT

4.0 Interferences

4.1. Since the method measures the turbidity of the barium sulfate suspension, color or suspended solids will cause a positive interference. Remove suspended solids by filtration. If samples are colored, run color blanks from which the barium chloride has been omitted and subtract the absorbency.

4.2. Silica in excess of 500 mg/L will interfere.

4.3. In waters containing large quantities of organic material it may not be possible to precipitate barium sulfate (BaSO4) satisfactorily.

5.0 Safety

Employees must abide by the policies and procedures in the Corporate Safety Manual, Radiation Safety Manual and this document.

This procedure may involve hazardous material, operations and equipment. This SOP does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of the method to follow appropriate safety, waste disposal and health practices under the assumption that all samples and reagents are potentially hazardous. Safety glasses, gloves, lab coats and closed-toe, nonabsorbent shoes are a minimum.

5.1 Specific Safety Concerns or Requirements

None

5.2 Primary Materials Used

The following is a list of the materials used in this method, which have a serious or significant hazard rating. **NOTE: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the MSDS for each of the materials listed in the table.** A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the MSDS for each material before using it for the first time or when there are major changes to the MSDS.

Material [1]	**Hazards**	**Exposure Limit** [2]	**Signs and symptoms of exposure**
Hydrochloric Acid	Corrosive Poison	5 ppm-Ceiling	Inhalation of vapors can cause coughing, choking, inflammation of the nose, throat, and upper respiratory tract, and in severe cases, pulmonary edema, circulatory failure, and death. Can cause redness, pain, and severe skin burns. Vapors are irritating and may cause damage to the eyes. Contact may cause severe burns and permanent eye damage.
Barium Chloride	Poison Irritant	0.5 mg/m^3 TWA	May be fatal if swallowed. Harmful if inhaled. Avoid contact with eyes, skin, and clothing. Avoid breathing dust. Keep container closed and when in use adequate ventilation.
1 – Always add acid to water to prevent violent reactions.			
2 – Exposure limit refers to the OSHA regulatory exposure limit.			

6.0 Equipment and Supplies

6.1. Instrumentation

6.2. Konelab Aqua20 or 20XT multi-analyzer

6.3. Supplies

6.3.1. 2 mL sample cups

6.3.2. Eppendorfs, various sizes.

6.3.3. Class A volumetric flasks, various sizes

6.3.4. Sample analysis cuvettes

7.0 Reagents and Standards

7.1. **Stock Standard** (1000mg/L as SO_4): Purchased pre-made from various vendors.

7.2. **Precipitating solution**: Dissolve 5.0g of Barium Chloride (BaCl2), 10-g Sodium Chloride (NaCl) and 0.25g gelatin in 300mL of di water (requires prolonged stirring and gentle heating to dissolve). Carefully add 17.52mL of 0.1N Hydrochloric acid and dilute to 500mL with di water. This reagent is to be made monthly.

8.0 Sample Collection, Preservation, Shipment and Storage

Sample container, preservation techniques and holding times may vary and are dependent on sample matrix, method of choice, regulatory compliance, and/or specific contract or client requests. Listed below are the holding times and the references that include preservation requirements.

Matrix	Sample Container	Min. Sample Size	Preservation	Holding Time	Reference
Waters	Plastic or Glass	50mL	Cool 4 ± 2°C	28 Days	40 CFR Part 136.3 ASTM D516-90 SW-846 3rd Edition
Soils	Plastic or Glass	50g	Cool 4 ± 2°C	28 Days	N/A

9.0 Quality Control

9.1 Sample QC

Quality Controls	Frequency	Control Limit
Method Blank (MBLK)	1 in 20 or fewer samples	< Rpt. Limit
Laboratory Control Sample (LCS)	1 in 20 or fewer samples	Statistical Limits [3]
Matrix Duplicate (MD)[1]	1 in 20 or fewer samples	Statistical Limits [3]
Matrix Spike (MS)[1]	1 in 20 or fewer samples	Statistical Limits [3]

[1] The sample selected for MD/MS IS randomly done so, unless specifically requested by a client.

[2] Analytical and QC samples (MB, LCS, MD/MS)

[3] Statistical control limits are updated annually and are updated into LIMS.

10.0 Procedure

10.1 Calibration

Measure standards as indicated in the following table and dilute to 25 ml with Reagent Water to obtain the desired sulfate standards.

Concentration	Volume
5 mg/L	125uL 1,000mg/L Sodium Sulfate STD dilute to 25mLs with DiH_2O.
10 mg/L	250uL 1,000mg/L Sodium Sulfate STD dilute to 25mLs with DiH_2O.
15 mg/L	375uL 1,000mg/L Sodium Sulfate STD dilute to 25mLs with DiH_2O.
20 mg/L	500uL 1,000mg/L Sodium Sulfate STD dilute to 25mLs with DiH_2O.
25 mg/L	625uL 1,000mg/L Sodium Sulfate STD dilute to 25mLs with DiH_2O.
30mg/L	750uL 1,000mg/L Sodium Sulfate STD dilute to 25mLs with DiH_2O.
35 mg/L	875uL 1,000mg/L Sodium Sulfate STD dilute to 25mLs with DiH_2O.
40 mg/L	1.00 ml 1,000mg/L Sodium Sulfate STD dilute to 25mLs with DiH_2O.

10.1.1 Calibration Acceptance Summary

A calibration curve must be run at a minimum of once every three months. Acceptance criteria is a correlation coefficient (R value) of ≥ 0.990. The coefficient of detection listed on the Kone curve data is an R value. The curve shall consist of the preceding points

(Section 10.1). Analysis cannot begin without an acceptable calibration curve. Instrument maintenance may be required. Please refer to TESTAMERICA Corporate Policy for information on the proper selection of calibration points.

10.2 Sample Analysis

10.2.1 Each day it is necessary to check the quality of the water blank. This is done after the start up procedure. All results must be within +/-2mA (milliabsorbance). If not rerun this procedure.

10.2.2 Input the sample identification required by the data system and load samples in the instrument segments.

10.2.3 Analyze samples.

10.2.5 After the analysis has finished, you need to check the Kinetic Sulfate results for each sample. The kinetic data for sulfate shoes the responses at 30 second intervals over a four minute period. To find the kinetic data click F8 twice, then F2 (Result Archive.) You can now filter on test Kinetic Sulfate, and pull up all results from the day. Once you do this, you can now click on each result, then hit F1 (Request Details.) By comparing the highest response in this list with the response given by the single endpoint application (final response) you will be able to prove that you are taking the maximum response obtained in the four minute period. If a response is higher within the four minute period than your final response, you must use the highest response and manually calculate the final result. You must document this on your kinetic data and include the highest response and final result, explanation, date and initial.

11.0 Calculations / Data Reduction

11.1 Sample results are calculated from the calibration curve by using linear regression.

11.2 In the case of any dilution, the results have the dilution factor automatically calculated.

11.3 For liquid samples, the results are expressed in sulfate as $SO_4{}^{-2}$ mg/L.

11.4 For solid samples, the results are expressed as sulfate as $SO_4{}^{-2}$ mg/kg on a dry basis.

11.5 To convert the mg/L result obtained from the calibration curve to mg/kg use the following equation:

mg/kg (wet) = [mg/L X final vol. of leached sample] / grams sample used

mg/kg (dry) = mg/kg (wet) / decimal dry weight

11.6 **MS % Recovery** :

$$\% \text{ Recovery} = \left[\frac{(SSR - SR)}{SA}\right] \text{ x } 100$$

where:
SSR = spiked sample result
SR = sample result
SA = spike added

11.7 Relative Percent Difference (RPD):

$$RPD = \frac{|x_1 - x_2|}{\left(\frac{x_1 + x_2}{2}\right)} \text{ x } 100$$

where:
x_1 = analytical % recovery
x_2 = replicate % recovery

11.8 Measured Concentration by Linear Regression:

$$x = \frac{a - b}{m}$$

where:
a = area counts for analyte to be measured
m = slope
x = concentration
b = intercept

11.9 Percent Recovery for LCS:

$$\% \text{ Recovery (LCS)} = 100\left(\frac{E}{C}\right)$$

where:
E = obtained (experimental) value
C = true value

12.0 Method Performance

12.1 Method Detection Limit Study (MDL)

The method detection limit (MDL) is the lowest concentration that can be detected for a given analytical method and sample matrix with 99% confidence that the analyte is present. The MDL is determined according to the laboratory's MDL procedure in the

TestAmerica QA manual. MDLs reflect a calculated (statistical) value determined under ideal laboratory conditions in a clean matrix, and may not be achievable in all environmental matrices. The laboratory maintains MDL studies for analyes performed; these are verified at least annually unless method requirements require a greater frequency.

12.2 Demonstration of Capabilities

12.2.1 A one–time initial demonstration of performance for each individual method for both soils and water matrices must be generated.

12.2.2 This requires quadruplicate analysis of a mid–level check standard containing all of the standard analytes for the method using the same procedures used to analyze samples, including sample preparation.

12.2.3 Repeat the test for any analyte that does not meet the acceptance criteria. Repeated failure for any analyte indicates the need for the laboratory to evaluate the analytical procedure and take corrective action.

12.3 Training Requirements

12.3.1 The supervisor has the responsibility to ensure that this procedure is performed by an analyst who has been properly trained in its use and has the required experience.

12.3.2 The following analyst validation information is maintained for this method in the laboratory QA files.

12.3.3 The analyst must complete the laboratory safety orientation training that includes, but is not limited to, chemicals, PPE requirements, and electrical safety.

12.3.4 The analyst must read and understand this SOP.

12.3.5 The analyst must read and understand the Method used as reference for this SOP.

12.3.6 The analyst must complete a DOC or successfully analyze PT samples annually.

13.0 Pollution Control

It is TestAmerica’s policy to evaluate each method and look for opportunities to minimize waste generated (i.e., examine recycling options, ordering chemicals based on quantity needed, preparation of reagents based on anticipated usage and reagent stability).

13.1 Waste Streams Produced by the Method: The following waste streams are produced when this method is carried out.

13.1.1 Acid waste from acidified samples. Dispose of this waste in the “A” waste container.

14.0 Waste Management

All waste will be disposed of in accordance with Federal, State and Local regulations. Where reasonably feasible, technological changes have been implemented to minimize the potential for pollution of the environment. Employees will abide by this method and the policies in section 13 of the Corporate Safety Manual for “Waste Management and Pollution Prevention.”

15.0 References / Cross-References

15.1 Standard Methods for the Examination of Water and Wastewater, 19th Edition.

15.2 "Methods for Chemical Analysis of Water and Wastes"; USEPA; EPA-600/4-79-020, March 1989; Method 375.4.

15.3 U.S. Environmental Protection Agency, Test Methods for Evaluating Solid Waste; SW-846, Third Edition, Update III, 2/96, Method 9038.

15.4 D-516-90 Standard Test Method for Sulfate Ion in water, American Society for Testing and Materials. 1916 Race St., Philadelphia, PA 19103, 11.01, 1991.

16.0 Method Modifications:

Item	Method	Modification
7.2	375.4	Precipitation Solution made using gelatin rather than glycerol.
10.2.5	375.4	Sample turbidity is measured every 30 seconds by instrument; however the final absorbance is recorded. Analyst must check sample data to ensure that proper absorbance is recorded.

17.0 Attachments

17.1 Analytical Run Sequence.

17.2 Analytical Batch

17.3 Wet Chemistry Batch Summary & Data Review Checklist

18.0 Revision History

- Revision 0. December 1, 2007
    - Integration for TestAmerica and STL operations
    - Revised attachments 2 and 3
    - Added Section 10.8.1 to include procedure for kinetic data
    - Added Section 10.12 to include auto upload of data.

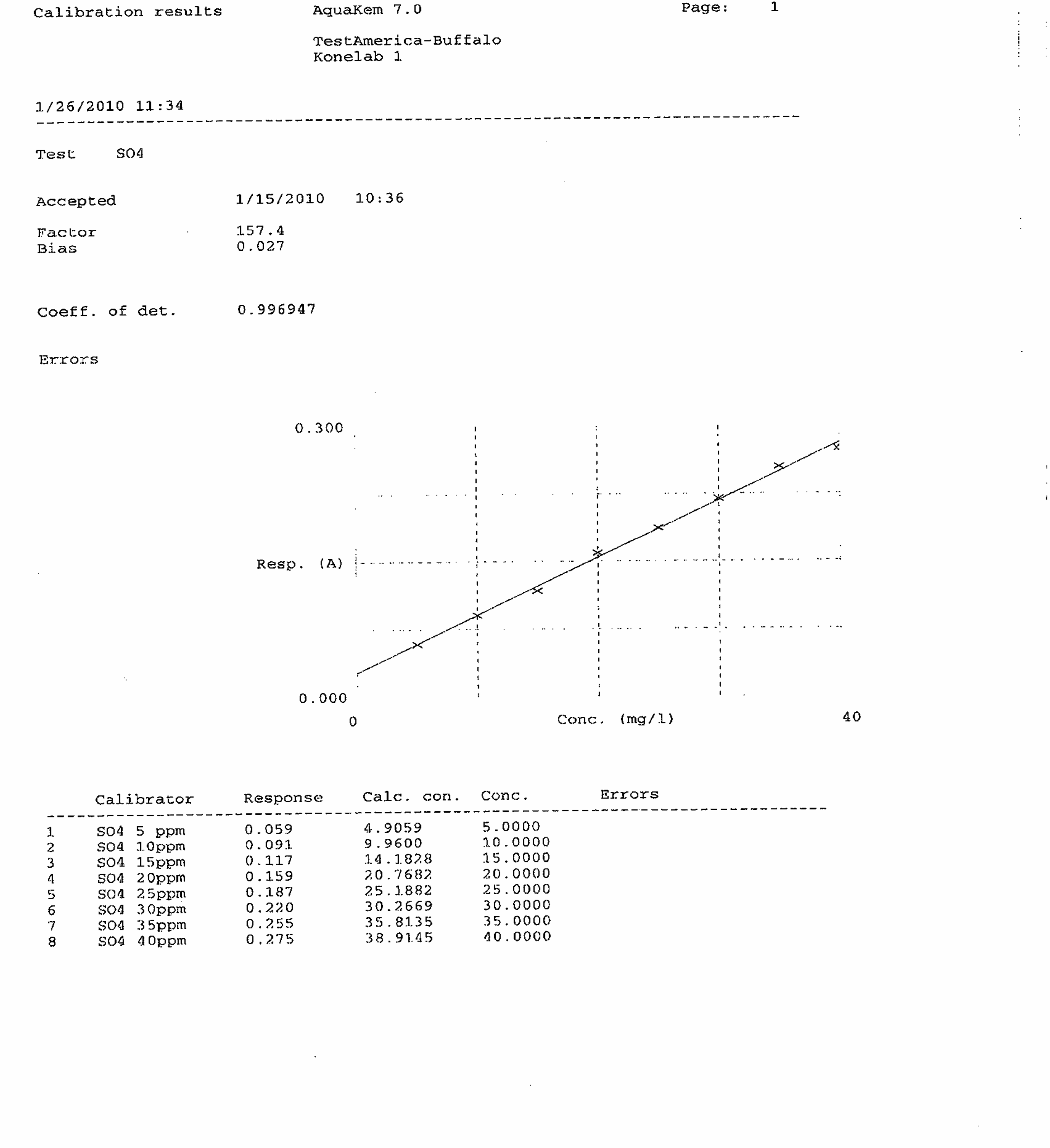
- Revision 1, March 4, 2009
    - Update Sects 17.2, 17.3, Remove Attachment 2
    - Update department and quality control managers names and signatures

- Revision 2, January 29 2010
    - Update Sect 17.2

Attachment 17.1
Analytical Run Sequence

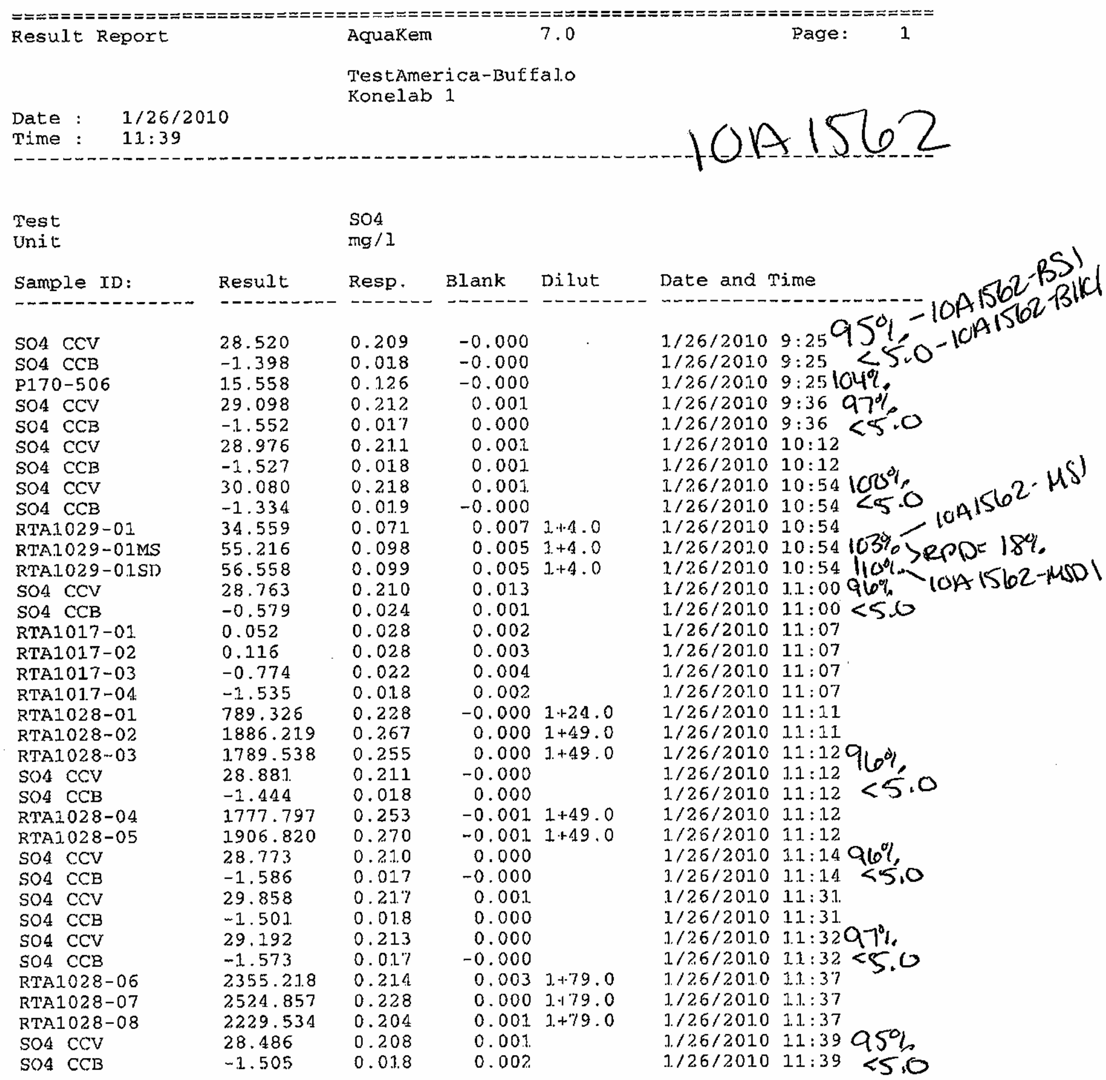
LCS
MBLK
Sample
Sample
Sample
Sample
Sample
Sample
Sample
Sample
Sample
Sample
LCS
MBLK
Sample
Sample
Sample
Sample
Sample
Sample
Sample
Sample
Sample duplicate (MD)
Sample spike (MS)
LCS
MBLK

Attachment 17.2
Analytical Batch

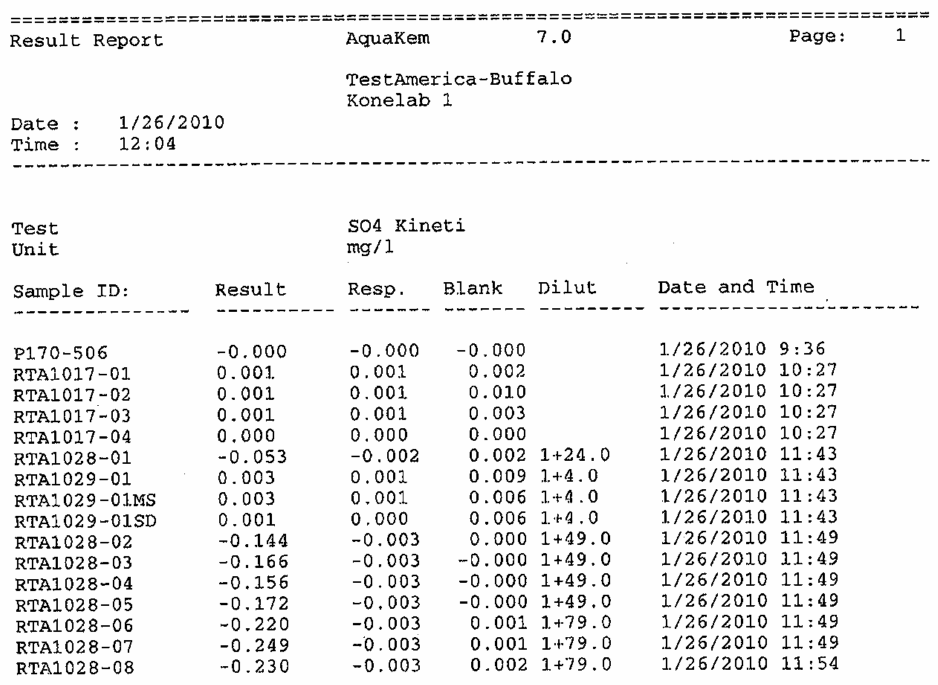


```
==============================================================================
Calibration results          AquaKem 7.0                     Page:     1

                             TestAmerica-Buffalo
                             Konelab 1

1/26/2010 11:34
------------------------------------------------------------------------------

Test     SO4

Accepted              1/15/2010    10:36

Factor                157.4
Bias                  0.027

Coeff. of det.        0.996947

Errors
```

```
       Calibrator        Response     Calc. con.   Conc.        Errors
---------------------------------------------------------------------------------
1      SO4  5 ppm        0.059        4.9059       5.0000
2      SO4  10ppm        0.091        9.9600       10.0000
3      SO4  15ppm        0.117        14.1828      15.0000
4      SO4  20ppm        0.159        20.7682      20.0000
5      SO4  25ppm        0.187        25.1882      25.0000
6      SO4  30ppm        0.220        30.2669      30.0000
7      SO4  35ppm        0.255        35.8135      35.0000
8      SO4  40ppm        0.275        38.9145      40.0000
```

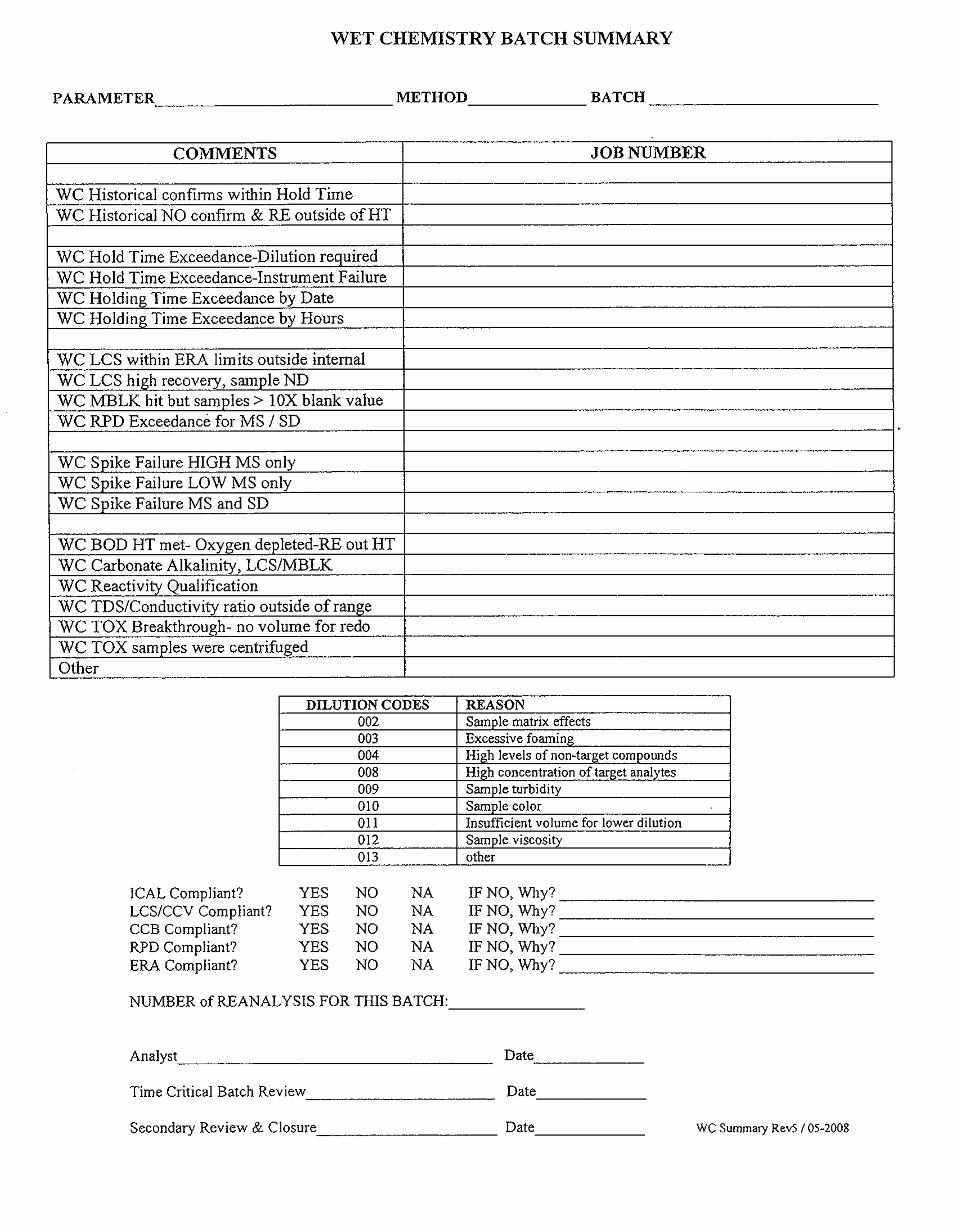


```
===============================================================================
Result Report                 AquaKem          7.0               Page:    1

                              TestAmerica-Buffalo
                              Konelab 1

Date :    1/26/2010
Time :    11:39                                              10A1562
-------------------------------------------------------------------------------


Test                          SO4
Unit                          mg/l

Sample ID:       Result     Resp.    Blank    Dilut      Date and Time
---------------  ---------- -------  -------  ---------  ----------------------


SO4 CCV          28.520     0.209    -0.000              1/26/2010 9:25  95% - 10A1562-BS1
SO4 CCB          -1.398     0.018    -0.000              1/26/2010 9:25  <5.0 - 10A1562-BLK1
P170-506         15.558     0.126    -0.000              1/26/2010 9:25  104%
SO4 CCV          29.098     0.212     0.001              1/26/2010 9:36  97%
SO4 CCB          -1.552     0.017     0.000              1/26/2010 9:36  <5.0
SO4 CCV          28.976     0.211     0.001              1/26/2010 10:12
SO4 CCB          -1.527     0.018     0.001              1/26/2010 10:12
SO4 CCV          30.080     0.218     0.001              1/26/2010 10:54 100%
SO4 CCB          -1.334     0.019    -0.000              1/26/2010 10:54 <5.0
RTA1029-01       34.559     0.071     0.007  1+4.0       1/26/2010 10:54      10A1562-MS1
RTA1029-01MS     55.216     0.098     0.005  1+4.0       1/26/2010 10:54 103%  RPD= 18%
RTA1029-01SD     56.558     0.099     0.005  1+4.0       1/26/2010 10:54 110%  10A1562-MSD1
SO4 CCV          28.763     0.210     0.013              1/26/2010 11:00 96%
SO4 CCB          -0.579     0.024     0.001              1/26/2010 11:00 <5.0
RTA1017-01       0.052      0.028     0.002              1/26/2010 11:07
RTA1017-02       0.116      0.028     0.003              1/26/2010 11:07
RTA1017-03       -0.774     0.022     0.004              1/26/2010 11:07
RTA1017-04       -1.535     0.018     0.002              1/26/2010 11:07
RTA1028-01       789.326    0.228    -0.000  1+24.0      1/26/2010 11:11
RTA1028-02       1886.219   0.267     0.000  1+49.0      1/26/2010 11:11
RTA1028-03       1789.538   0.255     0.000  1+49.0      1/26/2010 11:12
SO4 CCV          28.881     0.211    -0.000              1/26/2010 11:12 96%
SO4 CCB          -1.444     0.018     0.000              1/26/2010 11:12 <5.0
RTA1028-04       1777.797   0.253    -0.001  1+49.0      1/26/2010 11:12
RTA1028-05       1906.820   0.270    -0.001  1+49.0      1/26/2010 11:12
SO4 CCV          28.773     0.210     0.000              1/26/2010 11:14 96%
SO4 CCB          -1.586     0.017    -0.000              1/26/2010 11:14 <5.0
SO4 CCV          29.858     0.217     0.001              1/26/2010 11:31
SO4 CCB          -1.501     0.018     0.000              1/26/2010 11:31
SO4 CCV          29.192     0.213     0.000              1/26/2010 11:32 97%
SO4 CCB          -1.573     0.017    -0.000              1/26/2010 11:32 <5.0
RTA1028-06       2355.218   0.214     0.003  1+79.0      1/26/2010 11:37
RTA1028-07       2524.857   0.228     0.000  1+79.0      1/26/2010 11:37
RTA1028-08       2229.534   0.204     0.001  1+79.0      1/26/2010 11:37
SO4 CCV          28.486     0.208     0.001              1/26/2010 11:39 95%
SO4 CCB          -1.505     0.018     0.002              1/26/2010 11:39 <5.0
```



```
==============================================================================
Result Report                 AquaKem          7.0                 Page:     1

                              TestAmerica-Buffalo
                              Konelab 1

Date :    1/26/2010
Time :    12:04
------------------------------------------------------------------------------


Test                          SO4 Kineti
Unit                          mg/l

Sample ID:        Result      Resp.   Blank   Dilut      Date and Time
---------------   ----------  ------- ------- ---------  ----------------------

P170-506          -0.000      -0.000  -0.000             1/26/2010 9:36
RTA1017-01        0.001       0.001    0.002             1/26/2010 10:27
RTA1017-02        0.001       0.001    0.010             1/26/2010 10:27
RTA1017-03        0.001       0.001    0.003             1/26/2010 10:27
RTA1017-04        0.000       0.000    0.000             1/26/2010 10:27
RTA1028-01        -0.053      -0.002   0.002 1+24.0      1/26/2010 11:43
RTA1029-01        0.003       0.001    0.009 1+4.0       1/26/2010 11:43
RTA1029-01MS      0.003       0.001    0.006 1+4.0       1/26/2010 11:43
RTA1029-01SD      0.001       0.000    0.006 1+4.0       1/26/2010 11:43
RTA1028-02        -0.144      -0.003   0.000 1+49.0      1/26/2010 11:49
RTA1028-03        -0.166      -0.003  -0.000 1+49.0      1/26/2010 11:49
RTA1028-04        -0.156      -0.003  -0.000 1+49.0      1/26/2010 11:49
RTA1028-05        -0.172      -0.003  -0.000 1+49.0      1/26/2010 11:49
RTA1028-06        -0.220      -0.003   0.001 1+79.0      1/26/2010 11:49
RTA1028-07        -0.249      -0.003   0.001 1+79.0      1/26/2010 11:49
RTA1028-08        -0.230      -0.003   0.002 1+79.0      1/26/2010 11:54
```

Attachment 17.3
Wet Chemistry Batch Summary



WET CHEMISTRY BATCH SUMMARY

PARAMETER____________________________ METHOD______________ BATCH ___________________________

COMMENTS	JOB NUMBER
WC Historical confirms within Hold Time	
WC Historical NO confirm & RE outside of HT	
WC Hold Time Exceedance-Dilution required	
WC Hold Time Exceedance-Instrument Failure	
WC Holding Time Exceedance by Date	
WC Holding Time Exceedance by Hours	
WC LCS within ERA limits outside internal	
WC LCS high recovery, sample ND	
WC MBLK hit but samples > 10X blank value	
WC RPD Exceedance for MS / SD	
WC Spike Failure HIGH MS only	
WC Spike Failure LOW MS only	
WC Spike Failure MS and SD	
WC BOD HT met- Oxygen depleted-RE out HT	
WC Carbonate Alkalinity, LCS/MBLK	
WC Reactivity Qualification	
WC TDS/Conductivity ratio outside of range	
WC TOX Breakthrough- no volume for redo	
WC TOX samples were centrifuged	
Other	

DILUTION CODES	REASON
002	Sample matrix effects
003	Excessive foaming
004	High levels of non-target compounds
008	High concentration of target analytes
009	Sample turbidity
010	Sample color
011	Insufficient volume for lower dilution
012	Sample viscosity
013	other

ICAL Compliant? YES NO NA IF NO, Why? ______________________________
LCS/CCV Compliant? YES NO NA IF NO, Why? ______________________________
CCB Compliant? YES NO NA IF NO, Why? ______________________________
RPD Compliant? YES NO NA IF NO, Why? ______________________________
ERA Compliant? YES NO NA IF NO, Why? ______________________________

NUMBER of REANALYSIS FOR THIS BATCH:_________________

Analyst____ Date______________

Time Critical Batch Review________________________ Date______________

Secondary Review & Closure______________________ Date______________

WC Summary Rev5 / 05-2008

Title: Ammonia Nitrogen
Method 350.1 – Automated Phenate

Once printed, this is considered an uncontrolled document

Approvals (Signature/Date):

Lisa R Matecki 01/28/10
Lisa Matecki Date
Department Manager

Kenneth E. Kasperek 01/28/10
Kenneth Kasperek Date
Health & Safety Manager / Coordinator

Peggy Gray-Erdmann 01/28/10
Peggy Gray-Erdmann Date
Quality Assurance Manager

01/28/10
Christopher Spencer Date
Laboratory Director

Copyright Information:

This documentation has been prepared by TestAmerica Laboratories, Inc. and its affiliates ("TestAmerica"), solely for their own use and the use of their customers in evaluating their qualifications and capabilities in connection with a particular project. The user of this document agrees by its acceptance to return it to TestAmerica upon request and not to reproduce, copy, lend, or otherwise disclose its contents, directly or indirectly, and not to use if for any other purpose other than that for which it was specifically provided. The user also agrees that where consultants or other outside parties are involved in the evaluation process, access to these documents shall not be given to said parties unless those parties also specifically agree to these conditions.

THIS DOCUMENT CONTAINS VALUABLE CONFIDENTIAL AND PROPRIETARY INFORMATION. DISCLOSURE, USE OR REPRODUCTION OF THESE MATERIALS WITHOUT THE WRITTEN AUTHORIZATION OF TESTAMERICA IS STRICTLY PROHIBITED. THIS UNPUBLISHED WORK BY TESTAMERICA IS PROTECTED BY STATE AND FEDERAL LAW OF THE UNITED STATES. IF PUBLICATION OF THIS WORK SHOULD OCCUR THE FOLLOWING NOTICE SHALL APPLY:

©COPYRIGHT 2007 TESTAMERICA LABORATORIES, INC. ALL RIGHTS RESERVED

Facility Distribution No. ____________ **Distributed To:**_________________________

1.0 Scope and Application

This method is used for the determination of ammonia in drinking, surface and saline waters and domestic and industrial wastes.

Soils can be analyzed from leachates prepared using ASTM Method D 3987.

1.1 Analytes, Matrix(s), and Reporting Limits

Water, industrial wastes and soil (leachate). Soils can be analyzed from leachates prepared using ASTM method D3987.

The laboratory's reporting limit is 0.02 mg/L.

2.0 Summary of Method

Ammonia reacts with alkaline phenol and hypochlorite to form indophenol blue in an amount that is proportional to the ammonia concentration. The blue color is intensified with sodium nitroferricyanide. The absorbance is measured at 630nm.

3.0 Definitions

Standard definitions are used in this document as defined by the TestAmerica Buffalo Laboratory Quality Manual.

4.0 Interferences

4.1 Calcium and magnesium ions may precipitate if present in sufficient concentration. EDTA is added to the sample in-line, by the instrument, in order to prevent these problems.

4.2 Color and turbidity may interfere. Turbidity is removed by manual filtration.

4.3 Certain organic matter may cause interferences, resulting in biased high results. All leachate matrix samples and any sample where this may be suspected should be distilled before analysis. Dirty or improperly washed glassware may also cause interference.

5.0 Safety

5.1 Employees must abide by the policies and procedures in the Corporate Safety Manual, Radiation Safety Manual and this document.

5.2 This procedure may involve hazardous material, operations and equipment. This SOP does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of the method to follow appropriate safety, waste disposal and health practices under the assumption that all samples and reagents are potentially hazardous. Safety glasses, gloves, lab coats and closed-toe, nonabsorbent shoes are a minimum.

5.3 Specific Safety Concerns or Requirements

Sodium Nitroferricyanide will generate Hydrogen Cyanide (HCN) gas if combined with strong acids. Inhalation of CN gas can cause irritation, dizziness, nausea, unconsciousness, and potentially death.

5.4 Primary Materials Used

The following is a list of the materials used in this method, which have a serious or significant hazard rating. **Note: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the MSDS for each of the materials listed in the table.** A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the MSDS for each material before using it for the first time or when there are major changes to the MSDS.

Material	Hazards	Exposure Limit (1)	Signs and symptoms of exposure
Sodium Hydroxide	Corrosive	2 Mg/M3-Ceiling	Severe irritant. Effects from inhalation of dust or mist vary from mild irritation to serious damage of the upper respiratory tract, depending on severity of exposure. Symptoms may include sneezing, sore throat or runny nose. Contact with skin can cause irritation or severe burns and scarring with greater exposures. Causes irritation of eyes, and with greater exposures it can cause burns that may result in permanent impairment of vision, even blindness.
Sodium Nitroferri-cyanide	Poison	5 mg/m^3 as HCN gas	This material may cause irritation if it comes into the contact with the skin. The materials will give off HCN gas if combined with strong acids. Inhalation of HCN gas can cause irritation, dizziness, nausea, unconsciousness and potentially death.
Sulfuric Acid	Corrosive Oxidizer Dehydrator Poison Carcinogen	5 mg/m^3 as HCN gas	This material is extremely destructive to the tissue of the mucous membranes and upper respiratory tract. May be harmful if inhaled. May be harmful if swallowed. Symptoms of exposure may include burning sensation, coughing, wheezing, laryngitis, shortness of breath, headache, nausea and vomiting.
Phenol	Corrosive	5 ppm-TWA	Breathing vapor, dust or mist results in digestive disturbances. Will irritate, possibly burn respiratory tract. Rapidly absorbed through the skin with systemic poisoning effects to follow. Discoloration and severe burns may occur, but may be disguised by a loss in pain sensation. Eye burns with redness, pain, blurred vision may occur. May cause severe damage and blindness.

1 – Always add acid to water to prevent violent reactions.
2 – Exposure limit refers to the OSHA regulatory exposure limit.

6.0 Equipment and Supplies

6.1 Instrumentation

6.1.1 Lachat Quikchem 8000
6.1.2 Autosampler
6.1.3 Multi-channel proportioning pump
6.1.4 Reaction Unit or Manifold
6.1.5 Colorimetric Detector
6.1.6 Balance – Analytical, capable of accurately weighing to the nearest 0.00001g
6.1.7 Eppendorf Pipette – range 10 microliters to 5 milliliters.

6.2 Supplies

6.2.1 Glassware – Class A volumetric flasks and pipettes or plastic containers as required. Eppendorf Pipette tips of varying volumes.
6.2.2 Glass Culture Tubes – 5 and 10 ml

7.0 Reagents and Standards

7.1 **Alkaline phenol** - Add 40mL 10 N NaOH (or 16.0g NaOH pellets) + 44mL 88% liquefied phenol to ~250mL DiH_2O in a 500mL volumetric flask. Dilute to 500mL. Prepare weekly.

7.2 **Sodium hypochlorite** - 500mL volumetric flask, add 218mL 6% sodium hypochlorite. Dilute to the mark with DiH_2O, for a final concentration of 2.5% Sodium Hypochlorite. Prepare daily.

7.3 **5% EDTA Buffer** - In a IL volumetric flask, add 50.0g disodium ethylenediamine tetraacetate dihydrate (Na_2EDTA $2H_2O$) + 20Ml 10 N NaOH (or 9.0g NaOH). Dilute to the mark with DiH_2O and stir with magnetic stirrer until dissolved. De-gas with helium. Prepare bi-weekly.

7.4 **Sodium nitroferricyanide** - In a IL volumetric flask, dissolve 3.50g sodium nitroferricyanide in ~500mL DiH_2O. Dilute to the mark with DiH_2O. Prepare monthly.

7.5 **0.2% Sulfuric acid carrier/blank** - In a IL volumetric flask, add 2.0-mL concentrated H_2SO_4 into 900mL DiH_2O. Dilute to 1000mL with DiH_2O. Prepare daily.

7.6 **Sodium Hydroxide** - 0.1N: Dissolve 4g of NAOH in reagent water and dilute to 1 L. Prepare daily.

7.7 **Borate Buffer Solution** - Add 88mL of 0.1N NaOH to 500mL of 0.025M Sodium Tetraborate and dilute to 1L with reagent water. This is also available as a commercially prepared solution from various vendors. The life of the reagent will be two years from receipt, unless otherwise noted by the manufacturer.

7.8 **Ammonia Nitrogen 1000mg/L STD** - Available as a commercially prepared standard from various vendors.

7.9 **Ammonia Nitrogen 1000mg/L SRM** - Available as a commercially prepared standard from various vendors.

7.10 **Intermediate 100mg/L Ammonia Nitrogen STD** - To a 100mL volumetric flask, add 10mL of stock ammonia nitrogen standard to ~50mL DiH_2O. Add 0.2mL concentrated H_2SO_4 and dilute to the mark. Prepare weekly.

7.11 **LCS at 0.75ppm** - to a 100mL volumetric flask, add 75uL of stock ammonia nitrogen to ~50 mL DiH_2O. Add 200uL concentrated H_2SO_4 and dilute to the mark with DiH20. Prepare daily.

7.12 **ICV at 0.375ppm** - to a 100mL volumetric flask, add 37.5uL of stock ammonia nitrogen to ~50mL DiH2O. Add 200uL concentrated H_2SO_4 and dilute to the mark with DiH_20. This is to be analyzed once after every calibration curve and is to be made from a separate source than the calibration standards.

7.13 **Matrix Spike at 0.2ppm** - To prepare sample spikes; add 0.02mL of the 100ppm Ammonia Nitrogen standard to 10mL of sample.

7.14 **Distilled LCS at 1.0 ppm** - To a 50mL volumetric flask, add 50uL of stock ammonia nitrogen to ~30mL DiH_2O. Add 200uL concentrated H_2SO_4 and dilute to the mark with DiH_20. Distill as you would samples (Sect 14.1) then analyze.

7.15 **Distilled Matrix Spikes at 0.5ppm** - to a 50mL volumetric flask, add 25uL of stock ammonia nitrogen standard to 50mL of sample. Distill as you would samples (Sect 14.1) then analyze.

8.0 Sample Collection, Preservation, Shipment and Storage

Samples are preserved by adding 2ml of concentrated Sulfuric acid per liter of sample to obtain a pH of less than 2. Samples are then stored at 4^oC, and must be analyzed within 28 days of collection.

Matrix	Sample Container	Min. Sample Size	Preservation	Holding Time [1]	Reference
Waters	Plastic	200mLs	H_2SO_4, pH < 2; Cool 4 ± 2^oC	28 Days	40 CFR, Part 36
Soils	Glass	3 grams	Cool 4 ± 2^oC	180 Days	N/A

[1] Inclusive of digestion and analysis.

9.0 Quality Control

9.1 **Sample QC** - The following quality control samples are prepared with each batch of samples.

Quality Controls	Frequency	Control Limit
Method Blank (MBLK, CCB)	1 in 10 or fewer samples	< Rpt. Limit
Laboratory Control Sample (LCS, CCV)	1 in 10 or fewer samples	Statistical Limits [3]
Matrix Spike (MS)[1]	1 in 20 or fewer samples	Statistical Limits [3]
Matrix Duplicate (MD)[1]	1 in 20 or fewer samples	Statistical Limits [3]

[1] The sample selection for MS/MSD are randomly selected, unless specifically requested by a .
[2] Analytical and QC samples (MB, LCS, MS/MSD)
[3] Statistical control limits are updated annually and are updated into LIMS.

9.2 Instrument QC

9.2.1 Initial Calibration Verification (ICV) - This is to be analyzed once after every calibration curve and is to be made from a separate source than the calibration standards. If the ICV is within acceptance limits, proceed with samples to start run. If the ICV fails, a new calibration will need to be performed.

9.2.2 Initial Calibration Blank (ICB) - This is to be analyzed once after every calibration curve. If the ICB is within acceptance limits, proceed with samples to start run. If the ICB fails, a new calibration will need to be performed.

9.2.3 Continuing Calibration Verification (CCV) - A CCV/LCS must be performed every ten samples. The obtained value must be ± 10% of the accepted value. If the result is outside of these acceptance limits, a second CCV/LCS may be analyzed.

9.2.3.1 If 2nd analysis is acceptable, analytical sequence can continue, however the previous 10 samples must be reanalyzed.

9.2.3.2 If 2nd analysis is unacceptable, analyze a new ICAL.

9.2.4 Continuing Calibration Blank (CCB) - Analyze a CCB/MBLK every 10 samples. Obtained value must be less than the method detection limit. Reanalyze all samples associated with an unacceptable method blank unless:

9.2.4.1 Detected concentrations < PQL

9.2.4.2 Detected concentrations < 10X amount in associated sample

9.2.5 Calibration Acceptance Summary

Step	***Standards***	***Type***	***Control Limit***	***Frequency***
Method # 350.1				
Initial Cal	0.0, 0.02, 0.05, 0.2, 0.5, 1.0, 2.0	Linear	$r^2 \geq 0.995$	Monthly, unless necessary sooner.
ICV	0.375ppm		90-110%	Immediately after calibration
ICB	0.0ppm		<Reporting Limit	Immediately after calibration
CCV	0.75ppm		90-110%	Once every 10 samples
CCB	0.0ppm		<Reporting Limit	Once every 10 samples

After the calibration has been established, it must be verified by the analysis of a suitable quality control sample (ICV). If measurements exceed +/- 10% of the established value, the analysis should be terminated and the instrument re-calibrated. The new calibration must be verified before continuing analysis.

10.0 Procedure

10.1 Sample Preparation

10.1.1 DISTILLATION:

10.1.1.1 Steam out the distillation glassware using 50mLs of reagent water with a pinch of boiling chips until no trace of ammonia can be detected.

10.1.1.2 To 50mL of sample add 1N NaOH drop wise until the pH reaches 9.5.

10.1.1.3 To the receiver tube, add 5mL of 0.05% Sulfuric Acid. The tip of the long stem must be below the sulfuric acid. Set the temperature to 160^{o}C and watch for bumping of the sample to increase the temperature.

10.1.1.4 Collect a minimum of 30mL of distillate.

10.1.1.5 Dilute the distillate to the 50mL graduation mark with ammonia free water.

10.1.1.6 One LCS and MBLK must be distilled for every batch of twenty samples or less.

10.1.1.7 One sample duplicate must be distilled for every batch of twenty samples or less.

10.1.1.8 One Matrix Spike must be distilled for every batch of twenty samples or less.

10.2 Calibration

10.2.1 Prepare reagents and standards

10.2.2 Set up instrument manifold

10.2.3 Input data system parameters

10.2.4 Pump DI water through all reagent lines and check for leaks and smooth flow. Switch to reagents and allow the system to equilibrate until a stable baseline is achieved.

10.2.5 Place samples and/or standards in the autosampler. Input the information required by the data system, such as concentration, replicates and QC scheme.

10.2.6 Calibrate the instrument by injecting the standards. The data system will then associate the concentrations with the instrument responses for each standard.

10.3 Sample Analysis

10.3.1 If samples are preserved and determined without distillation, the level of preservation acid is critical.

10.3.2 Allow 15 min for heating unit to warm up to 60°C.

10.3.3 If baseline drifts, peaks are too wide or other problems with precision arise, clean the manifold by the following procedure.

10.3.4.1 Place all reagent lines in deionized water and pump to clear reagents (2 to 5 min).

10.3.4.2 Place all reagent lines in 1 M hydrochloric acid (1 volume concentrated HCl added to 11 volumes of deionized water) and pump for several minutes.

10.3.4.3 Place all reagent lines in deionized water and pump until the HCl is thoroughly washed out.

10.3.4.4 Resume pumping reagents.

11.0 Calculations / Data Reduction

11.0.1 Sample results are calculated from the calibration curve by using linear regression.

11.0.2 In the case of any dilution, the results have the dilution factor automatically calculated.

11.0.3 For liquid samples, the result is expressed as Ammonia mg/L-N.

11.0.4 For solid samples, the result is expressed as Ammonia mg/kg-N on a dry basis.

11.1 Accuracy

11.1.1 **ICV / CCV, LCS % Recovery**

$$\text{ICV / CCV, LCS \% Recovery} = \frac{\text{observed concentration}}{\text{known concentration}} \times 100$$

11.1.2 **MS % Recovery**

$$\text{MS \% Recovery} = \frac{(\text{spiked sample}) - (\text{unspiked sample})}{\text{spiked concentration}} \times 100$$

11.2 Precision (RPD)

Matrix Duplicate (MD)

$$\text{Matrix Duplicate (MD)} = \frac{|\text{orig. sample value} - \text{dup. sample value}|}{[(\text{orig. sample value} + \text{dup. sample value})/2]} \times 100$$

11.3 Concentration

$$\text{Concentration} = \text{mg/kg or L} = \frac{C \times V \times D}{W}$$

Where:
C = sample concentration in extract (ppm)
V = Volume of extract (mL)
D = Dilution Factor
W = Weight/Volume of sample aliquot extracted (grams or mLs)

NOTE: All dry weight corrections are made in LIMS at the time the final report is prepared.

12.0 Method Performance

12.1 Method Detection Limit Study (MDL)

Method Detection Limit: A valid method detection limit for each analyte of interest must be generated. The MDL must be below the reporting limit for each analyte. The procedure for determination of the method detection limit is given in 40 CFR Part 136, Appendix B. See TestAmerica SOP S-Q-003, “Method Detection Limit Studies,” current revision, for further guidance. Current TestAmerica Buffalo MDLs are maintained the QA department and are easily viewed in the laboratory LIMs system.

12.2 Demonstration of Capabilities

12.2.1 A one–time initial demonstration of performance for each individual method for both soils and water matrices must be generated.

12.2.2 This requires quadruplicate analysis of a mid–level check standard containing all of the standard analytes for the method using the same procedures used to analyze samples, including sample preparation.

12.2.3 Calculate the individual recovery for each analyte of interest.

12.2.4 Compare these results with the acceptance criteria given in the Method or to laboratory historical limits (if available).

12.2.5 Repeat the test for any analyte that does not meet the acceptance criteria. Repeated failure for any analyte indicates the need for the laboratory to evaluate the analytical procedure and take corrective action.

12.3 Training Requirements

12.3.1 The supervisor has the responsibility to ensure that this procedure is performed by an analyst who has been properly trained in its use and has the required experience.

12.3.2 The following analyst validation information is maintained for this method in the laboratory QA files.

12.3.3 The analyst must complete the laboratory safety orientation training that includes, but is not limited to, chemicals, PPE requirements, and electrical safety.

12.3.4 The analyst must read and understand this SOP.

12.3.5 The analyst must read and understand the Method used as reference for this SOP.

12.3.6 The analyst must complete a DOC or successfully analyze PT samples annually.

12.3.7 The analyst must complete the STL Quality Assurance Training.

13.0 Pollution Control

It is TestAmerica's policy to evaluate each method and look for opportunities to minimize waste generated (i.e., examine recycling options, ordering chemicals based on quantity needed, preparation of reagents based on anticipated usage and reagent stability). Employees must abide by the policies in Section 13 of the Corporate Environmental Health and Safety Manual (CW-E-M-001) for "Waste Management and Pollution Prevention."

14.0 Waste Management

Waste management practices are conducted consistent with all applicable rules and regulations. Excess reagents, samples and method process wastes are disposed of in an accepted manner. Waste description rules and land disposal restrictions are followed.

14.1 The following waste streams are produced when this method is carried out.

14.1.1 Basic waste to be disposed of in 'D' waste containers.
14.1.2 Following analysis all remaining sample volume shall be disposed in the "A" waste containers.

15.0 References / Cross-References

15.1 U.S. Environmental Protection Agency, Methods for Chemical Analysis of Water and Wastes, EPA-600/4-79-020, Revised March 1983, Method 350.1.

15.2 U.S. Environmental Protection Agency, 40 CFR, Part 36 Table 1B, footnote 6, 1994.

15.3 Lachat Quik Chem Method 10-107-06-1-B Revision Date March 13, 1998

16.0 Method Modifications:

N/A

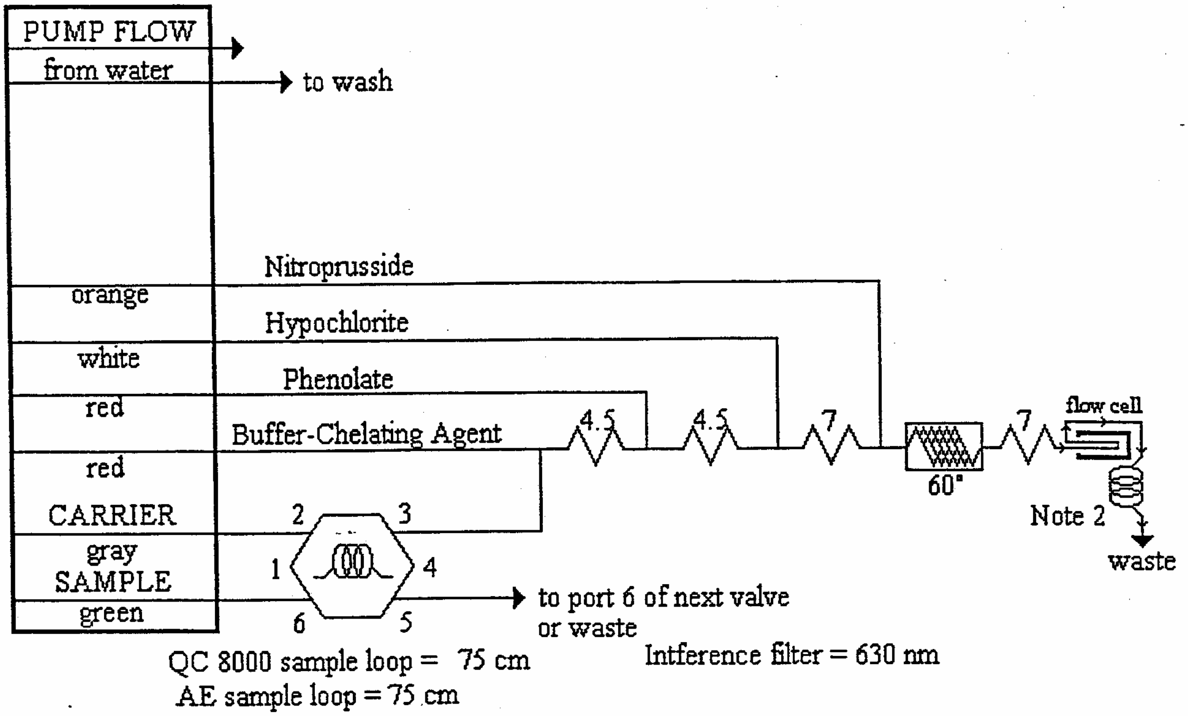
17.0 Attachments

17.1 Ammonia Manifold Program
17.2 Data System Parameters for Quikchem 8000
17.3 Calibration Curve
17.4 Analytical Batch
17.5 Wet Chemistry Batch Summary Sheet

18.0 Revision History

- Revision 0, dated June 9, 2008
    - Integration for TestAmerica operations.
    - Sec 4.3-Distillation of leachate/high interference samples
    - Updated Attachments 17.3-17.8
    - Quality Manager change, signature updated
    - Department Manager change, signature updated.
- Revision 1, dated January 27, 2010
    - Updated Attachments.

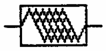
Attachment 17.1
Ammonia Manifold Program



CARRIER is Reagent 5.

Manifold tubing is **0.8 mm (0.032 in) i.d.** This is **5.2 uL/cm.**

4.5	is	**70**	cm of tubing on a 4.5 cm coil support
7	is	**135**	cm of tubing on a 7 cm coil support

APPARATUS: The  indicates 650 cm of tubing wrapped around the heater block at the specified temperature.

Note 1: TYGON PUMP TUBES MUST BE USED FOR THIS METHOD

Note 2: 200 cm x 0.022" i.d. backpressure loop.

Attachment 17.2
Data System Parameters for Quikchem 8000

The timing values listed below are approximate and will need to be optimized using graphical events programming.

Sample throughput:	60 samples/h, 60 s/sample
Pump seed:	35
Cycle Period:	60

Analyte Data:

Concentration Units:	mg N/L
Peak Base Width:	27.0 s
% Width Tolerance:	100
Threshold:	10000
Inject to Peak Start:	41.8 s
Chemistry:	Direct

Calibration Data:

Level	1	2	3	4	5	6	7
Concentration (mg/L)	2.0	1.0	0.5	0.2	.05	.02	0.0

Calibration Fit Type:	1st Order Polynomial
Calibration Rep. Handling:	Average
Weighting Method:	None
Concentration Scaling:	None
Force Through Zero:	No

Sampler Timing:

Min. Probe in Wash Period:	5.0 s
Probe in Sample Period:	24 s

Valve Timing:

Load Time:	0.0 s
Load Period:	15 s

Inject Period: 45 s

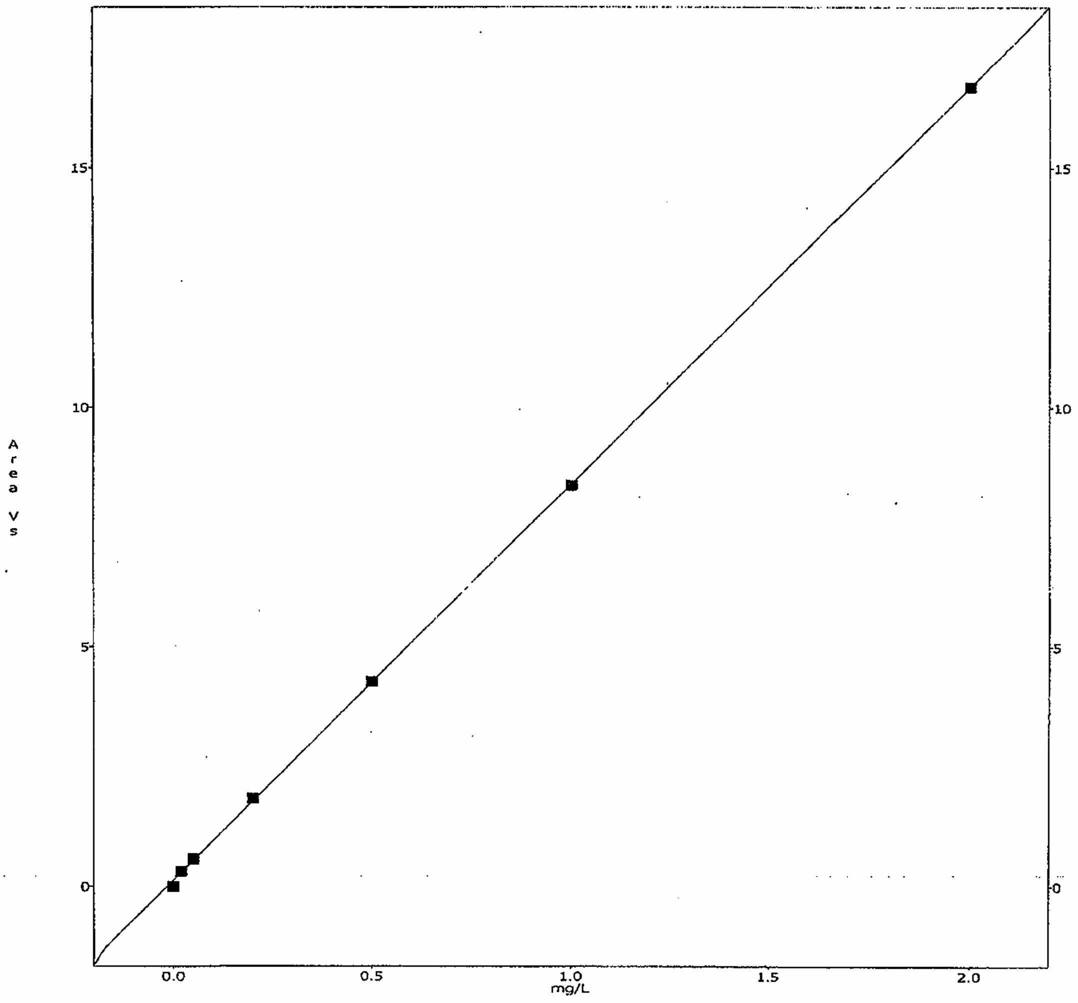
Attachment 17.3
Calibration Curve

Ammonia

Lvl	Area	mg/L	Rep 1	Rep 2	Rep 3	Rep 4	Rep 5	Replic STD	Replic % RSD	Residual 1st Poly
1	16688103	2.00	16688103					0.0	0.0	-0.0
2	8376660	1.00	8376660					0.0	0.0	0.3
3	4276212	0.50	4276212					0.0	0.0	-0.3
4	1841286	0.20	1841286					0.0	0.0	-3.8
5	578822	0.05	578822					0.0	0.0	-10.5
6	314176	0.02	314176					0.0	0.0	-16.4
7	0	0.00	0					0.0	0.0	

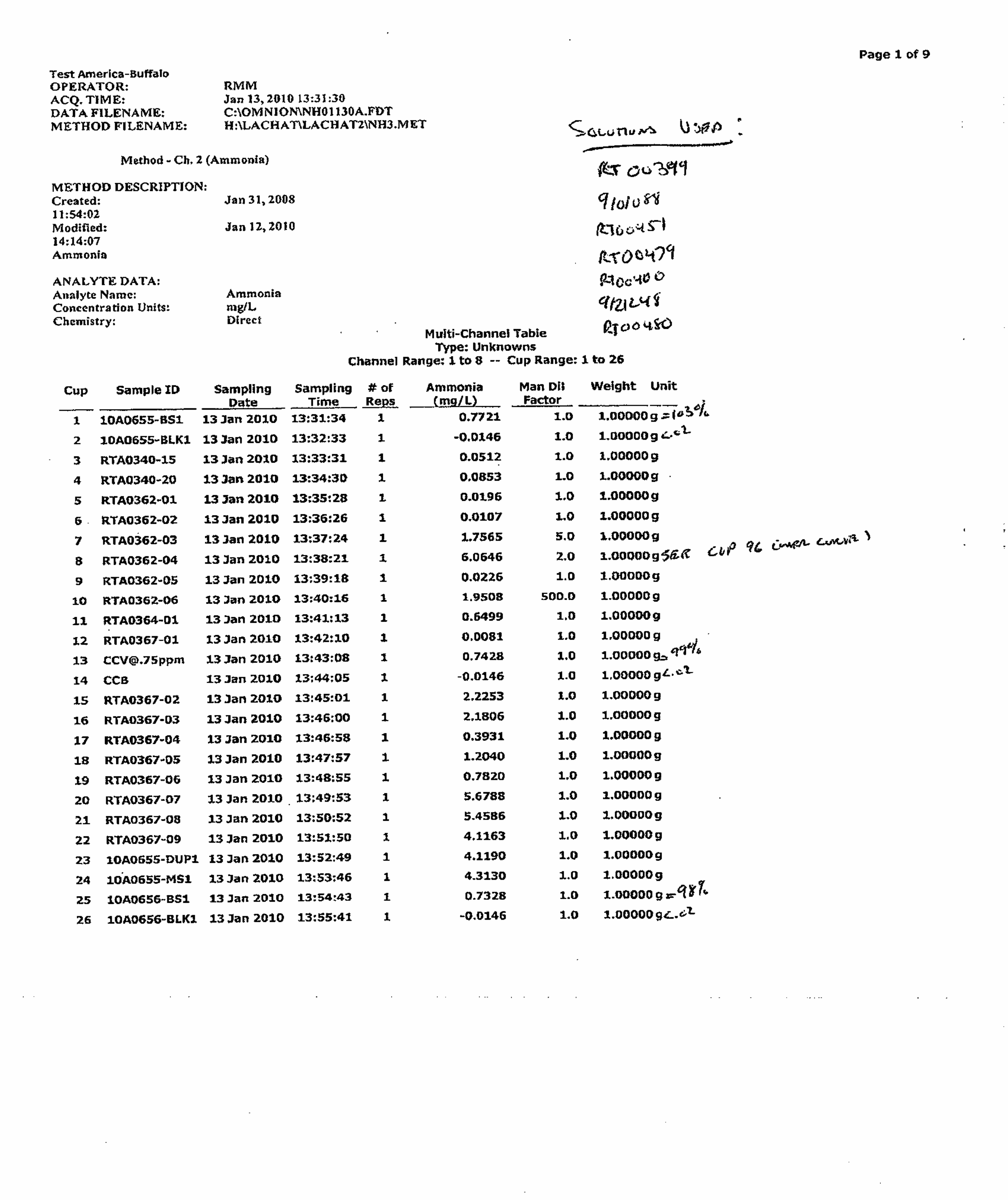
1st Order Poly
Conc = 1.207e-007 Area - 1.465e-002
r = 1.0000

Scaling: None - Weighting: None



Printed: Wednesday, January 13, 2010 - 04:01 PM

Attachment 17.4
Analytical Batch



Page 1 of 9

Test America-Buffalo
OPERATOR: RMM
ACQ. TIME: Jan 13, 2010 13:31:30
DATA FILENAME: C:\OMNION\NH01130A.FDT
METHOD FILENAME: H:\LACHAT\LACHAT2\NH3.MET

SOLUTIONS USED:
RT00399
9101088
RT00451
RT00479
RT00400
9121248
RT00480

Method - Ch. 2 (Ammonia)

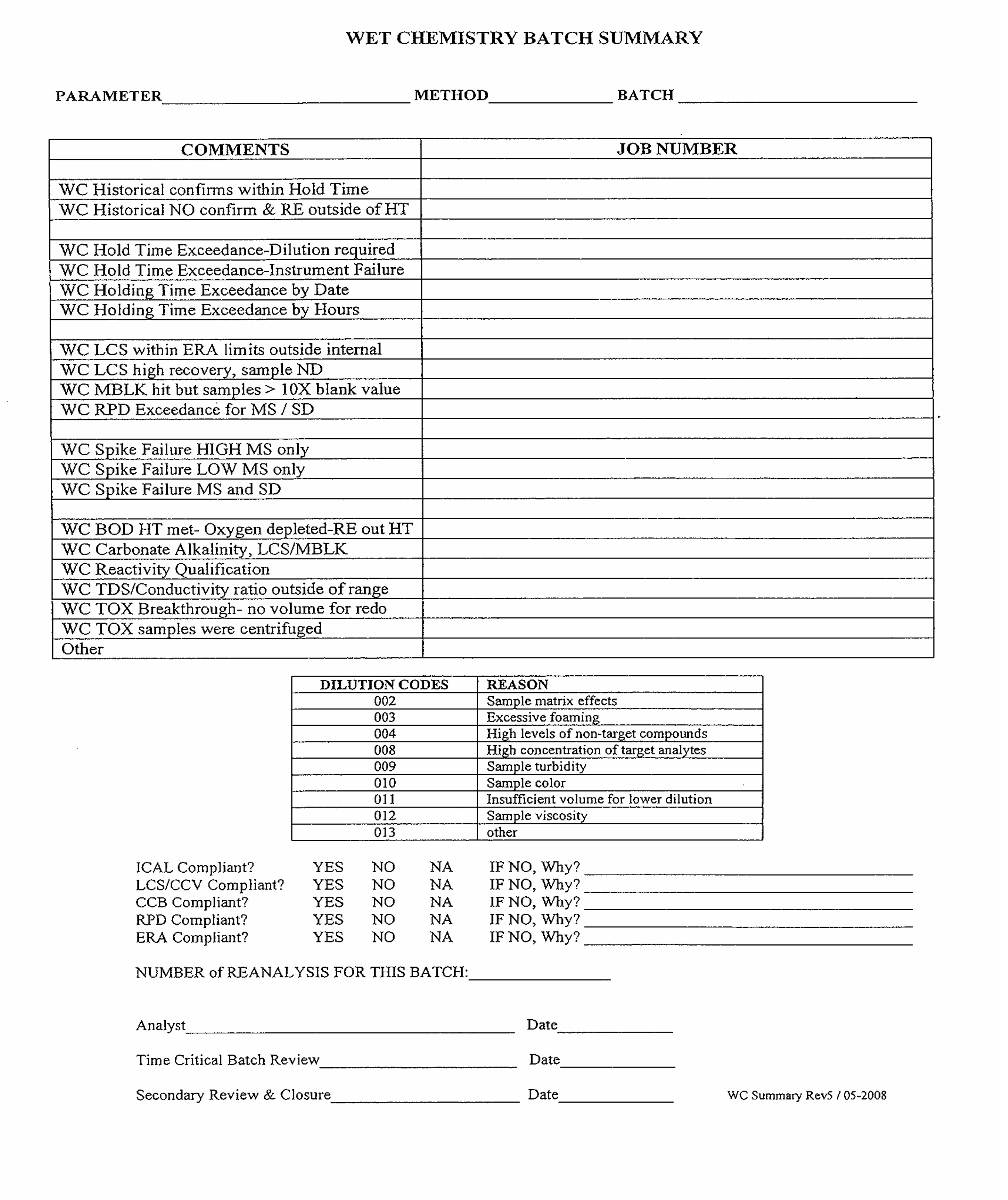
METHOD DESCRIPTION:
Created: Jan 31, 2008
11:54:02
Modified: Jan 12, 2010
14:14:07
Ammonia

ANALYTE DATA:
Analyte Name: Ammonia
Concentration Units: mg/L
Chemistry: Direct

Multi-Channel Table
Type: Unknowns
Channel Range: 1 to 8 -- Cup Range: 1 to 26

Cup	Sample ID	Sampling Date	Sampling Time	# of Reps	Ammonia (mg/L)	Man Dil Factor	Weight	Unit
1	10A0655-BS1	13 Jan 2010	13:31:34	1	0.7721	1.0	1.00000	g =103%
2	10A0655-BLK1	13 Jan 2010	13:32:33	1	-0.0146	1.0	1.00000	g <.02
3	RTA0340-15	13 Jan 2010	13:33:31	1	0.0512	1.0	1.00000	g
4	RTA0340-20	13 Jan 2010	13:34:30	1	0.0853	1.0	1.00000	g
5	RTA0362-01	13 Jan 2010	13:35:28	1	0.0196	1.0	1.00000	g
6	RTA0362-02	13 Jan 2010	13:36:26	1	0.0107	1.0	1.00000	g
7	RTA0362-03	13 Jan 2010	13:37:24	1	1.7565	5.0	1.00000	g
8	RTA0362-04	13 Jan 2010	13:38:21	1	6.0646	2.0	1.00000	g SEE CUP 96 LOWER CURVE)
9	RTA0362-05	13 Jan 2010	13:39:18	1	0.0226	1.0	1.00000	g
10	RTA0362-06	13 Jan 2010	13:40:16	1	1.9508	500.0	1.00000	g
11	RTA0364-01	13 Jan 2010	13:41:13	1	0.6499	1.0	1.00000	g
12	RTA0367-01	13 Jan 2010	13:42:10	1	0.0081	1.0	1.00000	g
13	CCV@.75ppm	13 Jan 2010	13:43:08	1	0.7428	1.0	1.00000	g = 99%
14	CCB	13 Jan 2010	13:44:05	1	-0.0146	1.0	1.00000	g <.02
15	RTA0367-02	13 Jan 2010	13:45:01	1	2.2253	1.0	1.00000	g
16	RTA0367-03	13 Jan 2010	13:46:00	1	2.1806	1.0	1.00000	g
17	RTA0367-04	13 Jan 2010	13:46:58	1	0.3931	1.0	1.00000	g
18	RTA0367-05	13 Jan 2010	13:47:57	1	1.2040	1.0	1.00000	g
19	RTA0367-06	13 Jan 2010	13:48:55	1	0.7820	1.0	1.00000	g
20	RTA0367-07	13 Jan 2010	13:49:53	1	5.6788	1.0	1.00000	g
21	RTA0367-08	13 Jan 2010	13:50:52	1	5.4586	1.0	1.00000	g
22	RTA0367-09	13 Jan 2010	13:51:50	1	4.1163	1.0	1.00000	g
23	10A0655-DUP1	13 Jan 2010	13:52:49	1	4.1190	1.0	1.00000	g
24	10A0655-MS1	13 Jan 2010	13:53:46	1	4.3130	1.0	1.00000	g
25	10A0656-BS1	13 Jan 2010	13:54:43	1	0.7328	1.0	1.00000	g =98%
26	10A0656-BLK1	13 Jan 2010	13:55:41	1	-0.0146	1.0	1.00000	g <.02

Attachment 17.5
Wet Chemistry Batch Summary Sheet



WET CHEMISTRY BATCH SUMMARY

PARAMETER______________________________ METHOD_______________ BATCH _____________________________

COMMENTS	JOB NUMBER
WC Historical confirms within Hold Time	
WC Historical NO confirm & RE outside of HT	
WC Hold Time Exceedance-Dilution required	
WC Hold Time Exceedance-Instrument Failure	
WC Holding Time Exceedance by Date	
WC Holding Time Exceedance by Hours	
WC LCS within ERA limits outside internal	
WC LCS high recovery, sample ND	
WC MBLK hit but samples > 10X blank value	
WC RPD Exceedance for MS / SD	
WC Spike Failure HIGH MS only	
WC Spike Failure LOW MS only	
WC Spike Failure MS and SD	
WC BOD HT met- Oxygen depleted-RE out HT	
WC Carbonate Alkalinity, LCS/MBLK	
WC Reactivity Qualification	
WC TDS/Conductivity ratio outside of range	
WC TOX Breakthrough- no volume for redo	
WC TOX samples were centrifuged	
Other	

DILUTION CODES	REASON
002	Sample matrix effects
003	Excessive foaming
004	High levels of non-target compounds
008	High concentration of target analytes
009	Sample turbidity
010	Sample color
011	Insufficient volume for lower dilution
012	Sample viscosity
013	other

ICAL Compliant? YES NO NA IF NO, Why? ____
LCS/CCV Compliant? YES NO NA IF NO, Why? ____
CCB Compliant? YES NO NA IF NO, Why? ____
RPD Compliant? YES NO NA IF NO, Why? ____
ERA Compliant? YES NO NA IF NO, Why? ____

NUMBER of REANALYSIS FOR THIS BATCH:_________________

Analyst____ Date______________

Time Critical Batch Review________________________ Date______________

Secondary Review & Closure_______________________ Date______________

WC Summary Rev5 / 05-2008

Title: Total Phosphorus and Orthophosphate
Method Nos. EPA 365.2 / SM4500-P E

Once printed, this is considered an uncontrolled document

Approvals (Signature/Date):

Lisa R Matecki 01/28/10
Lisa R. Matecki Date
Department Supervisor

Kenneth E. Kasperek 01/28/10
Kenneth Kasperek Date
Health & Safety Manager / Coordinator

Peggy Gray-Erdmann 01/28/10
Peggy Gray-Erdmann Date
Quality Assurance Manager

01/28/10
Christopher Spencer Date
Laboratory Director

Facility Distribution No. ___________ **Distributed To:**________________________

1.0 Scope and Application

1.1 Analytes, Matrix(s), and Reporting Limits

This method covers the determination of specified forms of phosphorus in drinking, surface and saline waters, and domestic and industrial wastes. The reporting limit for this method is 0.01mg P/L.

2.0 Summary of Method

Ammonium molybdate and antimony potassium tartrate react in an acid medium with dilute solutions of phosphorus to form a heteropoly acid-phosphomolybdic acid that is reduced to an intensely colored molybdem blue by ascorbic acid.

Orthophosphate forms a blue color in this test. Polyphosphates, and some organic phosphorus compounds, may be converted to the orthophosphate form by sulfuric acid hydrolysis. Organic phosphorus compounds may be converted to the orthophosphate form by persulfate digestion.

3.0 Definitions

3.1 Total Phosphorus - All of the phosphorus present in the sample, regardless of form, as measured by the persulfate digestion procedure.

3.2 Total Orthophosphate - Inorganic phosphorus [(PO4)-3] in the sample as measured by the direct colorimetric analysis procedure.

3.3 Total Organic Phosphorus - Phosphorus in the sample measured by the persulfate digestion procedure and minus hydrolyzable phosphorus and orthophosphate.

3.4 Dissolved Phosphorus -All of the phosphorus present in the filtrate of a sample filtered through a 0.45-micron membrane filter. Membrane filters must be soaked in distilled water before use so as not to contribute significant amounts of phosphorus to samples.

3.5 Dissolved Orthophosphate - As measured by the direct colorimetric analysis procedure in the filtrate of a sample filtered through a 0.45-micron membrane filter.

3.6 Dissolved Organic Phosphorus- As measured by the persulfate digestion procedure and minus dissolved hydrolyzable phosphorus and orthophosphate.

4.0 Interferences

4.1 Arsenates react with the molybdate reagent to produce a blue color similar to that formed with phosphate.

4.2 Hexavalent chromium and NO interfere to give results about 3% low at concentrations of 1 mg/L and 10 to 15% low at 10 mg/L.

4.3 Sulfide and silicate do not interfere at concentrations of 1.0 and 10 mg/L.

4.4 If samples are turbid an absorbance blank may be used.

5.0 Safety

Employees must abide by the policies and procedures in the Corporate Safety Manual, Radiation Safety Manual and this document.

5.1 SPECIFIC SAFETY CONCERNS OR REQUIREMENTS: None

5.2 Primary Materials Used

The following is a list of the materials used in this method, which have a serious or significant hazard rating. **Note: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the MSDS for each of the materials listed in the table.** A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the MSDS for each material before using it for the first time or when there are major changes to the MSDS.

Material (1)	Hazards	Exposure Limit (2)	Signs and symptoms of exposure
Potassium Persulfate	Oxidizer	None	Causes irritation to the respiratory tract. Symptoms may include coughing, shortness of breath. Causes irritation to skin and eyes. Symptoms include redness, itching, and pain. May cause dermatitis, burns, and moderate skin necrosis.
Sodium Hydroxide	Corrosive	2 Mg/M3-Ceiling	Severe irritant. Effects from inhalation of dust or mist vary from mild irritation to serious damage of the upper respiratory tract, depending on severity of exposure. Symptoms may include sneezing, sore throat or runny nose. Contact with skin can cause irritation or severe burns and scarring with greater exposures. Causes irritation of eyes, and with greater exposures it can cause burns that may result in permanent impairment of vision even blindness.
Sulfuric Acid	Corrosive Oxidizer Dehydrator Poison Carcinogen	1 Mg/M3-TWA	Inhalation produces damaging effects on the mucous membranes and upper respiratory tract. Symptoms may include irritation of the nose and throat, and labored breathing. Symptoms of redness, pain, and severe burn can occur. Contact can cause blurred vision, redness, pain and severe tissue burns. Can cause blindness.
1 – Always add acid to water to prevent violent reactions.			
2 – Exposure limit refers to the OSHA regulatory exposure limit.			

6.0 Equipment and Supplies

6.1 Instrumentation

- Spectrophotometer suitable for measurements at 880nm for ortho-phosphorus and 880nm for total phosphorus with a light path of 1 cm or longer.
- COD reactor

6.2 Supplies

- Glass test tubes for ortho-phosphorus analyses.
- Acid-washed glassware: all glassware used should be washed with hot 1:1 HCl and rinsed with distilled water to remove the last traces of phosphorus that might be adsorbed on the glassware. Commercial detergents should never be used.

7.0 Reagents and Standards

7.1 Orthophosphate

7.1.1 PhosVer 3 Phosphate Reagent Powder Pillows (#21060-69), 10ml purchased from HACH.

7.1.2 Two different Phosphorus stock standards (1000ppm) purchased from Ultra Scientific and SCP Science.

7.1.3 Intermediate Standard phosphorus solutions (1.0ppm), dilute 1.0ml of stock standards (see 7.1.2) into 1000 ml of DiH2O. These are at a 1.0ppm concentration where one volume is used for the quality controls and the other is used for the spikes.

7.1.4 ICV/LCS is at 0.2ppm: 2.0ml of 1.0ppm Phosphate standard (see 7.1.3) and 8.0ml of deionized water.

7.1.5 CCV is at 0.5ppm: 5.0ml of 1.0ppm Phosphate standard (see 7.1.3) and 5.0ml of deionized water.

7.1.6 Spikes are at 1.0ppm: 5.0mls of 1.0ppm Phosphate standard (see 7.1.3) is always added to 5.0mls of sample.

7.2 Total Phosphorus

7.2.1 Total and Acid hydrolyzable test tube reagent set purchased by HACH containing: PhosVer 3 phosphate reagent powder pillows, potassium persulfate powder pillows, Sodium hydroxide solution (1.54 N), and Total and acid hydrolyzed test vials.

7.2.2 Two different Phosphorus stock standards (1000ppm) purchased from Ultra Scientific and SCP Science.

7.2.3 Intermediate Standard phosphorus solutions (1.0ppm), dilute 1.0ml of stock standards (see 7.2.2) into 1000 ml of DiH2O. One is used for the quality controls and the other is used for the spikes

7.2.4 ICV/LCS is at 0.2ppm: 1.0ml of 1.0ppm Phosphate standard (see 7.2.3) and 4.0ml of deionized water.

7.2.5 CCV is at 0.5ppm: 2.5ml of 1.0ppm Phosphate standard (see 7.2.3) and 2.5ml of deionized water.

7.2.6 Spikes are at 0.5ppm: 1.25-mL of 1.0ppm Phosphate standard (see 7.2.3) is added to 2.5-mLs of sample and brought to final volume of 5.0-mL with diH2O.

7.2.7 Soil Quality Control (ERA): Approximately 0.1500g of soil added to 5.0ml of deionized water

8.0 Sample Collection, Preservation, Shipment and Storage

Sample container, preservation techniques and holding times may vary and are dependent on sample matrix, method of choice, regulatory compliance, and/or specific contract or client requests. Listed below are the holding times and the references that include preservation requirements.

Total Phosphorus:

Matrix	Sample Container	Min. Sample Size	Preservation	Holding Time [1]	Reference
Waters	HDPE	50 mLs	H2SO$_4$, pH < 2; Cool 4 ± 2°C	28 Days	40 CFR Part 136
Soils	Glass	3 grams	Cool 4 ± 2°C	180 Days	N/A

[1] Inclusive of digestion and analysis.

Orthophosphate:

Matrix	Sample Container	Min. Sample Size	Preservation	Holding Time [1]	Reference
Waters	HDPE	50 mLs	Cool 4 ± 2°C	48 Hours	40 CFR Part 136
Soils	Glass	3 grams	Cool 4 ± 2°C	180 Days	N/A

[1] Inclusive of digestion and analysis.

9.0 Quality Control

9.1 Sample QC - The following quality control samples are prepared with each batch of samples.

Quality Controls	Frequency	Control Limit
Method Blank (MBLK)	1 in 20 or fewer samples	< Rpt. Limit
Laboratory Control Sample (LCS)	1 in 20 or fewer samples	Statistical Limits [3]
Matrix Duplicate (MD)	1 in 20 or fewer samples	Statistical Limits [3]
Matrix Spike (MS)[1]	1 in 20 or fewer samples	Statistical Limits [3]
MS Duplicate (SD) [1]	1 in 20 or fewer samples	Statistical Limits [3]

[1] The samples selected for MS/SD are random, unless specifically requested by a client.
[2] Analytical and QC samples (MB, LCS, MS/SD)
[3] Statistical control limits are updated annually and are updated into LIMS.

9.2 Instrument QC

9.2.1 Initial Calibration Verification (ICV): The ICV is prepared from a source other than that used to prepare the calibration curve. ICV is at 0.2 ppm

9.2.2 Laboratory Control Standard (LCS): Prepare a LCS at a concentration of 0.2 ppm at the beginning of each analytical procedure. All further LCS are analyzed at a concentration of 0.5ppm after every ten samples and at the end of the procedure.

9.2.3 Calibration Acceptance Summary:

Step	*Standards*	*Type*	*Control Limit*	*Frequency*
Method #365.2				
Initial Cal	0.0, 0.01, 0.02, 0.06, 0.1, 0.2, 0.4, 0.6, 0.8, 1.0	Type of Cal: Linear	$r \geq 0.990$	Every three months, unless necessary sooner.
ICV	0.2ppm		90-110%	Immediately after calibration
ICB	0.0ppm		< Detection Limit	Immediately after calibration
LCS	0.5ppm		90-110%	Once every 10 samples
MBLK	0.0		< Detection Limit	Once every 10 samples

10.0 Procedure

10.1 Sample Preparation

Matrix	**Sample Size**
Waters	~50 mLs, of sample or dilution
Soils/Wastes	10 gram / 100 mLs DI water [1]

[1] More sample should be used if total solids are very low. Mix for 10 minutes, then filter and analyze the filtrate.

10.2 Calibration

10.2.1 A separate calibration curve is run every three months for Ortho-Phosphorus and Total Phosphorus.

10.2.2 Prepare the calibration standards by diluting the stock phosphorus solutions (7.1.2/7.2.2).

TOTAL PHOSPHORUS

ml of Intermediate phosphorus standard (7.2.3)	ml diH2O	Conc. mg/L
0.00	5.0	0.00
0.05	4.95	0.01
0.10	4.9	0.02
0.3	4.7	0.06
0.5	4.5	0.10
1.0	4.0	0.20
2.0	3.0	0.40
3.0	2.0	0.60
4.0	1.0	0.80
5.0	0.0	1.0

ORTHO PHOSPHATE

ml of Intermediate phosphorus standard (7.1.3)	ml diH2O	Conc. mg/L
0	10	0.00
0.10	9.9	0.01
0.20	9.8	0.02
0.60	9.4	0.06
1.00	9.0	0.10
2.00	8.0	0.20
4.00	6.0	0.40
6.00	4.0	0.60
8.00	2.0	0.80
10.00	0.0	1.00

10.3 Sample Analysis

Total Phosphorus

10.3.1 Aqueous sample: Pipette 5.0ml of sample into a total and hydrolyzed test vial. Soil Sample: Transfer approximately 0.1500g of soil sample into a total and hydrolyzed test vial with 5.0ml of deionized-water.

10.3.2 Add the contents of one Potassium persulfate powder pillow into each vial. Cap tightly and shake to dissolve.

10.3.3 Heat vials for 30 minutes @ 150^{o}C +/- 2^o on a pre-heated COD reactor.
Temperature must be recorded on spreadsheet

10.3.4 Carefully remove the vials from the reactor and allow to cool to room temperature.

10.3.5 Pipette 2ml of 1.54N Sodium hydroxide to each vial.

10.3.6 Add the contents of one PhosVer3 Phosphate reagent pillow to each vial. Cap tightly and shake for 10-15 seconds.

10.3.7 Let stand for 8 to 10 minutes, and then read on spectrophotometer at 880nm.

NOTE: If sample is turbid, read on spec after step 10.3.5 and record value, and then continue on to remaining steps.

Orthophosphate

10.3.8 A blank correction must be done for all colored samples. To blank correct the samples fill a sample cell with 10ml of sample (the blank correction). This sample is not spiked nor do you add the Phosver3 phosphate Powder Pillow packet.

10.3.9 Add 10ml of sample to glass test tube.

10.3.10 Place the blank correction sample into the cell holder and record the absorbance.

10.3.11 Add the contents of one Phosver3 phosphate Powder Pillow packet to each sample tube and invert to mix.

10.3.12 After a reaction period of two minutes, measure the color absorbance of each sample at 880nm with a spectrophotometer, using the reagent blank as the reference solution.

11.0 Calculations / Data Reduction

11.1 Obtain concentration value of sample directly from prepared standard curve. Report results as P, mg/l.

11.2 When phosphate is requested as Phosphate as PO4, analyze the sample as you would total phosphorus, and multiply the result by 3.065. This becomes the phosphate result. If the data is auto uploaded into LIMS, you need not multiply the result by 3.065, LIMS will do this for you.

11.3 If absorbance is above the highest point on curve, a dilution may be necessary.

11.4 Accuracy

ICV / CCV, LCS % Recovery

$$\text{ICV / CCV, LCS \% Recovery} = \frac{\text{observed concentration}}{\text{known concentration}} \times 100$$

MS % Recovery

$$\text{MS \% Recovery} = \frac{\text{(spiked sample) - (unspiked sample)}}{\text{spiked concentration}} \times 100$$

11.5 Precision (RPD)

Matrix Duplicate (MD)

$$\text{Matrix Duplicate (MD)} = \frac{|\text{orig. sample value - dup. sample value}|}{[(\text{orig. sample value + dup. sample value})/2]} \times 100$$

11.6 Concentration

$$\text{Concentration} = \text{mg/kg or L} = \frac{C \times V \times D}{W}$$

Where:
C = sample concentration in extract (ppm)
V = Volume of extract (mL)
D = Dilution Factor

W = Weight/Volume of sample aliquot extracted (grams or mLs)

NOTE: All dry weight corrections are made in LIMS at the time the final report is prepared.

12.0 Method Performance

12.1 Method Detection Limit Study (MDL)

The method detection limit (MDL) is the lowest concentration that can be detected for a given analytical method and sample matrix with 99% confidence that the analyte is present. The procedure for determination of the MDL is found in 40 CFR Part 136, Appendix B. See TestAmerica SOP S-Q-003, “Method Detection Limit Studies”, current revision for further guidance. MDLs reflect a calculated (statistical) value determined under ideal laboratory conditions in a clean matrix, and may not be achievable in all environmental matrices. The laboratory maintains MDL studies for analyses performed; these are verified at least annually unless method requirements require a greater frequency.

12.2 Demonstration of Capabilities

12.2.1 A one–time initial demonstration of performance for each individual method for both soils and water matrices must be generated.

12.2.2 This requires quadruplicate analysis of a mid–level check standard containing all of the standard analytes for the method using the same procedures used to analyze samples, including sample preparation.

12.2.3 Compare these results with the acceptance criteria given in the method, or to laboratory historical limits (if available).

12.2.4 Repeat the test for any analyte that does not meet the acceptance criteria. Repeated failure for any analyte indicates the need for the laboratory to evaluate the analytical procedure and take corrective action.

12.3 Training Requirements

12.3.1 The supervisor has the responsibility to ensure that this procedure is performed by an analyst who has been properly trained in its use and has the required experience.

12.3.2 The following analyst validation information is maintained for this method in the laboratory QA files:

- The analyst must complete the laboratory safety orientation training that includes, but is not limited to, chemicals, PPE requirements, and electrical safety.

- The analyst must read and understand this SOP.

- The analyst must read and understand the Method used as reference for this SOP.

- The analyst must complete a DOC or successfully analyze PT samples annually.

- The analyst must complete the TestAmerica Quality Assurance Training.

13.0 Pollution Control

It is TestAmerica’s policy to evaluate each method and look for opportunities to minimize waste generated (i.e., examine recycling options, ordering chemicals based on quantity needed, preparation of reagents based on anticipated usage and reagent stability). Employees must abide by the policies in Section 13 of the Corporate Environmental Health and Safety Manual (CW-E-M-001) for “Waste Management and Pollution Prevention.”

14.0 Waste Management

Waste management practices are conducted consistent with all applicable rules and regulations. Excess reagents, samples and method process wastes are disposed of in an accepted manner. Waste description rules and land disposal restrictions are followed. The following waste streams are produced when this method is carried out.

- Acidic sample waste generated by the analysis. Dispose of this waste in the “A” waste container.

- Contaminated disposable glass or plastic materials utilized in the analysis. Empty the contents of the glassware into the “A” waste and dispose of the glassware in the recycling bins located throughout the lab.

15.0 References / Cross-References

15.1 Standard Methods for the Examination of Water and Wastewater, 20th Edition 4500-P E

15.2 EPA Methods for Chemical Analysis of Water and Wastes, Method 365.2

16.0 Method Modifications:

Item	Method	Modification
10.3.7	EPA 365.2 SM4500-P E	As per HACH method sample absorbance is read back after a reaction time of 8-10 minutes.
10.3.12	EPA 365.2 SM4500-P E	As per HACH method sample absorbance is read back after a reaction time of 2 minutes.

17.0 Attachments

17.1 Analytical Run Sequence

17.2 Analytical Batch

17.3 Wet Chemistry Batch Summary

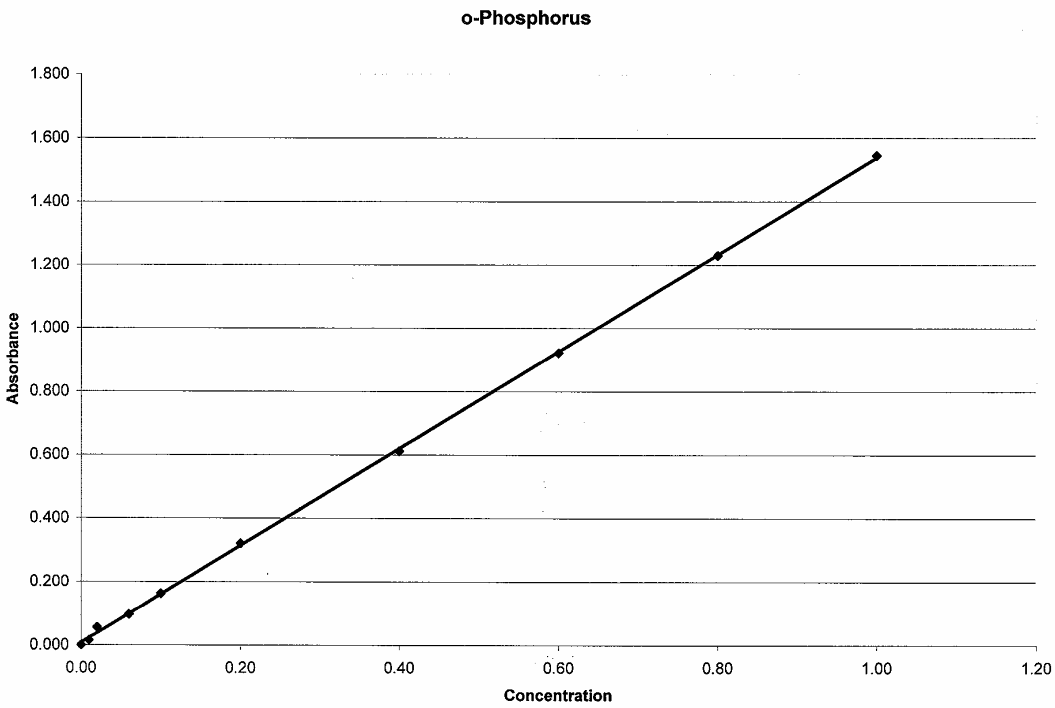
18.0 Revision History

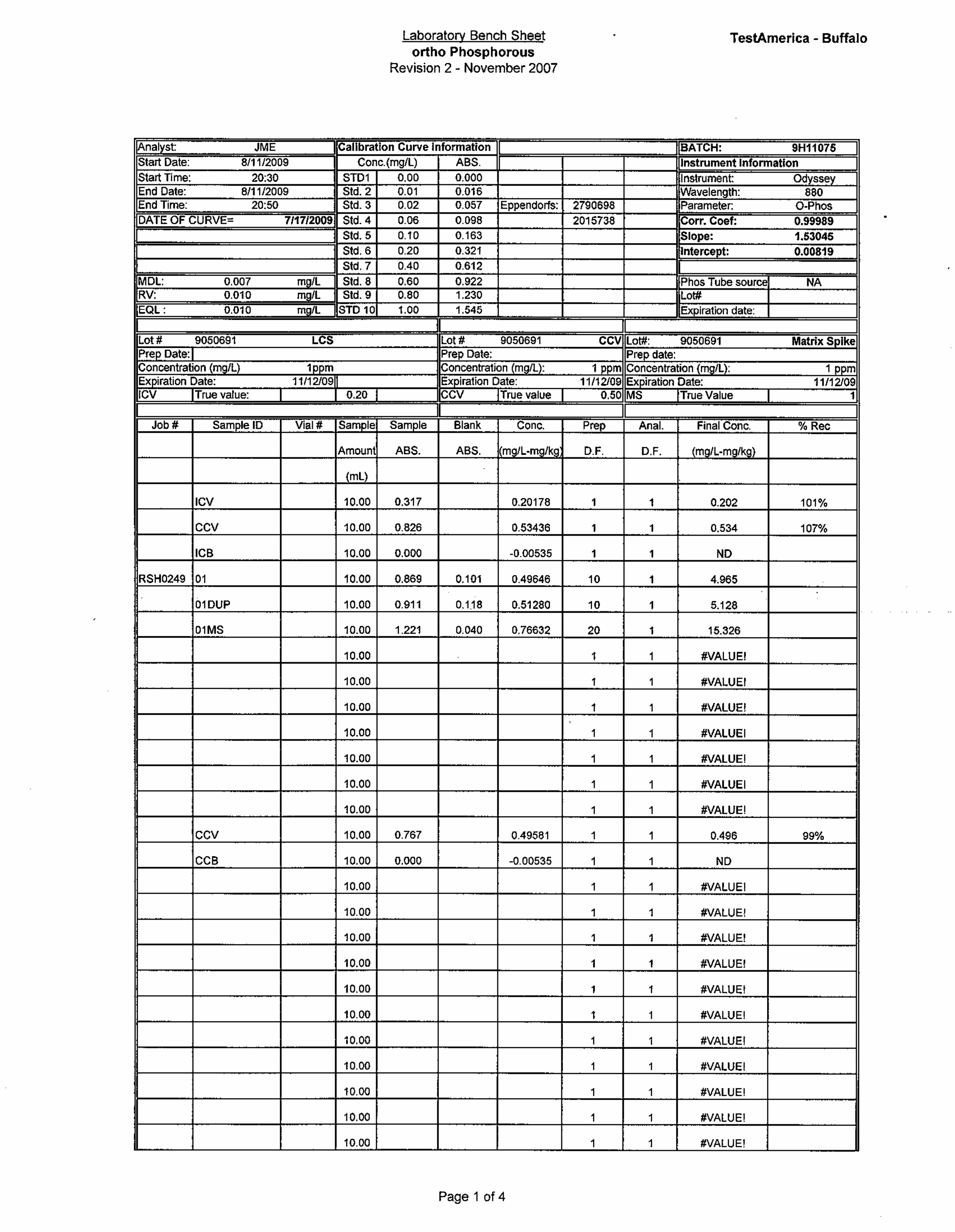
- Revision 0, dated 04 September 2008
  - Integration for TestAmerica operations
  - Department and QA manager changed, signatures updated
- Revision 1, 27 August 2009
  - Clearly defined Dissolved Orthophosphate
  - Updated Attachment 17.2
- Revision 2, 27 January 2010
  - Minor typographical errors

Attachment 17.1
Analytical Run Sequence

ICV
ICB
Sample
Sample
Sample
Sample
Sample
Sample
Sample
Sample
Sample
Sample
LCS
MBLK
Sample
Sample
Sample
Sample
Sample
Sample
Sample
Sample
Sample duplicate (MD)
Sample spike (MS)
LCS
MBLK

Attachment 17.2
Analytical Batch – Orthophosphate





Laboratory Bench Sheet
ortho Phosphorous
Revision 2 - November 2007

TestAmerica - Buffalo

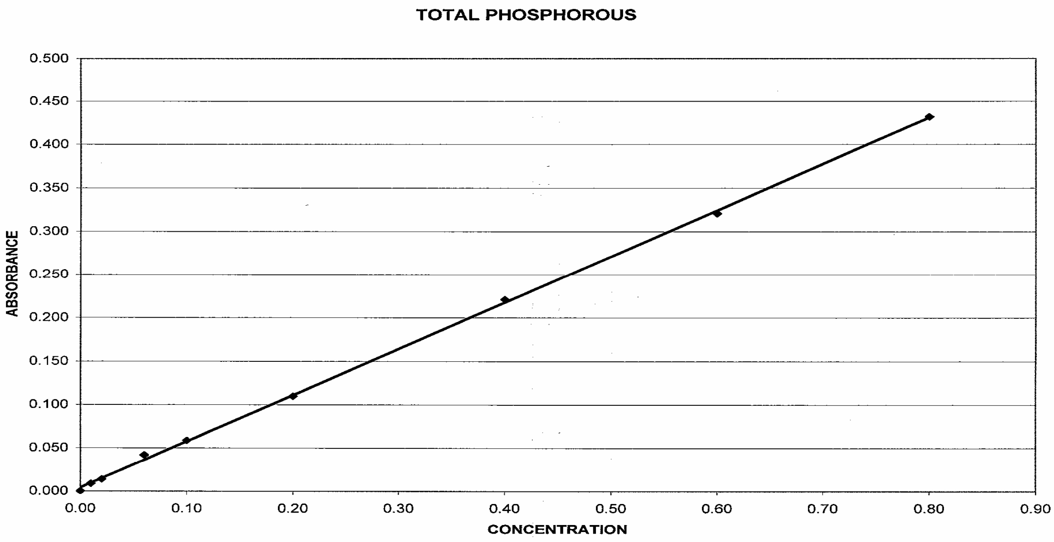
Analyst:	JME		**Calibration Curve Information**						**BATCH:**	**9H11075**
Start Date:	8/11/2009		Conc.(mg/L)		ABS.				**Instrument Information**	
Start Time:	20:30		STD1	0.00	0.000				Instrument:	Odyssey
End Date:	8/11/2009		Std. 2	0.01	0.016				Wavelength:	880
End Time:	20:50		Std. 3	0.02	0.057	Eppendorfs:	2790698		Parameter:	O-Phos
DATE OF CURVE=	**7/17/2009**		Std. 4	0.06	0.098		2015738		**Corr. Coef:**	**0.99989**
			Std. 5	0.10	0.163				**Slope:**	**1.53045**
			Std. 6	0.20	0.321				**Intercept:**	**0.00819**
			Std. 7	0.40	0.612					
MDL:	0.007	mg/L	Std. 8	0.60	0.922				Phos Tube source	NA
RV:	0.010	mg/L	Std. 9	0.80	1.230				Lot#	
EQL :	0.010	mg/L	STD 10	1.00	1.545				Expiration date:	

Lot #	9050691	**LCS**	Lot #	9050691	**CCV**	Lot#:	9050691	**Matrix Spike**
Prep Date:			Prep Date:			Prep date:		
Concentration (mg/L)	1ppm		Concentration (mg/L):	1 ppm		Concentration (mg/L):	1 ppm	
Expiration Date:	11/12/09		Expiration Date:	11/12/09		Expiration Date:	11/12/09	
ICV	True value:	0.20	CCV	True value	0.50	MS	True Value	1

Job #	Sample ID	Vial #	Sample Amount (mL)	Sample ABS.	Blank ABS.	Conc. (mg/L-mg/kg)	Prep D.F.	Anal. D.F.	Final Conc. (mg/L-mg/kg)	% Rec
	ICV		10.00	0.317		0.20178	1	1	0.202	101%
	CCV		10.00	0.826		0.53436	1	1	0.534	107%
	ICB		10.00	0.000		-0.00535	1	1	ND	
RSH0249	01		10.00	0.869	0.101	0.49646	10	1	4.965	
	01DUP		10.00	0.911	0.118	0.51280	10	1	5.128	
	01MS		10.00	1.221	0.040	0.76632	20	1	15.326	
			10.00				1	1	#VALUE!	
			10.00				1	1	#VALUE!	
			10.00				1	1	#VALUE!	
			10.00				1	1	#VALUE!	
			10.00				1	1	#VALUE!	
			10.00				1	1	#VALUE!	
			10.00				1	1	#VALUE!	
	CCV		10.00	0.767		0.49581	1	1	0.496	99%
	CCB		10.00	0.000		-0.00535	1	1	ND	
			10.00				1	1	#VALUE!	
			10.00				1	1	#VALUE!	
			10.00				1	1	#VALUE!	
			10.00				1	1	#VALUE!	
			10.00				1	1	#VALUE!	
			10.00				1	1	#VALUE!	
			10.00				1	1	#VALUE!	
			10.00				1	1	#VALUE!	
			10.00				1	1	#VALUE!	
			10.00				1	1	#VALUE!	
			10.00				1	1	#VALUE!	

Page 1 of 4

Analytical Batch –Total Phosphorus



Laboratory Bench Sheet

Total Phosphorous

Revision 2 - November 2007

TestAmerica - Buffalo

PHOSPHORO

Analyst:	JM
Start Date:	08/14/2009
Start Time:	08:10
End Date:	08/14/2009
End Time	08:40
DATE OF CURVE=	07/21/09
EQL:	0.01 mg/L

Calibration Curve Information		
	Conc.(mg/L)	ABS.
STD1	0.00	0.000
Std. 2	0.01	0.009
Std. 3	0.02	0.014
Std. 4	0.06	0.042
Std. 5	0.10	0.059
Std. 6	0.20	0.110
Std. 7	0.40	0.221
Std. 8	0.60	0.321
Std 9	0.80	0.432
Std 10	1.00	0.534

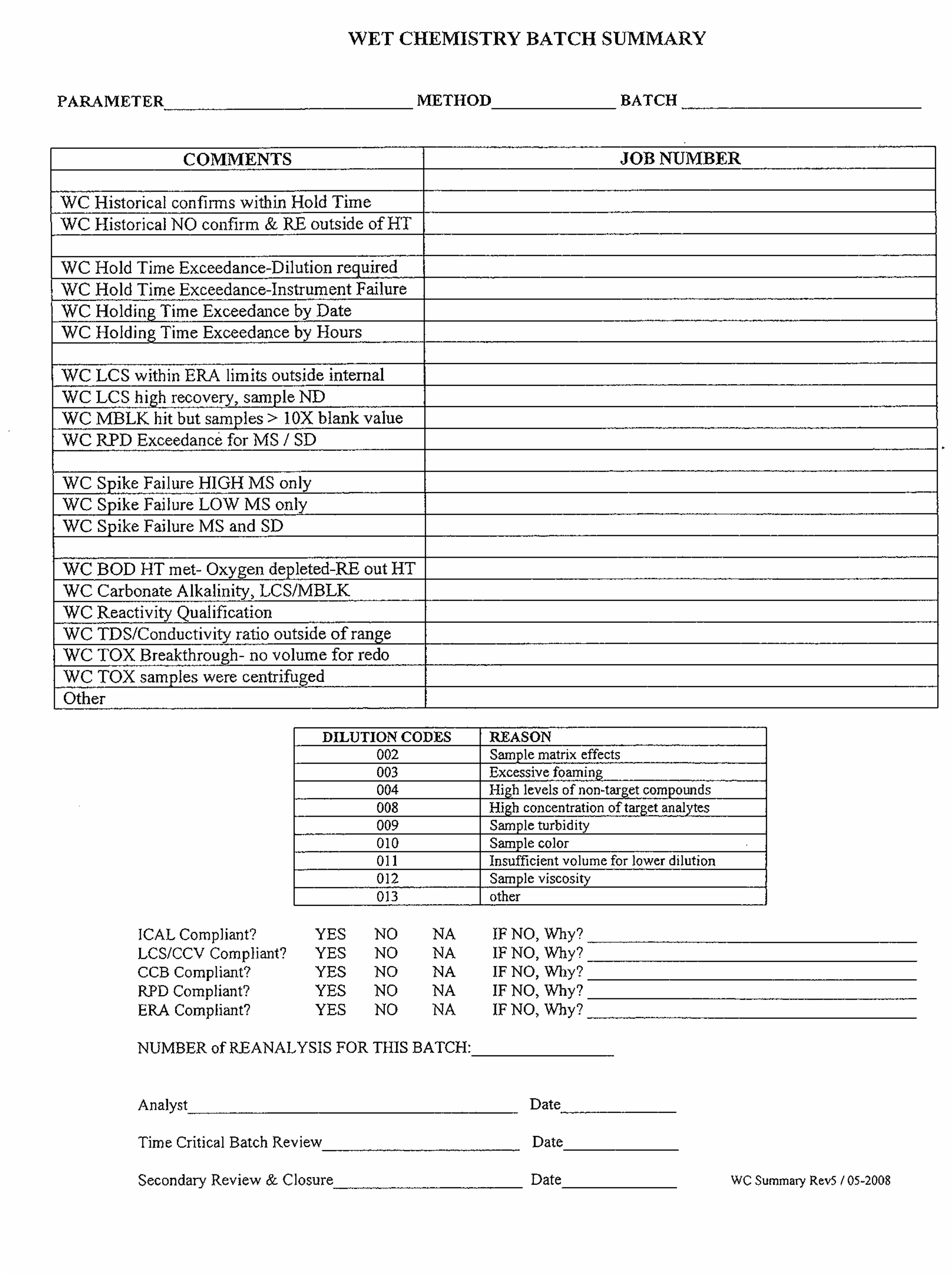
Reactor Temperatu	
Reactor #1	
Reactor #2	150
Reactor #3	
Reactor #4	
CAI Reactor	
3383527	

BATCH #	**9H14014**
Instrument Information	
Instrument:	Odyssey
Wavelength:	880
Parameter:	Total Phosp
Corr. Coef:	**0.99987**
Slope:	**0.53130**
Intercept:	**0.00472**
Phos Tube source:	
Lot#	9061788
Expiration date:	6/2011

LCS Information:		
Lot #		9050691
Concentration (mg/L)		1.0
LCS	True value	0.2
CCV Information:		
Lot #		9050691
Concentration (mg/L):		1.0
CCV	True value	0.5
Matrix Spike Information:		
Lot#:		9050691
Concentration (mg/L):		1.0
MS	True Value	0.5

P-Code	Job #	Sample ID	Vial #	Sample vol.	Blank Absor	Sample Abs.	Conc. (mg/l)	Prep DF	Final Conc. mg/L	% Rec.	Start Date	Start Time
T7723-14-0	0.2 ppm	LCS	1	5.00		0.109	0.19628	1	0.1963	98%	08/14/2009	08:10
T7723-14-0		MBLK	2	5.00		0.000	-0.00888	1	-0.0089		08/14/2009	08:10
T7723-14-0	RSH0275	1	3	1.00		0.161	0.29416	5	1.4708		08/14/2009	08:10
T7723-14-0	RSH0292	1	4	2.50		0.812	1.51946	2	3.0389		08/14/2009	08:10
7778-77-0	RSH0335	1	5	2.50		0.433	0.80611	2	4.9415		08/14/2009	08:10
T7723-14-0	RSH0360	1	6	5.00		0.000	-0.00888	1	-0.0089		08/14/2009	08:10
7778-77-0	RSH0363	1	7	2.50		0.402	0.74776	2	4.5838		08/14/2009	08:10
T7723-14-0	RSH0367	1	8	5.00		1.626	3.05156	1	3.0516		08/14/2009	08:10
T7723-14-0		1MD	9	5.00		1.635	3.06850	1	3.0685		08/14/2009	08:10
T7723-14-0		1MS	10	2.50		1.104	2.06906	2	4.1381		08/14/2009	08:10
T7723-14-0			11	5.00				1	#VALUE!		08/14/2009	08:10
T7723-14-0			12	5.00				1	#VALUE!		08/14/2009	08:10
T7723-14-0	0.5 ppm	CCV	13	5.00		0.277	0.51249	1	0.5125	102%	08/14/2009	08:10
T7723-14-0	CCB	CCB	14	5.00		0.000	-0.00888	1	-0.0089		08/14/2009	08:10

Attachment 17.3
Wet Chemistry Batch Summary



WET CHEMISTRY BATCH SUMMARY

PARAMETER____________________________ METHOD______________ BATCH ___________________________

COMMENTS	JOB NUMBER
WC Historical confirms within Hold Time	
WC Historical NO confirm & RE outside of HT	
WC Hold Time Exceedance-Dilution required	
WC Hold Time Exceedance-Instrument Failure	
WC Holding Time Exceedance by Date	
WC Holding Time Exceedance by Hours	
WC LCS within ERA limits outside internal	
WC LCS high recovery, sample ND	
WC MBLK hit but samples > 10X blank value	
WC RPD Exceedance for MS / SD	
WC Spike Failure HIGH MS only	
WC Spike Failure LOW MS only	
WC Spike Failure MS and SD	
WC BOD HT met- Oxygen depleted-RE out HT	
WC Carbonate Alkalinity, LCS/MBLK	
WC Reactivity Qualification	
WC TDS/Conductivity ratio outside of range	
WC TOX Breakthrough- no volume for redo	
WC TOX samples were centrifuged	
Other	

DILUTION CODES	REASON
002	Sample matrix effects
003	Excessive foaming
004	High levels of non-target compounds
008	High concentration of target analytes
009	Sample turbidity
010	Sample color
011	Insufficient volume for lower dilution
012	Sample viscosity
013	other

ICAL Compliant? YES NO NA IF NO, Why? ________________________________
LCS/CCV Compliant? YES NO NA IF NO, Why? ________________________________
CCB Compliant? YES NO NA IF NO, Why? ________________________________
RPD Compliant? YES NO NA IF NO, Why? ________________________________
ERA Compliant? YES NO NA IF NO, Why? ________________________________

NUMBER of REANALYSIS FOR THIS BATCH:________________

Analyst______________________________________ Date_____________

Time Critical Batch Review_______________________ Date_____________

Secondary Review & Closure______________________ Date_____________

WC Summary Rev5 / 05-2008

Title: Ferrous Iron
Method 3500-Fe

Once printed, this is considered an uncontrolled document

Approvals (Signature/Date):

Lisa R Matecki 06/17/2008
Lisa R. Matecki Date
Wet Chemistry Dept. Supervisor

Kenneth E. Kasperek 06/20/2008
Kene Kasperek Date
Health & Safety Manager / Coordinator

Peggy Gray-Erdmann 06/18/2008
Peggy Gray-Erdmann Date
Quality Assurance Manager

06/18/2008
Chris Spencer Date
Laboratory Director

Facility Distribution No. ____________ **Distributed To:** ____________________

1.0 Scope and Application

1.1 Analytes, Matrix(s), and Reporting Limits

This method is for determining total and ferrous iron in environmental surface and saline waters and aqueous domestic and industrial wastes. The quantitation limit is 0.10mg/L

2.0 Summary of Method

The sample is treated with acid and 1,10- phenanthroline, this forms an orange red color in the presence of iron, which obeys Beer's Law.

3.0 Definitions

Standard definitions are found in section 3 of the Laboratory Quality Manual.

4.0 Interferences

4.1 Strong oxidizing agents, cyanide, nitrite, and phosphates (especially polyphosphates), chromium, zinc in concentrations exceeding 10 times that of iron; cobalt, and copper in excess of 5mg/L and nickel in excess of 2mg/L.

4.2 Bismuth, cadmium, mercury, molybdate and silver precipitate phenanthroline.

4.3 If noticeable amounts of color or organic matter are present, the sample can be evaporated, gently ashed, and re-dissolved in acid.

5.0 Safety

Employees must abide by the policies and procedures in the Corporate Environmental Health and Safety Manual (CW-E-M-001), Radiation Safety Manual and this document. This procedure may involve hazardous material, operations and equipment. This SOP does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of the method to follow appropriate safety, waste disposal and health practices under the assumption that all samples and reagents are potentially hazardous. Safety glasses, gloves, lab coats and closed-toe, nonabsorbent shoes are a minimum.

5.1 Specific Safety Concerns or Requirements

Samples that contain high concentrations of carbonates or organic material or samples that are at elevated pH can react violently when acids are added.

5.2 Primary Materials Used

The following is a list of the materials used in this method, which have a serious or significant hazard rating. **Note: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the MSDS for each of the materials listed in the table.** A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the MSDS for each material before using it for the first time or when there are major changes to the MSDS.

Material	Hazards	Exposure Limit (1)	Signs and symptoms of exposure
Nitric Acid	Corrosive Oxidizer Poison	2ppm-TWA 4ppm-STEL	Nitric acid is extremely hazardous; it is corrosive, reactive, an oxidizer, and a poison. Inhalation of vapors can cause breathing difficulties and lead to pneumonia and pulmonary edema, which may be fatal. Other symptoms may include coughing, choking, and irritation of the nose, throat, and respiratory tract. Can cause redness, pain, and severe skin burns. Concentrated solutions cause deep ulcers and stain skin a yellow or yellow-brown color. Vapors are irritating and may cause damage to the eyes. Contact may cause severe burns and permanent eye damage.
1 – Exposure limit refers to the OSHA regulatory exposure limit.			

6.0 Equipment and Supplies

6.1 Instrumentation

- Spectrophotometer

6.2 Supplies

- 25ml sample cells
- Eppendorf Pipettes for measurements of 0.025 to 5ml

7.0 Reagents and Standards

7.1 Ferrous Iron Reagent Powder pillows from HACH.

7.2 Stock iron solution: FAS (Ferrous Ammonium Sulfate) slowly add 20ml conc. H_2SO_4 to 50ml of water and dissolve 1.404 grams of Fe $(NH_4)_2(SO_4)_2$ $6H_2O$. Add 0.1N potassium permanganate ($KMNO_4$) drop wise until a faint pink color persists. Dilute to 1000ml with water. Also available commercially from numerous vendors.

8.0 Sample Collection, Preservation, Shipment and Storage

Sample container, preservation techniques and holding times may vary and are dependent on sample matrix, method of choice, regulatory compliance, and/or specific contract or client requests. Listed below are the holding times and the references that include preservation requirements.

Matrix	Sample Container	Min. Sample Size	Preservation	Holding Time[1]	Reference
Waters	HDPE	50 mLs	Cool 4 ± 2°C	24 Hours	
Soils	Glass	10 grams	Cool 4 ± 2°C	24 Hours	N/A

[1] Inclusive of digestion and analysis.

8.1 The value of the determination depends greatly on the care taken to obtain a representative sample. Iron in well water or tap samples may vary greatly in both concentration and form, depending on the amount of flushing before and during sampling.

8.2 The rapid change in Ferrous Iron to Ferric Iron ratio after sampling requires that analysis be done as soon as possible. Optimally, the Ferrous Iron should be determined at the time of sampling.

9.0 Quality Control

9.1 **Sample QC** - The following quality control samples are prepared with each batch of samples.

Quality Controls	**Frequency**	**Control Limit**
Initial Calibration Verification (ICV)	Once, after calibration	90-110%
Initial Calibration Blank (ICB)	Once, after calibration	< Reporting Limit
Method Blank (MBLK)	1 in 10 or fewer samples	< Reporting Limit
Laboratory Control Sample (LCS)	1 in 10 or fewer samples	90-110%
Matrix Spike (MS)[1]	1 in 20 or fewer samples	Statistical Limits [3]
Matrix Duplicate (MD)[1]	1 in 20 or fewer samples	Statistical Limits [3]

[1] The sample selection for MD/MS are random, unless specifically requested by a client.
[2] Analytical and QC samples (MB, LCS, MD/MS)
[3] Statistical control limits are updated annually and are updated into LIMS.

9.2 Instrument QC

9.2.1 Initial Calibration Verification (ICV) 2.0ppm- Prepare by adding .25ml of the 200ppm stock standard into 25ml of distilled water. An ICV is made from a second source and must be analyzed once after every curve. LCSs must be analyzed after every 10 samples and at the beginning and end of the analytical batch.

9.2.2 Continuing Calibration Verification (LCS) 2.0ppm- An LCS is made from a second source and must be analyzed after every 10 samples and at the beginning and end of the analytical batch.

9.2.3 Calibration Acceptance Summary- A calibration curve must be analyzed every three months at a minimum. Acceptance criteria is a correlation coefficient (R value) of ≥ 0.995.

Step	***Standards***	***Type***	***Control Limit***	***Frequency***
Method #3500-Fe				
Initial Cal	0.0 0.5 1.0 3.0	Linear	$R\geq0.995$	Minimum of every three months.

10.0 Procedure

10.1 Sample Preparation

Matrix	Sample Size
Waters	25 mLs of sample or dilution
Soils/Wastes	10 gram / 100 mLs DI water [1]

10.2 Calibration

Before any instrument is used as a measurement device, the instrument response to known reference materials must be determined. All sample measurements must be made within the calibration range of the instrument. Preparation of all reference materials used for calibration must be documented.

10.2.1 A calibration curve must be analyzed at a minimum of once every three months. Acceptance criteria is a correlation coefficient (R value) of ≥ 0.995. The curve will consist of a total of 5 points: 0, 0.1, 0.5, 1.0, 3.0 ppm.

10.2.2 Prepare calibration by diluting the stock iron solution (FAS) according to the following table.

Fe Standard (diluted to 25mL w/Di H_2O)	Concentration Fe
0.0ppm	0ml
0.0125ml	0.1ppm
0.0625ml	0.5ppm
0.125ml	1.0ppm
0.375ml	3.0ppm

10.3 Sample Analysis

10.3.1 Mix sample thoroughly and fill a sample cell with 25 ml of sample. Place the prepared sample into the cell holder. Measure the color intensity on the spectrophotometer at 510nm. This sample is not spiked nor do you add the Ferrous Iron Reagent pillow packet. This is used for the blank correction. Enter this value under the blank absorbance column on the excel spreadsheet.

10.3.2 Take this sample and add the contents of one Ferrous Iron Reagent Powder Pillow to the sample cell. Swirl to mix. Wait three minutes for the reaction period. Place the prepared sample into the cell holder. Measure the color intensity on spectrophotometer at 510nm. Enter this value under the sample absorbance column on the excel spreadsheet.

11.0 Calculations / Data Reduction

11.1 Calculate sample results from the calibration curve by using the linear regression curve. NOTE: In the case of any dilutions, correct the result by the dilution factor.

Measured Concentration by Linear Regression:

$$x = \frac{a - b}{m}$$

where:

a = area counts for analyte to be measured
m = slope
x = concentration
b = intercept

and

$$m = \frac{\sum x_i a_i}{\sum x_i^2}$$

$$b = Y_{ave} - bx_{ave}$$

11.2 Accuracy

$$\textbf{ICV / CCV, LCS \% Recovery} = \frac{\text{observed concentration}}{\text{known concentration}} \times 100$$

$$\textbf{MS \% Recovery} = \frac{\text{(spiked sample) - (unspiked sample)}}{\text{spiked concentration}} \times 100$$

11.3 Precision (RPD)

$$\textbf{Matrix Duplicate (MD)} = \frac{|\text{orig. sample value - dup. sample value}|}{[(\text{orig. sample value + dup. sample value})/2]} \times 100$$

11.4 **Concentration** = mg/kg or L $= \frac{C \times V \times D}{W}$

Where:
C = sample concentration in extract (ppm)
V = Volume of extract (mL)
D = Dilution Factor

W = Weight/Volume of sample aliquot extracted (grams or mLs)

NOTE: All dry weight corrections are made in LIMS at the time the final report is prepared.

12.0 Method Performance

12.1 Method Detection Limit Study (MDL)

12.1 Method Detection Limit: A valid method detection limit for each analyte of interest must be generated. The MDL must be below the reporting limit for each analyte. Current TestAmerica Buffalo MDLs are maintained the QA department and are easily viewed in the laboratory LIMS system.

12.2 Demonstration of Capabilities

12.2.1 A one–time initial demonstration of performance for each individual method for water must be generated.

12.2.2 This requires quadruplicate analysis of a mid–level check standard containing all of the standard analytes for the method using the same procedures used to analyze samples, including sample preparation.

12.2.3 Compare these results with the acceptance criteria given in the Method or to laboratory historical limits (if available).

12.2.4 Repeat the test for any analyte that does not meet the acceptance criteria. Repeated failure for any analyte indicates the need for the laboratory to evaluate the analytical procedure and take corrective action.

12.3 Training Requirements

12.3.1 The supervisor has the responsibility to ensure that this procedure is performed by an analyst who has been properly trained in its use and has the required experience.

12.3.2 Analyst validation information is maintained for this method in the laboratory QA files.

12.3.3 The analyst must complete the laboratory safety orientation training that includes, but is not limited to, chemicals, PPE requirements, and electrical safety.

12.3.4 The analyst must read and understand this SOP.

12.3.5 The analyst must read and understand the Method used as reference for this SOP.

12.3.6 The analyst must complete a DOC or successfully analyze PT samples annually.

12.3.7 The analyst must complete the TestAmerica Quality Assurance Training.

13.0 Pollution Control

It is TestAmerica's policy to evaluate each method and look for opportunities to minimize waste generated (i.e., examine recycling options, ordering chemicals based on quantity needed, preparation of reagents based on anticipated usage and reagent stability). Employees must abide by the policies in Section 13 of the Corporate Environmental Health and Safety Manual (CW-E-M-001) for "Waste Management and Pollution Prevention."

14.0 Waste Management

Waste management practices are conducted consistent with all applicable rules and regulations. Excess reagents, samples and method process wastes are disposed of in an accepted manner. Waste description rules and land disposal restrictions are followed. Waste disposal procedures are incorporated by reference to the Corporate Safety Manual. The following waste streams are produced when this method is carried out. The following waste streams are produced when this method is carried out. Acidic waste generated by the analysis. All samples and expired reagents are to be disposed of as "A" waste.

15.0 References / Cross-References

15.1 Standard Methods 20th Edition, method 3500-Fe.

15.2 HACH Method 8146; Ferrous Iron.

16.0 Method Modifications:

N/A

17.0 Attachments

17.1 Analytical Sequence
17.2 Analytical Batch
17.3 Wet Chemistry Batch Summary

18.0 Revision History

- Revision 0, dated 20 June 2008
  - Integration for TestAmerica operations
  - Updated Attachments
  - Quality Manager change, signature updated
  - Department Manager change, signature updated

Attachment 17.1
Analytical Sequence

LCS
MBLK
Sample
Sample
Sample
Sample
Sample
Sample
Sample
Sample
Sample
Sample
LCS
MBLK
Sample
Sample
Sample
Sample
Sample
Sample
Sample
Sample
Sample Duplicate
Sample Spike
LCS
MBLK

Attachment 17.2
Analytical Batch

Laboratory Bench Sheet
FERROUS IRON
Revision 1 November - 2007

TestAmerica - Buffalo

Analyst: ERK
Start Date: 6/3/2008
Start Time: 10:15
End Time: 10:32
DATE OF CURVE= 2/5/2008

SOP Information
Number: AWC-IRON-56

EQL: 0.10 mg/L

ICV INFORMATION
Solution #
Concentration (mg/L) 2.00
ICV True value: 2.00

Calibration Curve Information

	Conc.(mg/L)	ABS.
STD1	0.000	0.000
Std. 2	0.100	0.029
Std. 3	0.500	0.134
Std. 4	1.000	0.265
Std. 5	3.000	0.721

BATCH #	A8B16365
Instrument Information	
Instrument:	Odyssey
Wavelength:	510
Parameter:	Ferrous Iron
Corr. Coef:	0.9994
Slope:	0.23902
Intercept:	0.00990

Reagents Used	Solution ID#
Ferrous Iron Reagent Powder Pillow	CHB-10-H

LCS Information:
Solution # CHC-9-A
Concentration (mg/L): 2
LCS True value: 2.00

Matrix Spike Information:
Solution # CHB-34-B
Concentration (mg/L): 1
MS True Value 1.00

Job #	Sample ID	Sample Volume (mL)	Sample ABS.	Blank ABS.	Corrected ABS.	D.F.	Curve Conc. (mg/L)	Final Conc. (mg/L)	% Rec.	Comments
LCS	LCS	25	0.495		0.495	1	2.0295	2.030	101%	
MBLK	BLANK	25	0.000		0.000	1	ND	ND		
6191	1	25	0.447	0.454	-0.007	2	ND	ND		
	2	25	0.067	0.028	0.039	1	0.1217	0.122		
	3	25	0.090	0.066	0.024	1	0.0590	ND		
	4	25	0.286	0.145	0.141	1	0.5485	0.548		
	5	25	0.034	0.035	-0.001	1	ND	ND		
	6	25	0.282	0.240	0.042	1	0.1343	0.134		
	6md	25	0.281	0.240	0.041	1	0.1301	0.130	RPD=3%	
	7	25	0.406	0.401	0.005	1	-0.0205	ND		
	7ms	25	0.684	0.401	0.283	1	1.1428	1.143	114%@1.0ppm	
		25			0.000	1	ND	ND		
LCS	LCS	25	0.506		0.506	1	2.0755	2.076	104%	
MBLK	BLANK	25	0.000		0.000	1	ND	ND		
		25			0.000	1	ND	ND		
		25			0.000	1	ND	ND		
		25			0.000	1	ND	ND		
		25			0.000	1	ND	ND		
		25			0.000	1	ND	ND		
		25			0.000	1	ND	ND		
		25			0.000	1	ND	ND		
		25			0.000	1	ND	ND		
		25			0.000	1	ND	ND		
		25			0.000	1	ND	ND		
LCS	lcs	25			0.000	1	ND	ND	#VALUE!	
mblk	blank	25			0.000	1	ND	ND		

Page 1 of 2

Attachment 17.3
Wet Chemistry Batch Summary

WET CHEMISTRY BATCH SUMMARY

PARAMETER________________________ METHOD____________ BATCH ________________________

COMMENTS	JOB NUMBER
WC Historical confirms within Hold Time	
WC Historical NO confirm & RE outside of HT	
WC Hold Time Exceedance-Dilution required	
WC Hold Time Exceedance-Instrument Failure	
WC Holding Time Exceedance by Date	
WC Holding Time Exceedance by Hours	
WC LCS within ERA limits outside internal	
WC LCS high recovery, sample ND	
WC MBLK hit but samples > 10X blank value	
WC RPD Exceedance for MS / SD	
WC Spike Failure HIGH MS only	
WC Spike Failure LOW MS only	
WC Spike Failure MS and SD	
WC BOD HT met- Oxygen depleted-RE out HT	
WC Carbonate Alkalinity, LCS/MBLK	
WC Reactivity Qualification	
WC TDS/Conductivity ratio outside of range	
WC TOX Breakthrough- no volume for redo	
WC TOX samples were centrifuged	
Other	

DILUTION CODES	REASON
002	Sample matrix effects
003	Excessive foaming
004	High levels of non-target compounds
008	High concentration of target analytes
009	Sample turbidity
010	Sample color
011	Insufficient volume for lower dilution
012	Sample viscosity
013	other

ICAL Compliant?	YES	NO	NA	IF NO, Why? __________________
LCS/CCV Compliant?	YES	NO	NA	IF NO, Why? __________________
CCB Compliant?	YES	NO	NA	IF NO, Why? __________________
RPD Compliant?	YES	NO	NA	IF NO, Why? __________________
ERA Compliant?	YES	NO	NA	IF NO, Why? __________________

NUMBER of REANALYSIS FOR THIS BATCH:____________

Analyst____________________________ Date___________

Time Critical Batch Review_________________ Date___________

Secondary Review & Closure_________________ Date___________

WC Summary Rev5 / 05-2008

Title: Alkalinity
Method No. 310.2

Once printed, this is considered an uncontrolled document

Approvals (Signature/Date):

Lisa R Matecki 04/20/09
Lisa R. Matecki Date
Technical Manager

Kenneth E. Kasperek 04/20/09
Kenneth E Kasperek Date
Health & Safety Manager / Coordinator

Peggy Gray-Erdmann 04/20/09
Peggy Gray-Erdmann Date
Quality Assurance Manager

04/20/09
Chris Spencer Date
Laboratory Director

Facility Distribution No. ____________ **Distributed To:**__________________________

1.0 Scope and Application

1.1 Analytes, Matrix(s), and Reporting Limits

This method is taken from EPA Method 310.2. It is applicable to drinking, surface, and saline waters, domestic and industrial wastes. Soil samples may also be analyzed using this method after the ASTM Leaching procedure has been completed. The reporting limit has been determined to be 10.0 mg/L.

2.0 Summary of Method

Methyl orange is used as a color reagent for this method because its pH range is the same as the pH of the equivalence point for the total alkalinity titration. The methyl orange indicator is in a dilute pH 3.1 buffer which is just below its color change pH. When an alkaline sample is injected, the poorly buffered methyl orange changes color in proportion to the alkalinity of the sample.

3.0 Definitions

Standard definitions are used in this document as defined by the TestAmerica Corporate Quality Assurance Plan.

4.0 Interferences

4.1 Turbidity and color will interfere. Turbidity can be removed by filtration.

4.2 The pH of samples is tested prior to analysis. Samples over a pH of 8.3 are run titrametrically (method SM2320B).

5.0 Safety

Employees must abide by the policies and procedures in the Corporate Environmental Health and Safety Manual (CW-E-M-001), Radiation Safety Manual and this document. This procedure may involve hazardous material, operations and equipment. This SOP does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of the method to follow appropriate safety, waste disposal and health practices under the assumption that all samples and reagents are potentially hazardous. Safety glasses, gloves, lab coats and closed-toe, nonabsorbent shoes are a minimum.

5.1 Specific Safety Concerns or Requirements

The following chemicals have the potential to be highly toxic or hazardous; for detailed explanations consult the MSDS.

5.1.1 Hydrochloric Acid
5.1.2 Methyl Orange

5.2 Primary Materials Used

There are no specialized safety concerns associated with this method.

6.0 Equipment and Supplies

6.1 Instrumentation

- Konelab

6.2 Supplies

- Analytical Balance capable of accurately weighing to the nearest 0.0001g.
- Glassware- Class A volumetric flasks and pipettes or plastic containers as required.
- pH strips measuring from 6.5 to 9.0.

7.0 Reagents and Standards

- **Methyl Orange Solution**: Dissolve 0.0125-g Methyl Orange in 100-mL of di water
- **PH 3.1 Buffer solution:** Dissolve 0.51047 of Potassium acid phthalate in 50-mL di water. Add ~8.76mL of 0.1N HCl and dilute to 100mL with carbon dioxide free di water. The pH of the solution should be 3.1. Adjust accordingly with more HCl or di water. Pour into a glass storage bottle and prepare fresh weekly.
- **Alk Mix:** Add 25-mL of the pH 3.1 Buffer to 5-mL of the Methyl Orange Solution. Mix well. Pour into a glass storage bottle and prepare fresh daily.
- **Sodium Carbonate Primary STD, 1000 ppm :** Dissolve 0.1060 g of anhydrous Sodium carbonate (oven dried at 250°C for 4 hours) in di water. Dilute to 100ml. 1.0ml=1.0 mg NaC03. A pre made Sodium Carbonate Primary STD, 1000 ppm can also be used.
- **Sodium Carbonate 100ppm Std:** Add 5.0-mL of 1000ppm sodium Carbonate Std and dilute to 50-mL using di water.

8.0 Sample Collection, Preservation, Shipment and Storage

Sample container, preservation techniques and holding times may vary and are dependent on sample matrix, method of choice, regulatory compliance, and/or specific contract or client requests. Listed below are the holding times and the references that include preservation requirements.

Matrix	Sample Container	Min. Sample Size	Preservation	Holding Time [1]	Reference
Waters	HDPE	50-mLs	Cool 4 ± 2°C	14-Days	40 CFR Part 136.3
Soils	Glass	10 grams	Cool 4 ± 2°C	14-Days	N/A

[1] Inclusive of digestion and analysis.

9.0 Quality Control

9.1 **Sample QC** - The following quality control samples are prepared with each batch of samples.

Quality Controls[2]	Frequency	Control Limit
Method Blank (MBLK)	1 in 10 or fewer samples	< Rpt. Limit
Laboratory Control Sample (LCS)	1 in 10 or fewer samples	Statistical Limits [3]
Matrix Duplicate (MD)[1]	1 in 20 or fewer samples	Statistical Limits [3]
Matrix Spike (MS)[1]	1 in 20 or fewer samples	Statistical Limits [3]

[1] The sample selection for MD/MS is random, unless specifically requested by a client.
[2] Analytical and QC samples (MB, LCS, MS/MSD)
[3] Statistical control limits are updated annually and are updated into LIMS.

9.2 Instrument QC

9.2.1 Initial Calibration Verification (ICV): The ICV must be prepared from a separate source from the calibration curve and must be analyzed immediately after the curve. Obtained values must be ± 10% of the true value.

9.2.2 Initial Calibration Blank (ICB): The ICB must be analyzed immediately after the curve and exhibit values less than the TestAmerica Buffalo Quantitation limit.

9.2.3 Laboratory Control Standard (LCS): The LCS must be analyzed at the beginning and end of the analytical procedure and after every ten samples. Obtained values of the LCS must be ± 10% of the true value.

9.2.4 Method Blank (MBLK): To determine freedom from contamination, Method Blanks are prepared at the beginning of the analytical procedure as well as after every ten samples and at the end of the analytical procedure. The Method Blank goes through the same treatment as the samples and standards. The Method Blank must exhibit values less than the TestAmerica Buffalo Quantitation limit.

9.2.5 Matrix Duplicate (MD): Sample duplicates should be analyzed at least once for every group of twenty or fewer samples. Samples should agree within 20% RPD. Samples that fail to meet these criteria should be reanalyzed.

9.2.6 Matrix Spike (MS): Sample spikes should be analyzed at least once for every group of twenty or fewer samples.

9.2.7 Calibration Acceptance Summary

Step	*Standards*	*Type*	*Control Limit*	*Frequency*
Method #310.2				
Initial Cal	5.0, 10.0, 25.0, 50.0, 75.0, 100	Linear		Once monthly, unless necessary sooner
ICV	50.0		90-110%	Every 10 samples
ICB	0.0		<10.0	Every 10 samples
LCS	50.0		90-110%	Every 10 samples

MBLK	0.0		<10.0	Every 10 samples

10.0 Procedure

10.1 Sample Preparation

Matrix	Sample Size
Waters	~2.0 mLs, of sample or dilution
Soils/Wastes	10 gram / 100 mLs DI water [1]

10.2 Calibration

10.2.1 Prepare a series of standards, covering the desired range of the calibration curve by diluting suitable volumes of standard solutions.

10.2.2 Prepare the standard calibration curve by plotting instrument response against concentration values. The curve for the Konelab is linear (1st Order). The calibration curve may be fitted to the calibration solution concentration/response data using the computer. Acceptance or control limits should be established using the difference between the measured values of the calibration solution and the “true value” concentration. Acceptance criteria for the calibration curve is a correlation coefficient (R value) $\geq$0.990.

10.2.3 After the calibration has been established, it must be verified by the analysis of a second source standard (ICV). If measurements exceed +/- 10% of the established ICV value, the analysis should be terminated and the instrument recalibrated. The new calibration must be verified before continuing analysis.

10.3 Sample Analysis

10.3.1 The pH of samples requiring Bicarbonate, Carbonate or Hydroxyl Alkalinity must be 8.3 or less. Samples which have a pH above 8.3 must be analyzed titrametrically (method SM2320B).

10.3.2 Each day it is necessary to check the quality of the water blank. This is done after the start up procedure. All results must be within +/-2mA (milliabsorbance). If not rerun the procedure.

10.3.3 Prepare reagents and check standards as described in Sect 7.0 and load into instrument.

10.3.4 Input the sample identification required by the data system and load samples in the appropriate instrument segments.

10.3.5 Analyze samples

10.3.6 Once a run is complete, you can then review it for compliancy. Check that all LCSs are 90-110% and MBLKs are less than the detection limit. Check to see that all results, and ones with their corresponding auto-dilutions fall within the curve range.

11.0 Calculations / Data Reduction

11.1 Accuracy

ICV, LCS % Recovery $= \dfrac{\text{observed concentration}}{\text{known concentration}} \times 100$

MS % Recovery $= \dfrac{\text{(spiked sample) - (unspiked sample)}}{\text{spiked concentration}} \times 100$

11.2 Precision (RPD)

Matrix Duplicate (MD) $= \dfrac{|\text{orig. sample value - dup. sample value}|}{[(\text{orig. sample value + dup. sample value})/2]} \times 100$

12.0 Method Performance

12.1 Method Detection Limit Study (MDL)

The method detection limit (MDL) is the lowest concentration that can be detected for a given analytical method and sample matrix with 99% confidence that the analyte is present. The MDL is determined according to the laboratory's MDL procedure in the QA Manual. MDLs reflect a calculated (statistical) value determined under ideal laboratory conditions in a clean matrix, and may not be achievable in all environmental matrices. The laboratory maintains MDL studies for analyses performed; these are verified at least annually unless method requirements require a greater frequency.

12.2 Demonstration of Capabilities

12.2.1 A one–time initial demonstration of performance for each individual method must be generated.

12.2.2 This requires quadruplicate analysis of a mid–level check standard containing all of the standard analytes for the method using the same procedures used to analyze samples, including sample preparation.

12.2.3 Calculate the recovery for each analyte of interest.

12.2.4 Compare these results with the acceptance criteria given in the Method or to laboratory historical limits (if available).

12.2.5 Repeat the test for any analyte that does not meet the acceptance criteria. Repeated failure for any analyte indicates the need for the laboratory to evaluate the analytical procedure and take corrective action.

12.3 Training Requirements

12.3.1 The supervisor has the responsibility to ensure that this procedure is performed by an analyst who has been properly trained in its use and has the required experience.

12.3.2 The following analyst validation information is maintained for this method in the laboratory QA files.

The analyst must complete the laboratory safety orientation training that includes, but is not limited to, chemicals, PPE requirements, and electrical safety.

The analyst must read and understand this SOP.

The analyst must read and understand the Method used as reference for this SOP.

The analyst must complete a DOC or successfully analyze PT samples annually.

The analyst must complete the TestAmerica Quality Assurance Training.

13.0 Pollution Control

It is TestAmerica's policy to evaluate each method and look for opportunities to minimize waste generated (i.e., examine recycling options, ordering chemicals based on quantity needed, preparation of reagents based on anticipated usage and reagent stability). Employees must abide by the policies in Section 13 of the Corporate Environmental Health and Safety Manual (CW-E-M-001) for "Waste Management and Pollution Prevention."

14.0 Waste Management

Waste management practices are conducted consistent with all applicable rules and regulations. Excess reagents, samples and method process wastes are disposed of in an accepted manner. Waste description rules and land disposal restrictions are followed. There are no special waste streams associated with this method.

15.0 References / Cross-References

15.1 Alkalinity Method 310.1, Methods for Chemical Analysis of Water and Wastes, EPA-600/4-712-020, March 112123.

15.2 Alkalinity Method 310.2, Methods for Chemical Analysis of Water and Wastes, EPA-600/4-712-020, March 11283.

15.3 EST Analytical Methodologies for Konelab instrumentation.

16.0 Method Modifications:

N/A

17.0 Attachments

17.1 Analytical Sequence
17.2 Analytical Batch
17.3 Wet Chemistry Batch Summary Sheet

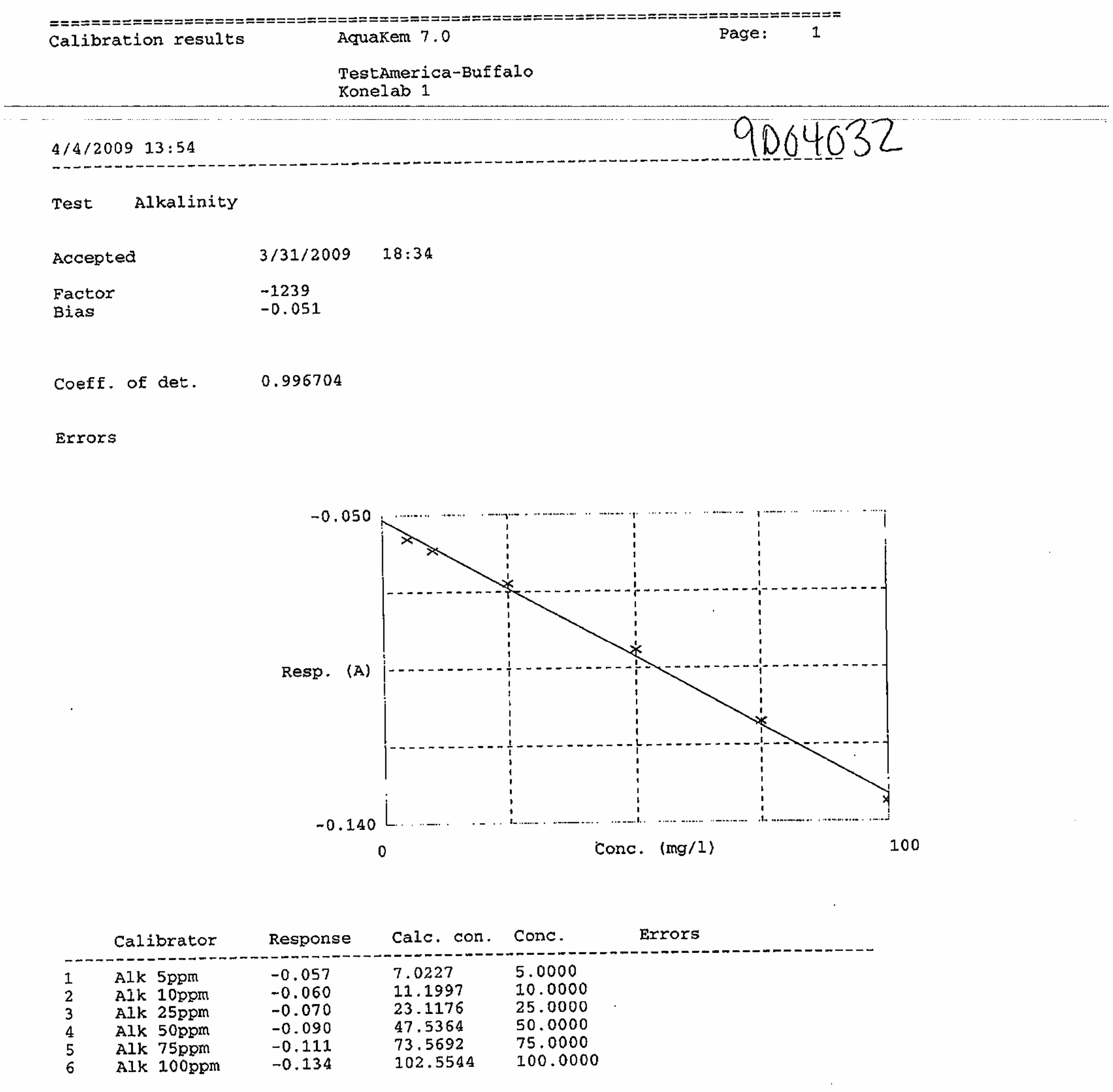
18.0 Revision History

- Revision 0, dated 14 April 2009
  - Integration for TestAmerica operations
  - Update Department and QA manager name and signature
  - Update Attachments 17.2, 17.3

Attachment 17.1
Analytical Sequence

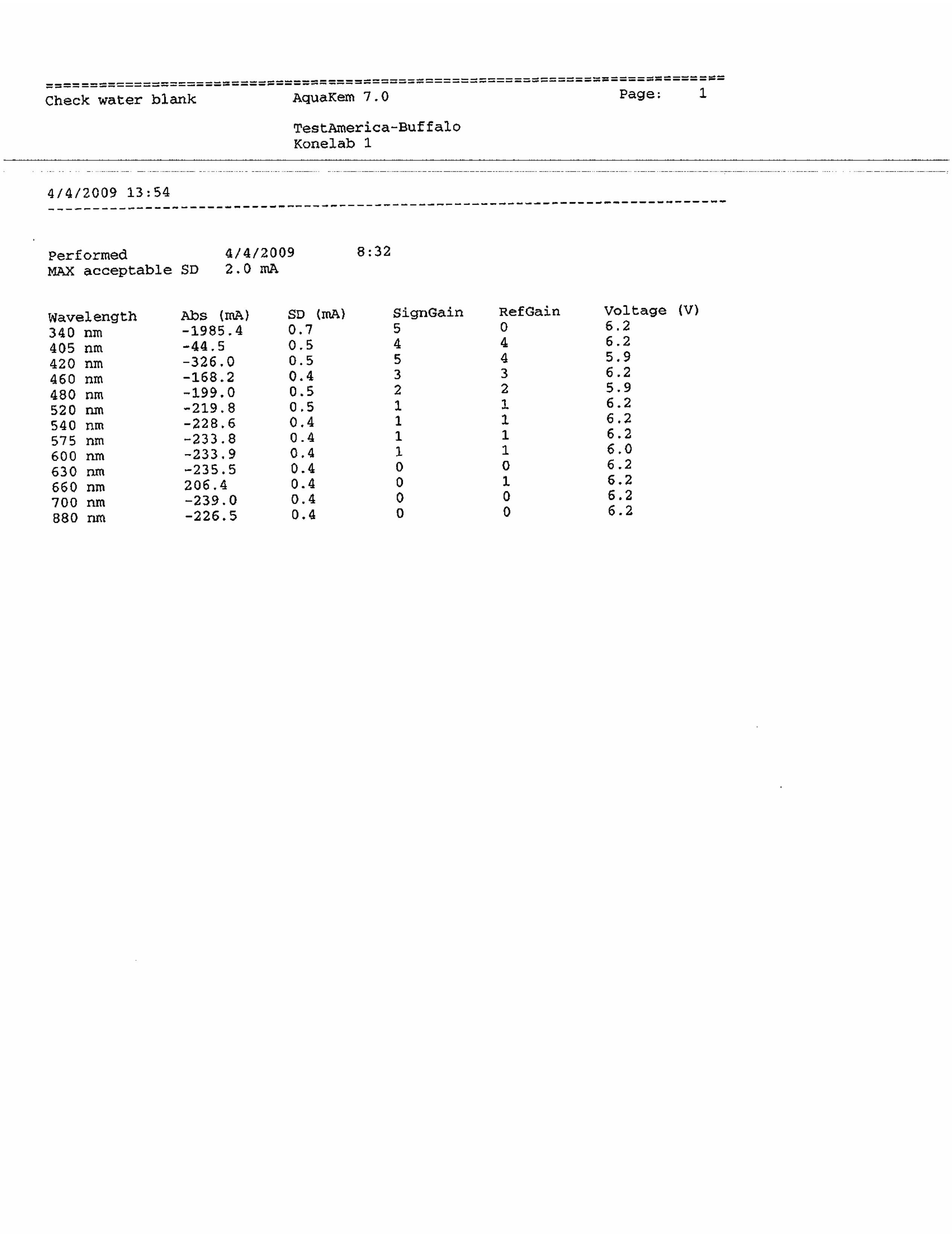
LCS
MBLK
SAMPLE
SAMPLE
SAMPLE
SAMPLE
SAMPLE
SAMPLE
SAMPLE
SAMPLE
SAMPLE
SAMPLE
LCS
MBLK
SAMPLE
SAMPLE
SAMPLE
SAMPLE
SAMPLE
SAMPLE
SAMPLE
SAMPLE
SAMPLE DUP
SAMPLE SPIKE
LCS
MBLK

Attachment 17.2
Analytical Batch

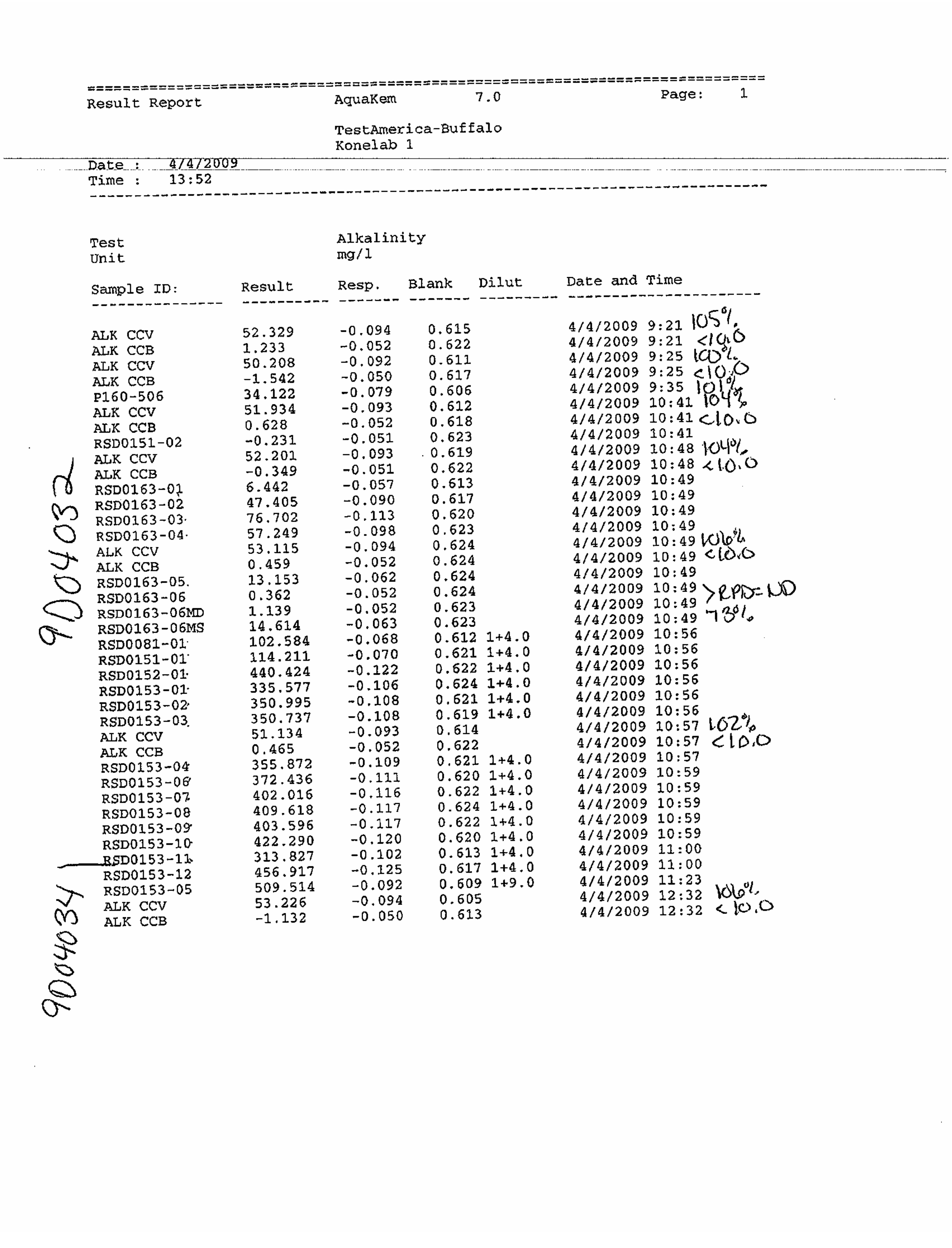


```
===============================================================================
Calibration results          AquaKem 7.0                       Page:     1

                             TestAmerica-Buffalo
                             Konelab 1
-------------------------------------------------------------------------------
                                                                     9D04032
4/4/2009 13:54
-------------------------------------------------------------------------------

Test     Alkalinity


Accepted                3/31/2009    18:34

Factor                  -1239
Bias                    -0.051


Coeff. of det.          0.996704


Errors
```

```
     Calibrator        Response      Calc. con.    Conc.        Errors
-----------------------------------------------------------------------------
1    Alk 5ppm          -0.057        7.0227        5.0000
2    Alk 10ppm         -0.060        11.1997       10.0000
3    Alk 25ppm         -0.070        23.1176       25.0000
4    Alk 50ppm         -0.090        47.5364       50.0000
5    Alk 75ppm         -0.111        73.5692       75.0000
6    Alk 100ppm        -0.134        102.5544      100.0000
```



```
==============================================================================
Check water blank            AquaKem 7.0                          Page:     1

                             TestAmerica-Buffalo
                             Konelab 1

4/4/2009 13:54
------------------------------------------------------------------------------

Performed              4/4/2009        8:32
MAX acceptable SD      2.0 mA

Wavelength        Abs (mA)      SD (mA)      SignGain      RefGain      Voltage (V)
340 nm            -1985.4       0.7          5             0            6.2
405 nm            -44.5         0.5          4             4            6.2
420 nm            -326.0        0.5          5             4            5.9
460 nm            -168.2        0.4          3             3            6.2
480 nm            -199.0        0.5          2             2            5.9
520 nm            -219.8        0.5          1             1            6.2
540 nm            -228.6        0.4          1             1            6.2
575 nm            -233.8        0.4          1             1            6.2
600 nm            -233.9        0.4          1             1            6.0
630 nm            -235.5        0.4          0             0            6.2
660 nm            206.4         0.4          0             1            6.2
700 nm            -239.0        0.4          0             0            6.2
880 nm            -226.5        0.4          0             0            6.2
```

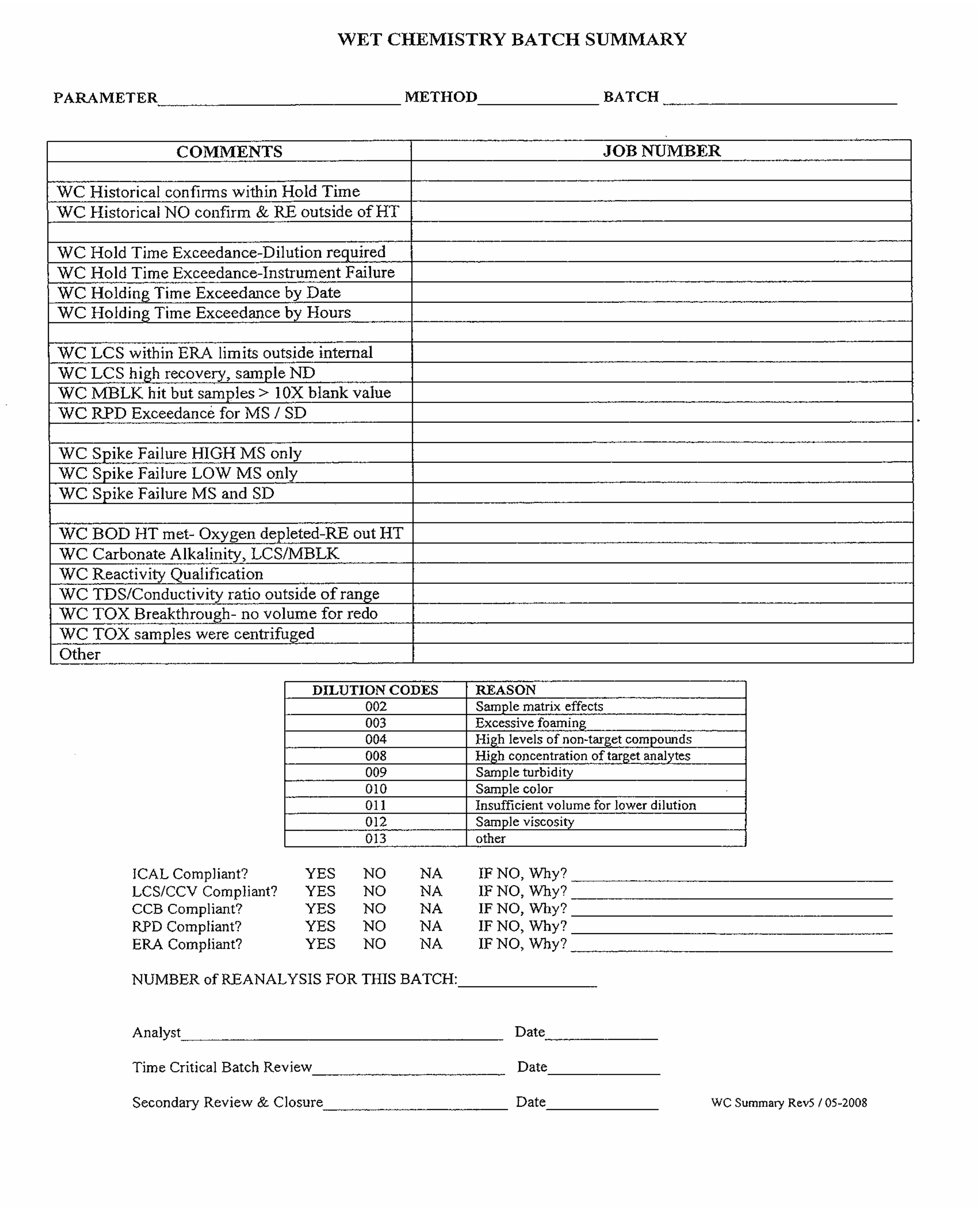


```
================================================================================
Result Report                  AquaKem          7.0                  Page:     1

                               TestAmerica-Buffalo
                               Konelab 1
--------------------------------------------------------------------------------
Date  :    4/4/2009
Time  :    13:52
--------------------------------------------------------------------------------


Test                           Alkalinity
Unit                           mg/l

Sample ID:         Result      Resp.    Blank    Dilut     Date and Time
----------------   ----------  -------  -------  --------- ----------------------

ALK CCV            52.329      -0.094    0.615             4/4/2009 9:21  105%
ALK CCB            1.233       -0.052    0.622             4/4/2009 9:21  <10.0
ALK CCV            50.208      -0.092    0.611             4/4/2009 9:25  100%
ALK CCB            -1.542      -0.050    0.617             4/4/2009 9:25  <10.0
P160-506           34.122      -0.079    0.606             4/4/2009 9:35  101%
ALK CCV            51.934      -0.093    0.612             4/4/2009 10:41 104%
ALK CCB            0.628       -0.052    0.618             4/4/2009 10:41 <10.0
RSD0151-02         -0.231      -0.051    0.623             4/4/2009 10:41
ALK CCV            52.201      -0.093    0.619             4/4/2009 10:48 104%
ALK CCB            -0.349      -0.051    0.622             4/4/2009 10:48 <10.0
RSD0163-01         6.442       -0.057    0.613             4/4/2009 10:49
RSD0163-02         47.405      -0.090    0.617             4/4/2009 10:49
RSD0163-03         76.702      -0.113    0.620             4/4/2009 10:49
RSD0163-04         57.249      -0.098    0.623             4/4/2009 10:49
ALK CCV            53.115      -0.094    0.624             4/4/2009 10:49 106%
ALK CCB            0.459       -0.052    0.624             4/4/2009 10:49 <10.0
RSD0163-05         13.153      -0.062    0.624             4/4/2009 10:49
RSD0163-06         0.362       -0.052    0.624             4/4/2009 10:49 >RPD=ND
RSD0163-06MD       1.139       -0.052    0.623             4/4/2009 10:49
RSD0163-06MS       14.614      -0.063    0.623             4/4/2009 10:49 73%
RSD0081-01         102.584     -0.068    0.612 1+4.0       4/4/2009 10:56
RSD0151-01         114.211     -0.070    0.621 1+4.0       4/4/2009 10:56
RSD0152-01         440.424     -0.122    0.622 1+4.0       4/4/2009 10:56
RSD0153-01         335.577     -0.106    0.624 1+4.0       4/4/2009 10:56
RSD0153-02         350.995     -0.108    0.621 1+4.0       4/4/2009 10:56
RSD0153-03         350.737     -0.108    0.619 1+4.0       4/4/2009 10:56
ALK CCV            51.134      -0.093    0.614             4/4/2009 10:57 102%
ALK CCB            0.465       -0.052    0.622             4/4/2009 10:57 <10.0
RSD0153-04         355.872     -0.109    0.621 1+4.0       4/4/2009 10:57
RSD0153-06         372.436     -0.111    0.620 1+4.0       4/4/2009 10:59
RSD0153-07         402.016     -0.116    0.622 1+4.0       4/4/2009 10:59
RSD0153-08         409.618     -0.117    0.624 1+4.0       4/4/2009 10:59
RSD0153-09         403.596     -0.117    0.622 1+4.0       4/4/2009 10:59
RSD0153-10         422.290     -0.120    0.620 1+4.0       4/4/2009 10:59
RSD0153-11         313.827     -0.102    0.613 1+4.0       4/4/2009 11:00
RSD0153-12         456.917     -0.125    0.617 1+4.0       4/4/2009 11:00
RSD0153-05         509.514     -0.092    0.609 1+9.0       4/4/2009 11:23
ALK CCV            53.226      -0.094    0.605             4/4/2009 12:32 106%
ALK CCB            -1.132      -0.050    0.613             4/4/2009 12:32 <10.0
```

9004032

9004034

Attachment 17.3
Wet Chemistry Batch Cover Sheet



WET CHEMISTRY BATCH SUMMARY

PARAMETER____________________________ METHOD______________ BATCH ___________________________

COMMENTS	JOB NUMBER
WC Historical confirms within Hold Time	
WC Historical NO confirm & RE outside of HT	
WC Hold Time Exceedance-Dilution required	
WC Hold Time Exceedance-Instrument Failure	
WC Holding Time Exceedance by Date	
WC Holding Time Exceedance by Hours	
WC LCS within ERA limits outside internal	
WC LCS high recovery, sample ND	
WC MBLK hit but samples > 10X blank value	
WC RPD Exceedance for MS / SD	
WC Spike Failure HIGH MS only	
WC Spike Failure LOW MS only	
WC Spike Failure MS and SD	
WC BOD HT met- Oxygen depleted-RE out HT	
WC Carbonate Alkalinity, LCS/MBLK	
WC Reactivity Qualification	
WC TDS/Conductivity ratio outside of range	
WC TOX Breakthrough- no volume for redo	
WC TOX samples were centrifuged	
Other	

DILUTION CODES	REASON
002	Sample matrix effects
003	Excessive foaming
004	High levels of non-target compounds
008	High concentration of target analytes
009	Sample turbidity
010	Sample color
011	Insufficient volume for lower dilution
012	Sample viscosity
013	other

ICAL Compliant?	YES	NO	NA	IF NO, Why? ______________________
LCS/CCV Compliant?	YES	NO	NA	IF NO, Why? ______________________
CCB Compliant?	YES	NO	NA	IF NO, Why? ______________________
RPD Compliant?	YES	NO	NA	IF NO, Why? ______________________
ERA Compliant?	YES	NO	NA	IF NO, Why? ______________________

NUMBER of REANALYSIS FOR THIS BATCH:_________________

Analyst____ Date______________

Time Critical Batch Review________________________ Date______________

Secondary Review & Closure______________________ Date______________

WC Summary Rev5 / 05-2008

Title: Mercury Preparation and Analysis [Methods 245.1, 7470A, 7471A]

Once printed, this is considered an uncontrolled document

Approvals (Signature/Date):

E. Scott Wagner 02/08/10
E. Scott Wagner Date
Department Manager

Jennifer Pierce 02/08/10
Jennifer Pierce Date
Operations Manager

Peggy Gray-Erdmann 02/08/10
Peggy Gray-Erdmann Date
Quality Assurance Manager

02/08/10
Christopher Spencer Date
Laboratory Director

Facility Distribution No. ___________ **Distributed To:**________________________

1.0 Scope and Application

1.1 This method is used for the determination of Mercury in aqueous and solid environmental samples. This procedure is used to analyze organic and inorganic mercury in drinking water, surface water, waste, and saline waters, both domestic and industrial wastes.

1.2 This method is based upon SW-846, 3rd edition method 7470A/7471A and also conforms to the EPA Environmental Methods Management Council's "Guidelines and Format for Methods to Be Proposed at 40 CFR, part 136" (Method 245.1).

1.3 This method is for the determination of Hg by cold-vapor atomic absorption (CVAA) in the range of 0.2 μg/L to 10.0 μg/L. The range may be extended to higher levels by selection of a smaller sample size or by dilution of existing samples.

1.4 This method is used only by analysts experienced in the use of the chemical principles outlined in this SOP and who are trained thoroughly in the sample handling and instrumental techniques described in this method.

1.5 This method is "performance based." The laboratory is permitted to modify the mehod to overcome interferences or lower the cost of measurements provided all performance criteria are met.

1.6 On occasion, clients may request modifications to this SOP. These modifications are addressed following the procedures outlined in the lab Quality Assurance Manual (QAM).

1.7 Analytes, Matrix(s), and Reporting Limits

1.7.1 Total, Total Recoverable, and Dissolved (Soluble) Mercury.

1.7.2 This SOP is used for the preparation and analysis of groundwater, surface water, drinking water, TCLP's, leachates, filtered collection wastes, sand, rock, concrete, soil, sediment, and sludge samples.

1.7.3 Reporting Limits are defined as the lowest concentration of an analyte determined by a given method in a given matrix that the laboratory feels can be reported with acceptable quantitative error, client requirements, values specified by the EPA methods or other project and client requirements. Wherever possible, reporting is limited to values approximately 3–5 times the respective MDL to ensure confidence in the value reported. Client specific requests for reporting to the IDL or MDL are special circumstances not to be confused with the previous statement. The reporting limit for mercury analysis in an aqueous matrix is 0.2 μg/L while the reporting limit for solid samples is typically 0.025 μg/g.

2.0 Summary of Method

2.1 Samples are digested first by oxidation of Hg in the samples to the Hg^{2+} oxidation state under strongly acidic and oxidizing conditions and near boiling temperatures. Potassium permanganate ($KMnO_4$) and potassium persulfate ($K_2S_2O_8$) (aqueous samples only) are added to aid in the oxidation of organic mercury compounds and to eliminate possible interference from sulfides and organic materials. Potassium permanganate is later reduced with hydroxylamine hydrochloride ($NH_2OH \bullet HCl$) prior to the digestate being analyzed.

2.2 Analysis by cold-vapor atomic absorption is based on the absorption of radiation at the 253.7-nm wavelength by Hg vapor. The Hg^{2+} in a digested sample is reduced to the elemental state and aerated from solution in-line. The Hg vapor passes through an optical cell positioned in the light path of an atomic absorption spectrometer. Hg concentration is determined as a function of the measured absorption.

2.3 Quality is assured through the analysis of preparation blanks, blank spikes, matrix spikes, duplicates, and reference standards (solids).

3.0 Definitions

3.1 **Total Mercury**: All oxidizable mercury forms and species found in an unfiltered aqueous or solid sample matrix. This includes, but is not limited to, Hg (0), Hg (I), Hg (II), strongly organo-complexed Hg (II) compounds, adsorbed particulate Hg, and several tested covalently bound organo-mercury compounds.

3.2 **Dissolved (Soluble) Mercury**: All oxidizable mercury forms and species found in the filtrate of an aqueous solution that has been filtered through a 0.45 micron filter and then acidified to a pH < 2.

3.3 Any other definitions contained within this document are standard definitions as defined by the TestAmerica Buffalo Laboratory Quality Manual.

4.0 Interferences

4.1 Contamination

4.1.1 Contamination Control: Any object or substance that contacts the sample should be mercury free and free from any material that may interfere with the analysis of mercury. Although contamination control is essential, personal health and safety remain the highest priority. Section 5 of this SOP gives suggestions and requirements for personal safety.

4.1.2 Avoiding Contamination**:** The best way to control contamination is to completely avoid exposure of the sample to contamination in the first place. Avoiding exposure means performing operations in an area known to be free of any traces of mercury. Two of the most important factors in avoiding and/or reducing sample contamination are (1) an awareness of potential sources of contamination and (2) strict attention to the work being done

4.1.3 Minimize Exposure: The apparatus and/or glassware that will come into contact with the samples, blanks, or standard solutions are to be opened or exposed only in a clean area of the lab. When any relevant materials, glassware or instruments are not being used, cover with a plastic liner or remove from the area of analysis to avoid accidental exposure.

4.1.4 Clean Work Surfaces: Before a given batch of samples is processed, the analyst makes certain that all work surfaces in the hood, the bench and other areas are clean, thereby minimizing potential for contamination from previous batches.

4.1.5 Wear Gloves: Sampling personnel wear clean, non-talc gloves during all operations involving handling of any instrument, glassware, samples or blanks. Only clean gloves may touch the instruments. If another object or substance is touched, the gloves must be changed before resuming work on the instrument. If it is suspected that gloves have become contaminated, work must be halted, the contaminated gloves removed, and a new pair put on. It is a good practice to change gloves between working on different sample matrices.

4.1.6 Use Mercury-Free Materials: All materials used for the preparation and analysis of mercury at ambient water quality criteria levels must be non-metallic, free of material that may contain metals, or both. Mercury thermometers are not to be used within the mercury preparation or analysis areas.

4.1.7 Containers: Each new container type is tested before use, because Mercury vapors can diffuse in or out of certain types of materials, resulting in results that are biased high or low.

4.1.8 Contamination from Reagents: Contamination can be introduced into samples from the method reagents used during processing and analysis. Reagents are monitored using method blanks included in each batch. When a reagent is suspected to be impure, it will be analyzed. If the blank is lower than MDL, that reagent can be used.

4.1.9 Contamination from Carryover: Contamination may occur when a sample containing a low concentration of mercury is analyzed immediately after a sample containing a high concentration of mercury. When an unusually concentrated sample (approximately 100+ ppb) is encountered, the cleaning (rinse) time is extended before proceeding with the next sample. To avoid this, samples that are known, or at least suspected of having the lowest mercury content should be analyzed first. As a guideline, samples with results *less than* 10x the RL which immediately follow a sample with a result greater than the LDR, should be reanalyzed to check for carryover.

4.1.10 Contamination from Samples (cross-contamination)**:** Significant laboratory or instrument contamination may result when untreated effluents, in-process waters, landfill leachates and other undiluted samples containing concentrations of mercury greater than 100 ppb are processed and analyzed. Samples known or suspected to contain Hg concentrations greater than 100 ppb should be diluted prior to bringing them into the laboratory whenever possible, or if prior dilution is not possible, the digestate should be diluted prior to analysis. Such samples should be handled with care to avoid contamination of other samples. Change gloves after handling samples known to contain high levels of mercury.

4.2 Chemical Interference

4.2.1 Any material which can absorb radiation at the 253.7-nm wavelength has the potential to cause a positive interference. Materials that inhibit the reduction of Hg^{2+} to Hg^0, or which inhibit the aeration of Hg^0 into the vapor phase have the potential to cause a negative interference. The sample digestion procedure is designed to eliminate common interferences of these types.

4.2.2 The most common interferences come from brine samples and samples containing high levels of sulfides. Use of additional potassium permanganate can remove most of these interferences, however, very high levels can lead to low mercury recoveries. Other interferences include chlorides and iodides (halides), gold, or copper (reported at levels >10 ppm). High levels of organic solvents, such as acetone, hexane, alcohols, and glycols can also interfere.

4.3 Physical Interference

4.3.1 Physical interference can result from a damaged or dirty optical cell (including cracks, smudges, or condensed water vapor), and air bubbles trapped in samples or introduced in-line due to leaks in tubing or junctions.

4.3.2 Inconsistent levels of water vapor within the optical cell can result in instrument drift. Water vapor is regulated through use of a dehydrator, however excessive variations in atmospheric conditions surrounding the dehydrator can result in varying performance. Temperature changes greater than 3-5 °C can also result in instrument drift. For best performance, the sample delivery and detection system should be kept in as stable an operating environment as possible.

5.0 Safety

5.1 Employees must abide by the policies and procedures in the Corporate Environmental Health and Safety Manual (CW-E-M-001), and in this document. This procedure may involve hazardous material, operations and equipment. This SOP does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of this SOP to follow appropriate safety, waste disposal and health practices under the assumption that all samples and reagents are potentially hazardous. Safety glasses, gloves, lab coats and closed-toe, nonabsorbent shoes are a minimum.

5.2 The toxicity or carcinogenicity of each chemical used in this method has not been precisely determined; however, each compound is treated as a potential health hazard. Exposure to these compounds is reduced to the lowest possible level.

5.3 The laboratory is responsible for maintaining a current awareness file of OSHA regulations for the safe handling of the chemicals specified in this procedure. OSHA rules require that a reference file of material safety data sheets (MSDS's) are made available to all personnel involved in these analyses. All MSDS's may be viewed on the Test America intranet.

5.4 Specific Safety Concerns or Requirements

5.4.1 This SOP uses several highly concentrated mineral acids, as well as strong oxidizers. Analysts must be familiar with proper response procedures for large and small spills, and for physical contact (see reference to CW-E-M-001 in sect 5.1). An acid spill kit is to be stored in a readily accessible location within the laboratory.

5.4.2 All digestion of acidified samples is to be conducted inside of a fume hood. The fume hood is periodically monitored to ensure its proper functioning and airflow requirements. This is especially important during soil digestion in which potassium permanganate can react with hydrochloric acid to produce chlorine gas.

5.4.3 Samples that contain high concentrations of carbonates or organic material, or samples that are at elevated pH may react violently when acids are added. Use extra care and add acids slowly to leachates, colored samples, samples containing bubbles or foam, samples with swollen containers, or sample with strong odors.

5.4.4 Chronic mercury exposure may cause kidney damage, muscle tremors, spasms, personality changes, depression, irritability and nervousness. Organo-mercurials may cause permanent brain damage. Because of the toxicological and physical properties of Hg, only trained personnel familiar with handling mercury standards should handle standards.

5.4.5 As recommended, the laboratory purchases a dilute standard of Hg so that its use won’t compromise the health and safety of the analyst. When samples known or suspected of containing high concentrations of mercury are handled, all operations are performed in a controlled area of the laboratory, preferably in a fume hood with adequate airflow and ventilation.

5.4.6 Mercury containing exhaust vapors leaving the instrument are passed through a column of activated carbon, filter trap containing gold or sulfur, or other suitable filter or trap in order to sequester mercury vapors away from the analyst.

5.4.7 While this procedure does call for the trace analysis of mercury at extremely small levels, it is still possible to be exposed to toxic levels of mercury during normal laboratory conditions. Mercury is at it most toxic when it is allowed to enter the bloodstream, therefore, any analyst who has an open wound or other such injury should take special care in avoiding mercury exposure.

5.4.8 The laboratory contains a mercury spill kit in case of serious mercury exposure. The kit is located in an area familiar to all that work in the laboratory, in the cabinet under the sink. Personnel can use mild soap with plenty of scrubbing in order to decontaminate skin. In the case of open wounds, professional help is to be sought immediately. All glassware, tools and surfaces are cleaned with sulfur powder in order to reduce any mercury present to non-volatile mercury sulfide. Washing the surface with reagent water will complete the cleansing process.

5.5 Primary Materials Used

The following is a list of the materials used in this method, which have a serious or significant hazard rating. **Note: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the MSDS for each of the materials listed in the table.** A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the MSDS for each material before using it for the first time or when there are major changes to the MSDS.

Material (1)	**Hazards**	**Exposure Limit (2)**	**Signs and symptoms of exposure**
Mercury (100 ppm in Reagent)	Oxidizer Corrosive Poison	0.1 Mg/M3 Ceiling (for Hg compounds)	Extremely toxic. Causes irritation to the respiratory tract. Causes irritation. Symptoms include redness and pain. May cause burns. May cause sensitization. Can be absorbed through the skin with symptoms to parallel ingestion. May affect the central nervous system. Causes irritation and burns to eyes. Symptoms include redness, pain, and blurred vision; may cause serious and permanent eye damage.
Sulfuric Acid	Corrosive Oxidizer Dehydrator Poison	1 Mg/M3-TWA	Inhalation produces damaging effects on the mucous membranes and upper respiratory tract. Symptoms may include irritation of the nose and throat, and labored breathing. Symptoms of redness, pain, and severe burn can occur. Contact can cause blurred vision, redness, pain and severe tissue burns. Can cause blindness.
Nitric Acid	Corrosive Oxidizer Poison	2 ppm-TWA 4 ppm-STEL	Nitric acid is extremely hazardous; it is corrosive, reactive, an oxidizer, and a poison. Inhalation of vapors can cause breathing difficulties and lead to pneumonia and pulmonary edema, which may be fatal. Other symptoms may include coughing, choking, and irritation of the nose, throat, and respiratory tract. Can cause redness, pain, and severe skin burns. Concentrated solutions cause deep ulcers and stain skin a yellow or yellow-brown color. Vapors are irritating and may cause damage to the eyes. Contact may cause severe burns and permanent eye damage.
Hydrochloric Acid	Corrosive Poison	5 PPM-Ceiling	Inhalation of vapors can cause coughing, choking, inflammation of the nose, throat, and upper respiratory tract, and in severe cases, pulmonary edema, circulatory failure, and death. Can cause redness, pain, and severe skin burns. Vapors are irritating and may cause damage to the eyes. Contact may cause severe burns and permanent eye damage.

Potassium Permanganate	Oxidizer	5 Mg/M3 Ceiling (for Mn compounds)	Causes irritation to the respiratory tract. Symptoms may include coughing, shortness of breath. Dry crystals and concentrated solutions are caustic causing redness, pain, severe burns, brown stains in the contact area and possible hardening of outer skin layer. Diluted solutions are only mildly irritating to the skin. Eye contact with crystals (dusts) and concentrated solutions causes severe irritation, redness, and blurred vision and can cause severe damage, possibly permanent.
Potassium Persulfate	Oxidizer	None	Causes irritation to the respiratory tract. Symptoms may include coughing, shortness of breath. Causes irritation to skin and eyes. Symptoms include redness, itching, and pain. May cause dermatitis, burns, and moderate skin necrosis.
1 – Always add acid to water to prevent violent reactions.			
2 – Exposure limit refers to the OSHA regulatory exposure limit.			

6.0 Equipment and Supplies

6.1 All equipment and supplies will be free of trace mercury, or at least at a level below the MDL of the method. All reusable equipment is cleaned according to the SOP BF-GP-003, Attachment 3, “Cleaning Procedure for Metals Glassware”.

6.2 Supplies and Equipment for Sample Preparation

6.2.1 50 mL graduated Digestion Tubes and Caps, with (at a minimum) marks at 30 mL and 50 mL. If cup volumes are not certified by the manufacturer, the 30 mL and 50 mL marks must be verified on a per lot basis. Verify by filling 5 individual tubes with reagent water to the appropriate (30 mL and/or 50 mL) mark by eye level and weighed. The average weight of water in the five cups must agree to within 1%. The results are logged into a spreadsheet and placed into a binder in the Mercury laboratory.

6.2.2 *Environmental Express* Hot Blocks capable of maintaining a temperature of 95°C. Hot Block temperature is verified daily.

6.2.3 Digestion tube racks to store and remove the samples from the hot blocks.

6.2.4 Bottle Repipettors for dispensing acids and reagents to samples.

6.2.5 Nalgene brand Wash Bottles for dispensing reagent water.

6.2.6 Thermometer covering a range of 0-150 °C. Thermometers are calibrated against a NIST certified thermometer in accordance with SOP BF-GP-020.

6.2.7 Analytical Balance accurate to ±0.1 mg (currently a Mettler AE200). Calibration to be verified daily. Balances are serviced yearly. (See SOP BF-GP-002)

6.2.8 Weigh boats and spatulas for soil samples and reagent preparation.

6.2.9 50 mL, 100 mL, and 2000 mL volumetric flasks (Class A) for preparation of reagents and standards.

6.2.10 *Eppendorf* Pipettes; varying volumes, preferably one for each volume to be dispensed. Minimally, pipettes in the following ranges are needed: 0.05-0.2 mL, 0.1-1.0 mL, 0.5-2.5 mL, and 2.0-10.0 mL. Pipettes are verified daily and calibrated quarterly in accordance with SOP BF-GP-001.

6.2.11 Time device for monitoring digestion step times.

6.3 Supplies and Instrumentation for Sample Analysis

6.3.1 15 mL test tubes for analyzing samples.

6.3.2 Mercury Adsorbent filter for the instrument exhaust line: currently used -- a mercury adsorbing activated carbon filter from *Perkin-Elmer*.

6.3.3 Pump tubing for delivery of samples and reagents to the instrument:
6.3.3.1 Yellow-Blue (0.51 mm) tubing: for $SnCl_2$ introduction.
6.3.3.2 Orange-Yellow (1.52 mm) tubing: for Sample introduction
6.3.3.3 Green-Green (1.88 mm) tubing: for waste drain

6.3.4 Cold-Vapor Atomic Absorption (CVAA) instrument capable of detecting in the range of 0.2 μg/L to 10.0 μg/L. Currently: (1) Leeman Labs PS200 II Automated Mercury Analyzer, and (1) Leeman Labs Hydra AA Automated Mercury Analyzer. Both instruments are operated using WinHg software version 1.1.

6.3.5 Various consumable and replacement instrument parts available and purchased from the instrument manufacturer.

7.0 Reagents and Standards

7.1 Reagents:

7.1.1 Laboratory Reagent Water: (DI H_2O); Deionized water from a purified source. Water will be monitored for Hg, especially after ion exchange beds are changed.

7.1.2 Silicon (IV) Oxide: (SiO_2); Used as a blank soil matrix substitute. High purity grade (typically 99.995% for metals).

7.2 Stock Acids: ***CAUTION!*** *Concentrated mineral acids are highly corro*sive.

7.2.1 Nitric Acid: (HNO_3); Concentrated, trace metals grade or equivalent.

7.2.2 Sulfuric Acid: (H_2SO_4): Concentrated, trace metals grade or equivalent.

7.2.3 Hydrochloric Acid: (HCl): Concentrated, trace metals grade or equivalent..

7.3 Prepared Reagents:

7.3.1 All prepared reagents are labeled accordingly at the time of preparation. This label must include the reagent name, preparation date, the analyst who prepared it, and the expiration date. Expiration dates must conform to the earliest expiration date of any chemical used in the preparation of the reagent. All information pertinent to the prepared reagents must be recorded in a reagent logbook or directly into the LIMS.

7.3.2 5% (wt/wt) Potassium Permanganate Solution ($KMnO_4$): Prepare by dissolving 100 g of $KMnO_4$ in 2000 mL of reagent water. This solution has a shelf life of six months. ***CAUTION****: strong oxidizer*.

7.3.3 5% (wt/wt) Potassium Persulfate Solution ($K_2S_2O_8$): Prepare by dissolving 100 g of $K_2S_2O_8$ in 2000 mL of reagent water. This solution has a shelf life of six months. Method 7470 only.

7.3.4 Sodium Chloride / Hydroxylamine Hydrochloride Solution ($NaCl$ / $NH_2OH{\bullet}HCl$): (*abbrev. HyHy*); Prepare by dissolving 240 g of NaCl and 240 g of NH_2OH-HCl in 2000 mL of reagent water. This solution has a shelf life of six months.

7.3.5 10% Hydrochloric Acid: (10% concentrated acid by volume.) Prepare by adding 2500 mL of concentrated HCL to a 25L container half-filled with reagent water and bring to the mark with reagent water.

7.3.6 Stannous Chloride Solution ($SnCl_2$ in HCl): Prepare by dissolving 100 g of $SnCl_2$ in 10% HCl. Dilute to the 1000 mL mark. The solution has a shelf life of one month. Store in a tightly closed container so that exposure to air is kept to a minimum. This solution should also be kept away from any mercury standard, reagent used in digestion or field sample.

7.3.7 Blank Matrix Solution (BMS): Fill a 2000 mL flask half way with reagent water. Measure 40 mL of concentrated HNO_3, 80 mL of concentrated H_2SO_4, 200 mL of $KMnO_4$, 80 mL of $K_2S_2O_8$ and 40 mL of Hydroxlyamine Hydrochloride. Swirl until solution is clear and colorless. Allow to cool to room temperature and bring to the 2000 mL mark with reagent water.

7.4 Purchased Standards:

7.4.1 100 µg/mL Hg Stock Standard #1: 100 ppm Hg#1 (SS). Purchased certified standard -- Certificate to be scanned and the original retained in the Mercury laboratory.

7.4.2 100 µg/mL Hg Stock Standard #2: 100 ppm Hg#2 (SS). Purchased certified standard -- Certificate to be scanned and the original retained in the Mercury laboratory. Purchased from a different vendor than #1.

7.4.3 Certified Soil Standard: ERA Soil Standard; “Metals in Soil” from *Environmental Resource Associates* – a standard reference material (SRM) containing a certified quantity of Mercury.

7.5 Prepared Standards:

7.5.1 All prepared standards must be properly labeled and recorded into a standards logbook or directly into the LIMS. For further information refer to SOP BF-GP-019 “Standards Traceability and Storage”.

7.5.2 10,000 ng/mL Hg Intermediate Standard #1: 10,000 ppb Hg#1 (IS). Measure 2 mL of concentrated HNO_3 to a 50 mL Class A volumetric flask half-filled with reagent water. Measure 5.0 mL of 100 µg/mL Hg Stock Standard #1, add to the flask and bring to the mark with reagent water. This standard expires in 6 months or when the original purchased stock standard is expired, whichever comes first.

7.5.3 10,000 ng/mL Hg Intermediate Standard #2: 10,000 ppb Hg#2 (IS). Measure 2 mL of concentrated HNO_3 to a 50 mL Class A volumetric flask half-filled with reagent water. Measure 5.0 mL of 100 µg/mL Hg Stock Standard #1, add to the flask and bring to the mark with reagent water. This standard expires in 6 months or when the original purchased stock standard is expired, whichever comes first.

7.5.4 Hg TCLP Spike: Add 5.0 mL of concentrated HNO_3 to a 100 mL Class A volumetric flask half-filled with reagent water. Measure 1.335 mL of 100 µg/mL Hg Stock Standard #1, add to the flask and bring to the mark with reagent water. The final concentration will be 1335 ng/mL. This solution expires in 6 months or when the original purchased stock standard is expired, whichever comes first.

7.5.5 100 ng/mL Hg Working Standard #1: 100 ppb Hg#1 (WS). Measure 2.0 mL of concentrated HNO_3 to a 50 mL Class A volumetric flask half-filled with reagent water. Measure 0.5 mL of 10,000 ng/mL Hg Intermediate Standard #1 to the flask and bring to the mark with reagent water. This standard expires 24 hours from the time of preparation and is to be prepared daily.

This standard is to be used for the preparation of the instrument calibration standards.

7.5.6 100 ng/mL Hg Working Standard #2: 100 ppb Hg#2 (WS). Measure 2.0 mL of concentrated HNO_3 to a 50 mL Class A volumetric flask half-filled with reagent water. Measure 0.5 mL of 10,000 ng/mL Hg Intermediate Standard #2 to the flask and bring to the mark with reagent water. . This standard expires 24 hours from the time of preparation and is to be prepared daily.

This standard is to be used for the preparation of the initial calibration verification solution (ICV), the continuing calibration verification solution (CCV), the addition of matrix spikes to samples (MS/MSD), and laboratory control samples (LCS) and blank spikes (BS).

8.0 Sample Collection, Preservation, Shipment and Storage

8.1 Aqueous samples are to be collected in plastic containers and preserved with Nitric Acid to pH < 2. Preserved samples can be stored at room temperature. Sample digestion and analysis must be completed within 28 days of sample collection.

8.2 Samples received at the laboratory unpreserved should be kept at 4°C and should be preserved as soon as possible. Allow samples preserved by the laboratory to stand for 24 hours prior to digestion.

Matrix	Sample Container	Min. Sample Size	Preservation	Holding Time [1]	Reference
Waters	HDPE	50 mL	HNO_3, pH < 2;	28 Days	40 CFR Part 136.3
Soils	Glass	3 g	Cool 4 $\underline{+}$ 2^oC	28 Days	N/A

[1] Inclusive of digestion and analysis.

9.0 Quality Control

**Refer to the TestAmerica Corporate Quality Assurance Plan for general information and more specific detail. Often project-specific quality assurance documents will provide overriding criteria to that presented below. Those criteria depending on project-specific data quality objectives may be more or less stringent than TestAmerica's QAP or the following criteria. The following criteria are subsequently presented as the minimum criteria of those criteria deemed applicable in the absence of project-specific DQO's.*

9.1 Sample QC

9.1.1 Method Blank (BLK or MBLK): A volume of reagent water (method 7470) or measured amount of silicon oxide (method 7471) which is carried through the sample preparation and analysis procedure along with each batch of samples (not to exceed 20 samples). This blank is useful in monitoring for contamination. May also be referred to as a Preparation Blank (PB).

9.1.1.1 Aqueous Blank (method 7470/245.1): Add 30 mL of reagent water to a digestion cup. Prepare and analyze as a sample with each batch of samples.

9.1.1.2 Soil Blank (method 7471): Add 0.6 g of Silicon (IV) Oxide Oxide (used as a soil matrix substitute) to a digestion cup. Prepare and analyze as a sample with each batch of solid samples.

9.1.1.3 TCLP Blank (method 7470): An associated extraction blank will accompany each set of TCLP extracts (EBLK). Add 30 mL of EBLK to a digestion cup (BLK1). Prepare and analyze with each batch of extracts. This is in addition to the regular aqueous blank (BLK2).

9.1.2 Blank Spike (BS):. A volume of reagent water spiked with a known concentration of mercury, which is carried through the preparation and analysis procedure along with each batch of aqueous samples (not to exceed 20 samples). The Blank Spike is employed to determine method accuracy. May also be referred to as a Laboratory Fortified Blank (LFB) or Laboratory Control Sample (LCS).

9.1.2.1 Water Blank Spike (method 7470/245.1): Add 30 mL of reagent water to a digestion cup and fortify with a known amount of mercury (spike with 2.0 mL of 100 ppb Hg#2 = 4 ppb Hg at a final volume of 50 mL). Prepare and analyze with each batch of samples.

9.1.2.2 TCLP Blank Spike (method 7470): Add 400 mL of EBLK to a 500 mL bottle and spike with 2.0 mL of Hg TCLP Spike, and preserve to pH <2 with nitric acid. Add 30 mL of the spiked EBLK to a digestion cup (4 ppb Hg at a final volume of 50 mL). Prepare and analyze with each batch of extracts.

9.1.2.3 Solid Blank Spike (for Wipes or project specific): Add 0.6 g of Silicon (IV) Oxide to a digestion cup and spike with 2.0 mL of 100 ppb Hg#2 (4 ppb Hg at a final volume of 50 mL).

9.1.3 Standard Reference Material (SRM): Method 7471. A solid matrix material containing a known quantity of mercury, which is carried through the preparation and analysis procedure along with each batch of solid samples (not to exceed 20 samples). The SRM serves the same purpose as the Blank Spike. The quantity of standard reference material used is selected to give a target result of 4 ppb in a final volume of 50 mL. This amount will vary by manufacturer lot.

Example: A lot of ERA Metals in Soil has a certified mercury concentration of 2.170 mg/kg. To obtain a theoretical instrument result of 4 ppb (µg/L) in a 50 mL final volume, use the following target amount of soil SRM:

$$4\ \mu g/L \times 0.050\ L \div 2.170\ \mu g/g = 0.09216\ g$$

9.1.4 Matrix Spikes: For each batch of samples (not to exceed 20 samples), a matrix spike (MS) should be processed on a routine basis. Spiked samples will be used to determine matrix effects on digestion and detection. A representative base sample is selected and a replicate quantity is added to an additional digestion cup. For SW846 and MCAWW this replicate sample is spiked with 2.0 mL of 100 ppb

Hg#2 (4 ppb Hg at a final volume of 50 mL).

9.1.5 Duplicates: For each batch of samples (not to exceed 20 samples), replicate samples should be processed on a routine basis. Replicate samples will be used to determine precision, and are either a method duplicate (MD) or matrix spike duplicate (SD or MSD; typical for SW846 and MCAWW). A matrix duplicate is just a replicate preparation of a selected representative base sample. A matrix spike duplicate is just a matrix duplicate that is spiked the same as a matrix spike.

9.1.6 Sample QC frequency and control limits:

Quality Controls	**Frequency**	**Control Limit**
Method Blank (MBLK or BLK)	1 in 20 or fewer samples	< Reporting Limit (SW846); < MDL (MCAWW)
Blank Spike (BS)[1]	1 in 20 or fewer samples	80-120% recovery (SW846); 85-115% recovery (MCAWW)
Matrix Spike (MS)[2]	1 in 20 or fewer (SW846); 1 in 10 or fewer (MCAWW	75-125% recovery (SW846) 70-130% recovery (MCAWW)
Matrix Spike Duplicate (MSD)[2] or Matrix Duplicate (MD)[2]	1 in 20 or fewer samples	75-125% recovery (MSD); or RPD < 20% (duplicates)
Standard Ref. Material (SRM)	1 in 20 or fewer samples	Specified by manufacturer on a per lot basis; typically about 70-130%

[1] Alternately referred to as a Laboratory Control Sample (LCS) or Laboratory Fortified Blank (LFB).
[2] The base sample for MS/MSD is arbitrarily selected, unless specifically requested by a client.

9.2 Instrument QC

9.2.1 All instrument QC standards are prepared and digested daily, typically at the same time as the instrument calibration standards (see section 10.2). Add 25-30 mL reagent water to a digestion cup. Spike with the volume of 100 ppb Hg#2 specified for each standard, and digest for a minimum of 30 minutes. The final QC sample volume is 50 mL.

9.2.2 Initial Calibration Verification (ICV): Prepared as described in 9.2.1 and spiked using 1.5 mL of 100 ppb Hg#2 (3 ppb at a final volume of 50 mL). The ICV checks the accuracy of the calibration and must be the first sample analyzed following a new calibration or at the start of a new analytical sequence.

9.2.3 Initial Calibration Blank (ICB): An unspiked blank sample prepared as described in 9.2.1. The ICB must be analyzed directly after the ICV.

9.2.4 Low Calibration Verification (LCV): Prepared as described in 9.2.1 and spiked using 0.1 mL of 100 ppb Hg#2 (0.2 ppb at a final volume of 50 mL). The LCV is at the same concentration as the lowest non-blank calibration point and is at or near the typical laboratory reporting limit (RL). The LCV must be analyzed following the

ICV and ICB at the beginning of any an analytical sequence. (Note: Many client QAPPs require that an additional LCV be analyzed at the end of each analytical sequence. Unless there is a specific reason not to, is a good practice to analyze an LCV at the end of each analytical sequence.) Formerly referred to as a CRA.

9.2.5 Continuing Calibration Verification (CCV): Prepared as described in 9.2.1 and spiked with 1.0 mL of 100 ppb Hg#2 (2 ppb at a final volume of 50 mL). The CCV is analyzed at the beginning and end of an analytical sequence, and at a frequency of every 10 samples, ensuring the continued accuracy of the calibration.

9.2.6 Continuing Calibration Blank (CCB): An unspiked blank sample identical to the ICB. The CCB must be analyzed directly after each CCV, and verifies that contamination has not accumulated over the analysis of the previous ten samples.

9.2.7 Serial Dilution (SRD): For each sample batch, a representative sample is selected (typically the base sample that is used for the MS/MSD). The sample is diluted 5X (1+4 dilution) using blank matrix solution (BMS) and is analyzed along with the base sample to evaluate sample matrix effects.

9.2.8 Instrument QC frequency and control limits:

Quality Check	**Conc.**	**Frequency**	**Control Limit**
ICV	3.0 ppb	Beginning of each analytical sequence	90-110% recovery (SW846); 95-105% recovery (MCAWW)
ICB	Blank	Beginning of each analytical sequence	< Reporting Limit
LCV	0.2 ppb	Beginning of each analytical sequence	50-150% recovery
CCV	2.0 ppb	Every 10 samples	80-120% recovery (SW846); 90-110% recovery (MCAWW)
CCB	Blank	Every 10 samples following each CCV	< Reporting Limit
SRD	N/A	1 for each sample batch	+/-10% of base sample / 5

10.0 Procedure

10.1 Sample Preparation

10.1.1 Samples to be prepared are selected from a report of available in-house samples separated by sample matrix and/or analysis method, and sorted by due dates (or any of a variety of user selectable sort criteria). Selected samples are added to a Preparation Bench Sheet (or Batch). For additional detail concerning batch creation criteria and procedures, refer to SOP BF-ME-001.

10.1.2 Samples of different matrix types (e.g. water, solid) or preparation/analysis methods (e.g. 7470, CLP) are typically prepared and analyzed separately. Samples assigned 7470 (SW846) and 245.1 (MCAWW) can be batched and analyzed together; but solid samples are separated from water samples, and CLP samples are separated from non-CLP samples. TCLP extracts may be analyzed with aqueous 7470 samples, but typically are not. Although not a method requirement, aqueous total samples are batched and analyzed separately from dissolved samples.

10.1.3 Based on the sample batches to be prepared and analyzed together, a sequential numerical order is established for the preparation and digestion cups are labeled as follows: Starting with '1' for the first client sample in the first batch, begin numbering samples sequentially until the base sample for the batch QC (MS/MSD or MD/MS) is reached. Assign the base sample 2 numbers and skip 1 cup leaving the rack position empty. (This is a placeholder for the serial dilution to be created prior to analysis.) The next two cups are the two batch QC samples. Continue with the next client sample and continue to the last client sample, followed by the batch blank spike (BS) and method blank (BLK). Continue with additional batches of the same type as necessary (up to 88 labeled sample cups – the number of positions on the Leeman autosampler). It is a good practice to clearly mark the cups to be used for spiked batch QC samples (MS/MSD/BS).

Note: This assigned sequential order is used throughout both the preparation and analysis procedures.

10.1.4 Obtain the appropriate client samples from the cooler(s) or metals sample storage area. Arrange the samples on a sample cart in the order designated above.

10.1.5 Aqueous Sample Digestion (method 7470, 245.1):

10.1.5.1 Making sure the cap is on securely, shake or invert the container several times to homogenize the sample, and pour 30 mL of the sample into the appropriately labeled digestion cup. (Refer to section 5.5 of SOP BF-GP-005 for further instruction on sample homogenization.) Take care to use the appropriate sample bottle when pouring sample, MD/MS or MS/MSD groups. Some clients provide additional bottles for each of the samples in this group, However, much of the time, a single sample bottle will be used for all three aliquots. Reagent Water (30 mL) is used for the BS and BLK samples. For TCLP extracts, use the pre-spiked MS/MSD and BS volumes (typically prepared in the Metals Digestion Lab).

10.1.5.2 Spike all MS, MSD, BS samples as specified in the Batch QC section.

10.1.5.3 Add the following reagents to all samples in each batch:

- 1.0 mL Nitric Acid (HNO_3): **Caution!** Add slowly to leachates. Acid may react vigorously or violently with some samples. Highly reactive samples may require additional nitric acid.
- 2.0 mL Sulfuric Acid (H_2SO_4): **Caution!** Add slowly to leachates. Acid may react vigorously or violently with some samples.
- 5.0 mL Potassium Permanganate ($KMnO_4$)
- 2.0 mL Potassium Persulfate ($K_2S_2O_8$)

10.1.5.4 Cap the digestion cups loosely enough so that pressure does not build up can be evacuated, but also tight enough so that the caps stay on, and that volume loss due to the heating minimized.

10.1.5.5 All samples should remain a purple color for at least 15 minutes after adding the potassium permanganate. If any sample becomes clear or otherwise loses its purple color, add an additional 5 mL of the potassium permanganate to ALL samples in the batch, including batch QC samples. If the purple color fades once again, re-prepare the affected sample(s) using a reduced initial volume, noting the volume used on the bench sheet, and dilute to 30 mL using reagent water.

10.1.5.6 Put the samples on the hot block at for 2 hours. Remove and let cool.

10.1.5.7 Uncap each digestion cup and add 2.5 mL of sodium chloride – hydroxylamine hydrochloride (HyHy) to each sample. Allow bubbling to subside, and top each sample to the 50 mL mark with reagent water.

10.1.5.8 Replace the cap tightly and shake vigorously for two to three seconds or until most of the purple color has faded. Vent the digestion cup. A brown residue of undissolved manganese dioxide (MnO_2) may remain on the bottom or sides of the digestion cup. It is of no concern, and may dissolve over time.

10.1.6 Solid Sample Digestion:

10.1.6.1 Homogenize each sample as described in section 5.6 of SOP BF-GP-005. Add 0.6 g of each sample to the appropriately labeled digestion cup. Take care to use the appropriate sample bottle when weighing sample, MD/MS or MS/MSD groups. Some clients provide additional bottles for each of the samples in this group, However, much of the time, a single sample bottle will be used for all three. For the SRM sample, add the calculated target amount of ERA Standard (see section 9.1.3). For the BLK sample add 0.6g Silicon (IV) Oxide (SiO_2). Add approximately 5-10 mL reagent water to each cup (enough to cover the sample).

10.1.6.2 Spike all MS, MSD, BS samples as specified in the Batch QC section.

10.1.6.3 Add the following reagents to all samples in each batch:

- 1.0 mL Nitric Acid (HNO_3). **Caution!** Acid may react vigorously or violently with some samples. Highly reactive samples may require additional nitric acid.
- 3.0 mL Hydrochloric Acid (HCl)

10.1.6.4 Heat the samples uncapped on the hot block at 95°C for 2 minutes. Remove and let cool.

10.1.6.5 Add the following to all samples in each batch:

- 5-10 mL Reagent Water

- 10-15 mL Potassium Permanganate ($KMnO_4$): **Caution!** $KMnO_4$ can react with HCl to produce chlorine gas.

10.1.6.6 Cap the digestion cups loosely enough so that pressure does not build up and can be evacuated, but also tight enough so that the caps stay on, and that volume loss due to the heating minimized.

10.1.6.7 Put the samples on the hot block at for 30 minutes. Remove and let cool.

10.1.6.8 Uncap each digestion cup and add 2.5 mL of sodium chloride – hydroxylamine hydrochloride (HyHy) to each sample. Allow bubbling to subside, and top each sample to the 50 mL mark with reagent water.

10.1.6.9 Replace the cap tightly and shake vigorously for two to three seconds or until most of the purple color has faded. Vent the digestion cup. A brown residue of undissolved manganese dioxide (MnO_2) may remain on the bottom or sides of the digestion cup. It is of no concern, and may dissolve over time.

10.1.7 Analysis of sample digestates must be performed within 24 hours of digestion or the sample will need to be re-digested.

10.1.8 Some aqueous samples and most solid samples will contain sediments or other solid material that may physically interfere with the analysis by clogging or restricting flow through the instrument sample introduction tubing. These samples may be filtered.

10.2 Calibration

10.2.1 Before any instrument is used as a measurement device, the instrument response to known reference materials must be determined. Instrument calibration for mercury analysis is performed at a minimum of each day the analysis is to be performed. A six point linear calibration is used.

10.2.2 Preparation of Calibration Standards: Six standards of known mercury concentration are prepared by dilution of the 100 ppb Hg#1 working standard.

10.2.2.1 Add 20-30 mL Reagent Water to six digestion cups. Spike each cup with the appropriate volume of 100 ppb Hg#1 (see chart below).

10.2.2.2 Digest each standard using the Aqueous digestion procedure from section 10.1.5 using a digest time of 30 minutes. This ensures that the instrument calibration is matrix matched to the samples to be analyzed. Final volume for the calibration standards is 50 mL.

10.2.2.3 Allow the calibration standards to fully cool to room temperature before using (minimum 30 minutes). Failure to allow the the standards to cool sufficiently will likely result in the need to recalibrate the instrument.

Calibration Standard	Standard Conc. in 50 mL vol. (ppb)	100 ppb Hg#1 Spike vol. (mL)	Final Conc. Aqueous Samples (μg/L)	Final Conc. Solid Samples (mg/kg)	Control Limit
S1	0	0	0	0	SD < 5000
S2	0.2	0.1	0.33	0.017	%RSD < 30
S3	1.0	0.5	1.67	0.083	%RSD < 5
S4	2.0	1.0	3.33	0.17	%RSD < 5
S5	5.0	2.5	8.33	0.42	%RSD < 5
S6	10.0	5.0	16.67	0.83	%RSD < 5

10.2.3 Calibrating the Instrument**:** This procedure outlines the basic steps to calibrating the instrument. For specific details concerning the operation of the Leeman Analyzer and/or instrument software (WinHg), refer to instrument User's Guide and Manual. For the purposes of this SOP it is assumed that the instrument software and settings are configured for the analysis of samples by the methods covered in this SOP and for the generation of data in a format and manner compatible with TestAmerica Buffalo laboratory operations. It is also assumed the analyst is familiar with and properly trained in the use of the instrument and software.

10.2.3.1 Instrument operating parameters have been demonstrated to meet the necessary requirements for the analyses described in this SOP. instrument parameters may be altered as need arises, however, significantly altering these parameters may necessitate reevaluation of instrument and method detection limits prior to implementation for sample analysis. Currently used instrument operating parameters are:

- Pump Rate = 7 mL/min
- Gas Flow Rate = 0.7 L/min
- Sample Uptake Time = 10 sec
- Sample Integration Time = 10 sec
- Rinse Time = 40-60 sec

10.2.3.2 Perform any needed instrument maintenance prior to calibration. Note any maintenance performed in the instrument maintenance log. Detailed instructions and manufacturer suggested scheduling for the performance of most routine instrument maintenance is available within the instrument software using the "Perform Maintenance" command under the "Utility" menu.

10.2.3.3 Turn on Lamp, Pump, and Gas. Fill the rinse bath with 10% HCl. The lamp will require a minimum of 15 minutes to warm up. For best performance, allow the pump to run 15 minutes to flush the tubing and allow it to settle into place. (For new tubing, allow a 30 minute or longer "break-in" time.)

10.2.3.4 Perform a Lamp Adjustment. Record the setting and baseline reading in the maintenance logbook. A large day-to-day change in the lamp setting (without changes to the instrument optical cell or optical bench) may indicate a need for further maintenance.

10.2.3.5 Pour the calibration standards to the appropriate cups on the autosampler. The 6 standards and concentrations are currently set up in the S1-S6 positions.

10.2.3.6 Use ‘StdAuto’ to analyze 3 replicates of each of the 6 calibration standards (select S1-S6 and Repetitons 1-3) to generate the calibration curve. In addition to the control limits specified above, a correlation coefficient (rho) of at least 0.995 is required; however, for best performance it is strongly recommended that the correlation coefficient be greater than 0.9995. A lower value may indicate a need for instrument maintenance or poorly prepared calibration standards.

10.2.3.7 Calibrations are evaluated in accordance with SOP BF-GP-006, and against the criteria specified in this section. If all criteria are met, accept the calibration. Print a copy of the calibration screen and export the calibration to a data file. Attach a copy of the calibration screen to each data set analyzed using that calibration.

10.3 Sample Analysis

10.3.1 Pre-Run Setup Checklist: Sample analysis is only performed on a properly maintained and calibrated instrument. Prior to beginning an analytical sequence verify or perform the following steps. Note: these items are not necessarily in a specific sequential order.

10.3.1.1 Select analyst initials in the “User Name” on the “Main” tab:

10.3.1.2 Lamp is on and wamed up (at least 15 min); Gas is turned on; Pump is on. Controls for all 3 are on the “Control” tab.

10.3.1.3 Instrument is calibrated and calibration is accepted. “Cal Curve” tab.

10.3.1.4 Stannous Chloride bottle has sufficient volume, and 10% HCl rinse bath is topped off.

10.3.1.5 Calibration Check Standard cups (Instrument QC) have sufficient volume. Current setup has instrument QC in the following autosampler rack positions: C1 = ICB/CCB; C2 = ICV; C3 = CCV; C4 = LCV.

10.3.1.6 Create a data file from the “File” menu, or select an existing file from the “Dataset” field. The file contains information which designates the instrument used, date and sample analysis/batch matrix type using the following convention:

- A single letter instrument identifier (Leeman1 = G; Leeman2 = H; Leeman 3 = J).
- Numeric date: month, day (2 digits each), and year (1 digit); e.g. Jan 15, 2008 = 01159.
- A two character analysis/batch matrix type identifier
    - C# = Calibration data file
    - D# = Dissolved Hg
    - S# = Total Hg - Solids
    - TC = TCLP extracts
    - W# = Total Hg – Waters

Example: The second analytical sequence (run data file) of aqueous samples for total Hg analyzed on August 12th, 2009 using the Leeman 2 instrument would have a Data File name of H08129W2.

10.3.1.7 For the samples to be analyzed, label test tubes using the same numbering scheme used for the digestion cups.

10.3.1.8 Pour approximately 8-10 mL of each sample from the digestion cups into the test tubes, and place them in a 44 position autosampler rack.

10.3.1.9 Create the serial dilution (SRD) samples: Combine 2 mL of the sample to be diluted with 8 mL of Blank Matrix Solution (BMS). The dilution factor for the SRD sample is 5 (@5 in the autosampler table).

10.3.1.10 Create Autosampler files (using the Rack Editor) containing all of the batch samples and calibration check standards (instrument QC) to be analyzed. Autosampler table files are named similar to Data Files substituting #1, #2, #3, etc. in place of the 2-character sample type identifier. One file is needed for each rack of up to 44 samples. Auto-sampler table columns are populated as follows:

- cup#: This column is pre-populated from 1-44 (the number of positions per sample rack). The cup number should match up with the digestion cup number and test tube number for the 1st rack of 44 samples. For the 2nd rack (if needed), the cup# will equal the digestion cup number minus 44.

- sample ID: Batch sample IDs (up to 10 characters) are typed (or scanned from a barcode) into this column. Any portion of a sample ID beyond 10 characters must be typed into the next column (extended ID)

- extended ID: used to continue the sample ID when longer than 10 characters; routinely used for Batch QC samples.

- weight and volume: these columns are not used and are pre-populated with 1.0000

- <u>? A D F P S U SC UI US… (Cup Macro Column)</u>: This column uses macro codes to send instructions to the instrument software. All instrument QC samples are analyzed by including the check standard cup position (C1=ICB/CCB; C2=ICV; C3=CCV; C4=LCV). The CP macro code tells the instrument to execute the preceding macro codes prior to analyzing the sample in that cup #; otherwise, the macro codes in a given row execute following analysis of the sample in that cup.The S and P macro code combination is used to calculate the %recovery of BS and SRM batch QC samples.

- <u>Macro Code Layout</u>: The following macro codes are used for a typical analysis (see example table layout below):

- Cup#1 = C2 C1 C4 C3 C1 CP
- Cup#10,20,30, etc = C3 C1
- Cup containing a BS or SRM sample = S P (Note: S P C3 C1 can be used if the sample falls on cup normally requiring a C3 C1.
- Last Cup = C3, C1 (or C4, C3, C1 as required)

<u>10.3.1.11</u> Select the Autosampler file(s) and corresponding cup positions to be analyzed on the “Sample” Tab. Check that the samples/sample racks are in their proper positions on the autosampler.

<u>10.3.1.12</u> Select “Run Auto” on the “Sample” tab. A full rack of 88 cups including all QC will take about 3.0 - 3.5 hours.

<u>10.3.1.13</u> Samples with results outside of the calibration range must be diluted to within range and reanalyzed. If sample dilutions are required then add them to the end of the run. Append the autosampler table as needed with the sample ID. The dilution factor is added to the ‘extended ID’ column preceded by the ‘@’ character.. Include any check standard macro codes as appropriate.

<u>10.3.1.14</u> Perform a preliminary on-screen review of the data for QC failures or other requirement compliances (eg QAPPs) . Due to the 24h holding time constraint in analyzing mercury digestates, it is strongly recommended that the analyst perform any needed reanalysis (not requiring sample redigestion) immediately and within the same data file. This will reduce the need for unnecessary sample redigestion and simplify data review and reporting.

Example Autosampler Table for Batch 9E06054.

cup	sample ID	extended ID	weight	volume	? A D F P S U SC UI C1..C7
1	RSE0050-01		1.0000	1.0000	C2 C1 C4 C3 C1 CP
2	RSE0050-02		1.0000	1.0000	
3	RSE0050-09		1.0000	1.0000	

cup	sample ID	extended ID	weight	volume	? A D F P S U SC UI C1..C7
4	SEQ-SRD	@5	1.0000	1.0000	
5	9E06054-MS	1	1.0000	1.0000	
6	9E06054-MS	D1	1.0000	1.0000	
7	RSE0050-12		1.0000	1.0000	
8	RSE0050-13		1.0000	1.0000	
9	RSE0050-14		1.0000	1.0000	
10	RSE0122-04		1.0000	1.0000	C3 C1
11	RSE0122-05		1.0000	1.0000	
12	9E06054-BS	1	1.0000	1.0000	S P
13	9E06054-BL	K1	1.0000	1.0000	C4 C3 C1
14			1.0000	1.0000	

The above table would result in a run sequence as follows:

ICV
ICB
LCV
CCV
CCB
↑
10 samples (with the given ID#s)
↓
CCV
CCB
↑
3 samples (with a spike recovery calculation on the cup12 sample)
↓
LCV
CCV
CCB

10.3.2 Post-Analysis Checklist: Performed once all sample analyses are complete.

10.3.2.1 In the WinHg Database program ‘Report’ tab, select the appropriate data file to be reported. (Samples to be reported can be selected using a combination of the ‘Batch List’ and ‘Records List’ sections.) Select ‘Generate Report’. Reports can be generated on-screen (“Report” Format, ‘Viewer” Destination -- viewable via the ‘Viewer’ tab), to the printer (‘Report” Format, ‘Printer” Destination), or to a file (‘PRN File” Format, “Disk File’ Destination). The current report format setting is ‘HgRpt’.

10.3.2.2 Generate a printed report of the raw data, and a PRN disk file (used to import data to the LIMS). The file should be named the same as the instrument data file that the samples were run in. Save the file directly to the H-Drive (Lab Data) in the folder *Ward/Sdgs/Instdata/Mercury*. Click ‘Generate’ after creating the file to write the data to the file. Raw instrument files are backed up to a network drive in accordance with SOP BF-IS-010.

10.3.2.3 Record solution ID#s for the ICV, ICB, LCV, CCV, and CCB directly onto the raw data report.

10.3.2.4 Attach a hard copy (screen–shot) of the Calibration Curve screen to the printed raw data report. Record the following information on the Calibration page:

- Analysis Date
- Analyst Initials
- Instrument Name
- Solution ID#s for the Calibration Standards
- Calibration File Name (eg H08129C1)
- Data File Name the calibration was used for (eg H08129CW)
- Batch ID#s for the analyzed batches

11.0 Calculations / Data Reduction

11.1 Accuracy

$$\text{ICV / CCV / LCV / Blank Spike \%Recovery} = \frac{\text{observed concentration}}{\text{known concentration}} \times 100$$

$$\text{MS / MSD \% Recovery} = \frac{\text{(spiked sample) - (unspiked sample)}}{\text{spiked concentration}} \times 100$$

11.2 Precision (RPD)

$$\text{Matrix Duplicate (MD)} = \frac{|\text{orig. sample value - dup. sample value}|}{[(\text{orig. sample value + dup. sample value})/2]} \times 100$$

11.3 Wet-Weight Basis

Sample Concentration (mg/kg) = $C \times V/W$

Where: C = concentration in extract (mg/L)
V = Volume of the digestate (L, 50 mL = 0.05 L)
W = Weight of sample aliquot (not dried) extracted (g × 0.001 = kg)

11.4 Pecent Solids

To report percent solids in solid samples, calculate as follows:

% Solid (S) = $DW / WW \times 100$

Where: DW = Sample weight (g) dried (dry weight)
WW = Sample weight (g) before drying (wet weight)

11.5 Dry-Weight Basis

Sample Concentration (mg/kg) = $(C \times V) / (W \times S)$, *or*
Sample Concentration (mg/kg) = $C \times V / WW$

11.6 Calculation of extract concentrations are automatically done by the system's software.

11.7 Calculation of sample concentrations from measured extract concentration are done by the LIMS system.

11.8 Contingencies for Handling Out-of Control or Unacceptable Data

11.8.1 Data is to be evaluated in accordance with SOPs BF-GP-012 and BF-ME-013.

11.8.2 If an ICV, ICB, or opening LCV falls out of acceptance limits, discontinue the analysis to correct the problem, then Restart the analysis. Note: Instrument re-calibration may be required.

11.8.3 If any CCV or CCB falls out of acceptance limits, the preceeding and following 10 samples must be evaluated. If a BS or BLK fails, the entire batch of samples must be evaluated.

- For high CCVs and Blank Spikes, non-detect samples may be accepted. All other affected samples must be reanalyzed.
- For low CCVs, all affected samples must be reanalyzed. For low Blank Spikes, the batch must be reprepared. and reanalyzed.
- For High CCBs and BLKs, non-detect samples may be accepted. Samples greater than 10x the CCB or BLK result may also be accepted. All other affected samples need to be reanalyzed or reprepared and reanalyzed.

11.8.4 A Job Exception Report form may need to be filed if extensive problems are noted within any one sample or analysis. The analyst performing the run completes these forms. A Job Exception Report form should be completed and filed with the Project Manager and QA Manager for any of the following conditions:

- Holding times exceeded
- Insufficient sample volume for re-digestion
- Re-digestion required due to sample batch QC failure
- Unusual sample matrix or sample reactivity which requires deviation from this SOP

11.8.5 In the event of unknown positives or sample matrix which present the analyst with questionable data, the project manager shall be notified so the client may be contacted and involved in the decision process and course of action.

11.8.6 When an out of control situation occurs, the analyst must use his/her best judgment and use any available resources to determine the corrective action to be taken. The analyst may need to seek immediate assistance from the supervisor, laboratory director, project manager, QA personnel or other experienced members of the staff if he/she is uncertain of the proper course of action. The test may need to be stopped until the problem is corrected since the problem may be instrumental and not chemical. Out of control data will never be released without the approval of the Supervisor, QA Manager, or Laboratory Director.

12.0 Method Performance

12.1 Method Detection Limit Study (MDL)

The method detection limit (MDL) is the lowest concentration that can be detected for a given analytical method and sample matrix with 99% confidence that the analyte is present. The MDL is determined according to the laboratory's MDL SOP BF-QA-001. MDLs reflect a calculated (statistical) value determined under ideal laboratory conditions in a clean matrix, and may not be achievable in all environmental matrices. The laboratory maintains MDL studies for analyses performed; these are verified at least annually unless method requirements require a greater frequency. MDLs are verified on each instrument to which they will apply via annual MDLV studies. Instrument detection limits (IDL) are determined for each instrument on a quarterly basis.

12.2 Training Requirements

12.2.1 Analyst training will adhere to requirements specified in SOP BF-QA-004

12.2.2 The department supervisor has the responsibility to ensure that this procedure is performed by analysts with the required experience and properly trained in its use.

12.2.3 The analyst must complete laboratory safety orientation training that includes, but is not limited to, PPE requirements, chemical handling, and electrical safety.

12.2.4 The analyst must read the MSDS for all chemicals used in this method.

12.2.5 The analyst must read and understand the contents of this SOP and the Method used as a reference for this SOP.

12.2.6 The analyst must successfully complete a Demonstration of Capability (DOC) before training in this method is deemed to be complete.

12.3 Demonstration of Capability (DOC)

12.3.1 Initial Demonstration of Capability is performed upon completion all other aspects of training. A completed IDOC is the final step of analyst training and allows the analyst to perform the method without trainer supervision.

12.3.2 Continuing Demonstration of Capability is performed annually. This ensures that the analyst has remained proficient in performing the method and no retraining is necessary.

12.3.3 DOC will be performed as described in SOP BF-QA-004 section 5.8.

13.0 Pollution Control

It is TestAmerica's policy to evaluate each method and look for opportunities to minimize waste generated (i.e., examine recycling options, ordering chemicals based on quantity needed, preparation of reagents based on anticipated usage and reagent stability). Employees must abide by the policies in Section 13 of the Corporate Environmental Health and Safety Manual (CW-E-M-001) for "Waste Management and Pollution Prevention."

14.0 Waste Management

14.1 Waste management practices are conducted consistent with all applicable rules and regulations. Excess reagents, samples and method process wastes are disposed of in an accepted manner. Waste description rules and land disposal restrictions are followed. Waste disposal procedures are incorporated by reference to section 13 of the Corporate Safety Manual for "Waste Management and Pollution Prevention".

14.2 The following waste streams are produced when this method is carried out:

- Acidic waste from samples and sample digests. Waste generated will contain Nitric Acid and will therefore be disposed of as "AN" waste in accordance with SOP BF-WM-001.

15.0 References / Cross-References

15.1 EPA Method 7470A – Mercury in Liquid Waste

15.2 EPA Method 7471A – Mercury in Solid Waste

15.3 40 CFR Part 136 (MCAWW) (Revision B), "Guidelines Establishing Test Procedures for the Analysis of Pollutants under the Clean Water Act" U.S. Environmental Protection Agency.

15.4 EPA 600/4-79-020 Methods 245.1, Revision B; SW-846, 3rd Edition, Method 7470A

15.5 "Method 1631, Revision B: Mercury in Water by Oxidation" (40 CFR 136, Revision B) U.S. Environmental Protection Agency, May 1999.

15.6 The following SOPs have been referenced, or are relevant to, procedures described in this document, and should be referred to for more detailed information on the indicated topics:

BF-ME-001	Metals Department Batching Procedure
BF-ME-013	Metals Data Review
BF-GP-001	Autopipets (Eppendorfs), Syringes, Repipettor Calibration
BF-GP-002	Balances, Reagent Water, Temperature Control Devices
BF-GP-003	Glassware Cleaning
BF-GP-004	Dry Weights
BF-GP-005	Sample homogenization and sub-sampling
BF-GP-006	Initial Calibration Evaluation
BF-GP-011	Sample Storage and Handling
BF-GP-012	Data Review Requirements
BF-GP-019	Standard Storage and Traceability
BF-GP-020	Thermometer Calibration
BF-IS-010	Instrument Data File Backup
BF-WM-001	Waste Management
BF-QA-001	Determination of MDLs
BF-QA-004	Personnel Training (for DOC's)

16.0 Method Modifications:

Item	Method xx	Modification
01	7470/7471/ 245.1	The volumes have been minimized for preparation of all methods listed, although the chemistry remains unchanged. This change fits our preparation equipment and minimizes waste.
02	7470/7471/ 245.1	*Environmental Express* Hot Blocks and plastic digestion cups replace Hot Plates and BOD bottles for sample preparation.

17.0 Attachments

17.1 Attachment 1: Manufacturer recommended positioning of the computer/analyzer/autosampler system.

17.2 Attachment 2: Sample Water Digestion Batch Bench Sheet

17.3 Attachment 3: Sample Soil Digestion Batch Bench Sheet

17.4 Attachment 4: Example Instrument Calibration Page

18.0 Revision History

Revision 3, dated 25 January 2010

- Replaces previous SOP BF-ME-011, revision 2
- Spelling, Grammar, & Formatting corrections
- Section 4.1.9 Added specific guideline for sample reanalysis to check for carryover from high level samples
- Section 4.2 & 4.3 Additional detail provided concerning various chemical vs. physical interferences
- Section 9.1.2.3 Removed references to AFCEE and USACE
- Sections 6.26, 6.27, 6.2.10, 10.2.3.7 Added missing cross-references to other relevant SOPs
- Attachment 4 - Title renamed for clarity

Revision 2, dated 02 September 2009

- Replaces previous SOP BF-ME-011, revision 1
- Section format changes from STL to TestAmerica Standard format completed. Several section numbers have changed, and several new tables have been included. References to outdated SOP reference numbers updated.
- Incorporated contents of interim change from July 07, 2008 concerning concentration of purchased mercury stock standards (1000 ppm → 100 ppm) and concentrations of prepared intermediate standards (20,000 ppb → 10,000 ppb, and elimination of 2000 ppb).
- Changed spike amount for the blank spike for aqueous total mercury batches from 2.0 ppb to 4.0 ppb so that the blank spike and matrix spike levels are now the same, which is in better accordance with SW-846. The 2.0 ppb blank spike had been implemented to accommodate AFCEE/USACE. A 2.0 ppb blank spike will still be used for AFCEE/USACE at which point that becomes necessary.
- Corrected Table 9.1.6 Sample QC control limits to agree with EPA methods:
  1. MS/MSD %recoveries from 20% to 25% (SW846) and 30% (MCAWW)
  2. MD/MSD %RSD from 30% to 20%
- Changes to several sections reflecting differences in operation between AIMS and ELEMENT LIMS systems; including the following abbreviation changes:
  1. CRA → LCV
  2. SD → MSD
  3. LCS and LFB → BS or SRM
  4. MBLK → BLK

- Added details concerning data file and autosampler file naming conventions
- Added details concerning sample preparation cup and analysis test tube numbering system
- Added an example autosampler table for demonstration of use of extended ID field for sample IDs longer than 10 characters, and for improved clarity in demonstrating the use of macro codes for execution of instrument QC.
- Reformatted sample preparation section to separate Aqueous from Solid digestion steps
- Rewrote calibration and sample analysis sections to better depict current use of instrument software in setting up, calibrating, and sample analysis. Added section on instrument operating parameters
- Added Sample Calibration Page Attachment
- Updated Example batch attachments from AIMS to ELEMENT batches
- Changed potassium permanganate added to soil samples from 10 mL to 10-15mL.

Revision 1, dated 15 April 2008

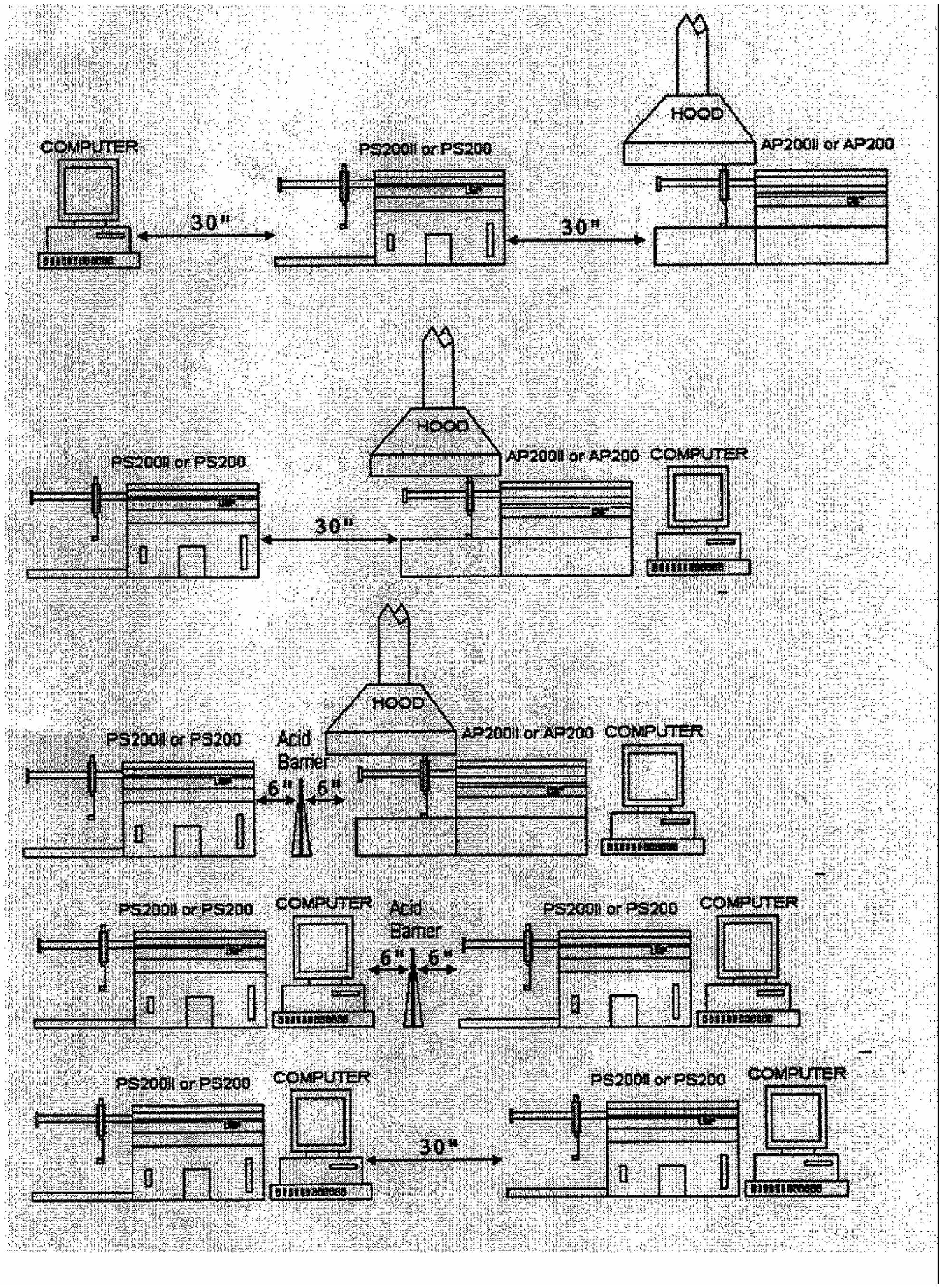
- Replaces previous SOP BF-ME-011, revision 0
- Sections 10.3.7, 10.3.8, 10.3.9 and 10.4. Edited for improved clarity regarding stock standard, intermediate standard, and working standard preparation; and to correct copying errors introduced in transitioning SOP formats from AME-MERCURY-50, rev.7 to BF-ME-011, rev.0.
- Section 12.7 and 12.8. Correct 100 ppb to Hg#2 instead of Hg#1
- Sections 10.4.3 and 10.4.4. Moved reference to preparation of MS/SD, LCS, and LFB from Section 10.4.3 to Section 10.4.4.
- Section 12.1.2. Changed quantity of water added to soil MBLK to 10 mLs and removed "Carry the MBLK through the entire digestion process" (redundant with 12.1)
- Section 14.4. Added addition of approximately 10 mLs reagent water to cover soil samples

Revision 0, dated 30 November 2007

- Replaces previous SOP AME-MERCURY-50, revision 7
- Section 9.2.5 correct to weekly to daily for pipette verification
- Sections 12.2.2, 12.2.3, 12.3, and 12.6 correct 100ppb to Hg#2 instead of Hg#1
- Section 12.4 correct from 1:3 to 1:5 serial dilution
- Section 14.26 replace 40CFR with MCAWW
- Section 14.31 deleted turn off argon gas valve

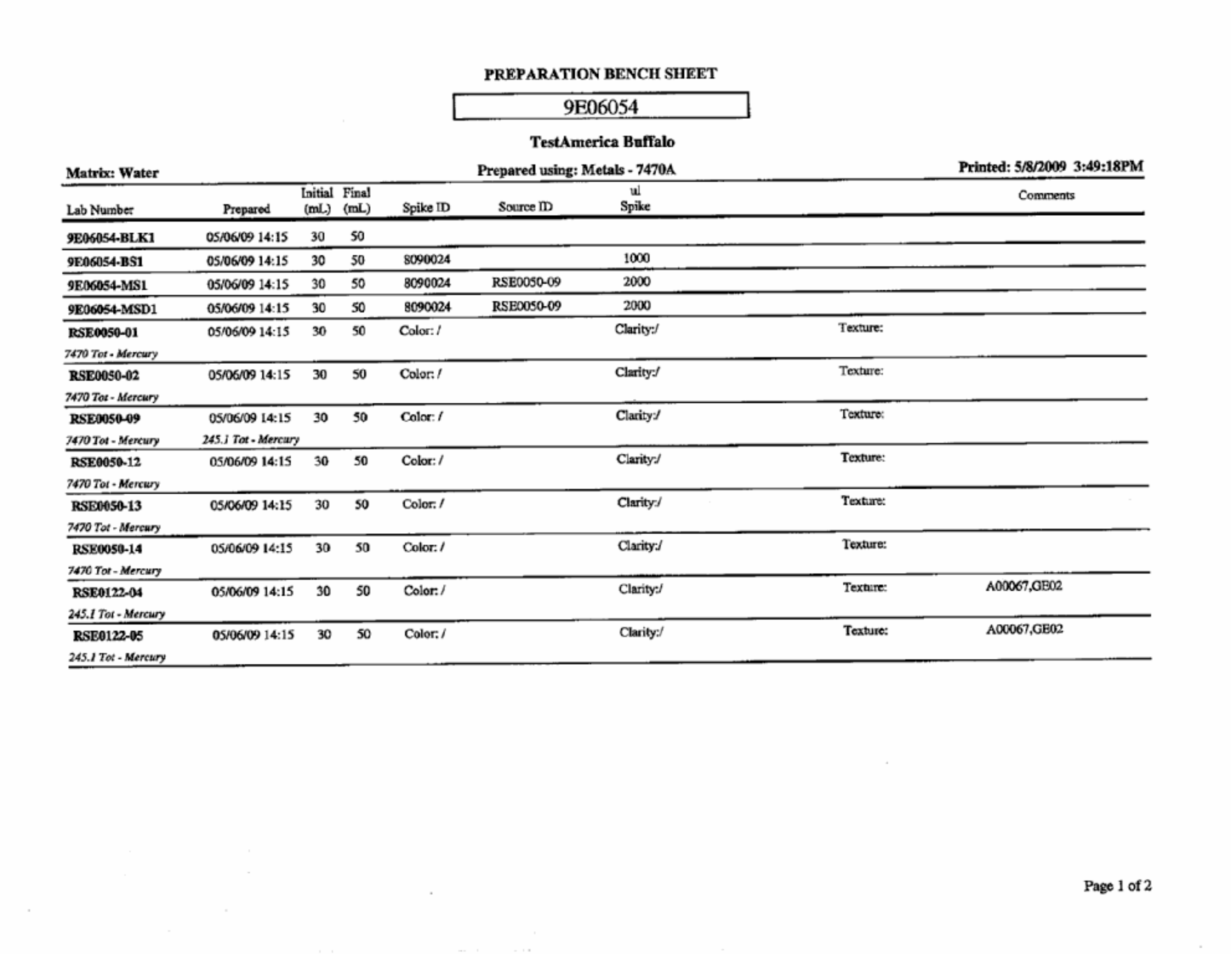
Attachment 1

Manufacturer recommended positioning of the computer/analyzer/autosampler setup.



Attachment 2

Sample Water Digestion Batch Bench Sheet



PREPARATION BENCH SHEET

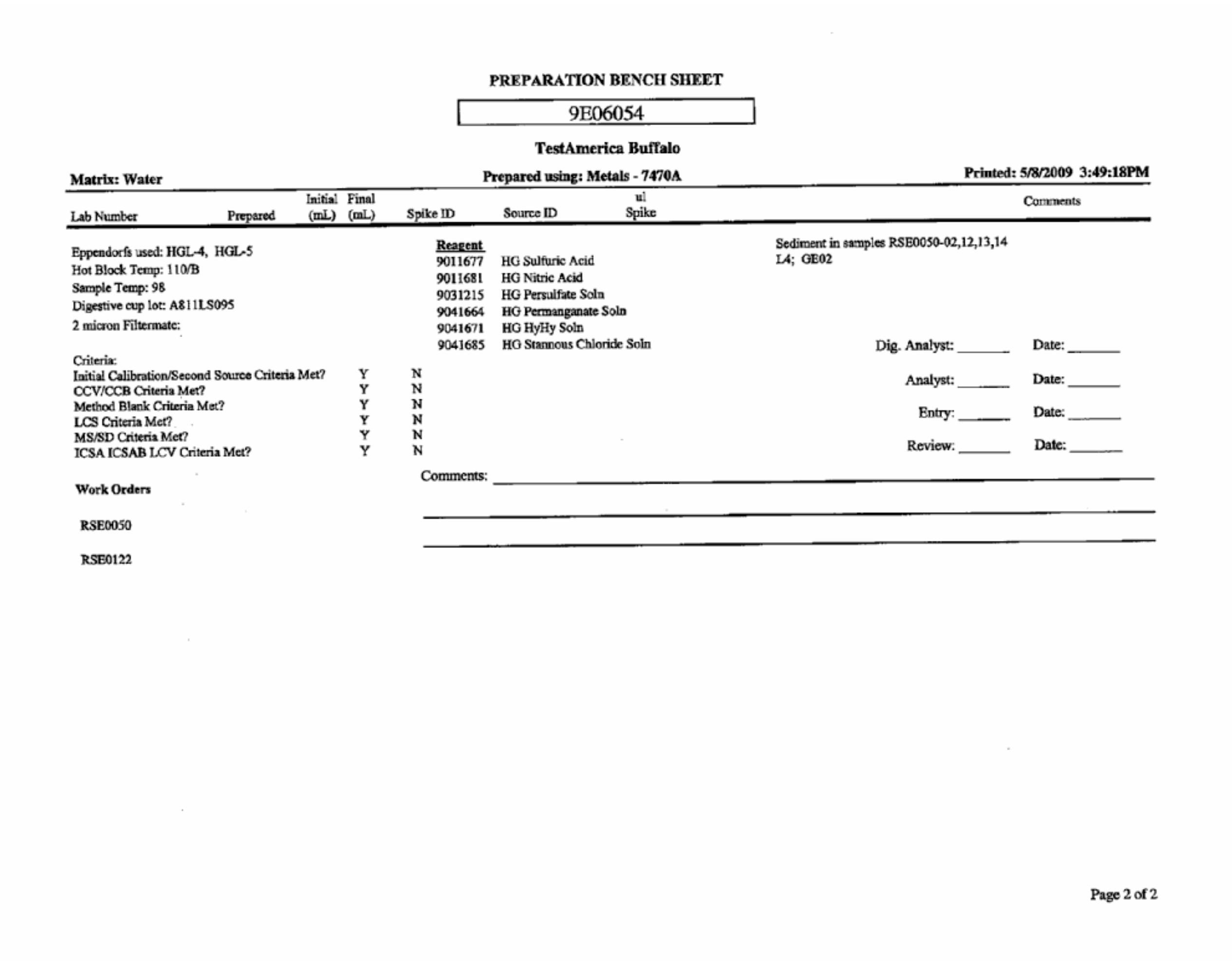
9E06054

TestAmerica Buffalo

Matrix: Water **Prepared using: Metals - 7470A** **Printed: 5/8/2009 3:49:18PM**

Lab Number	Prepared	Initial (mL)	Final (mL)	Spike ID	Source ID	ul Spike		Comments
9E06054-BLK1	05/06/09 14:15	30	50					
9E06054-BS1	05/06/09 14:15	30	50	8090024		1000		
9E06054-MS1	05/06/09 14:15	30	50	8090024	RSE0050-09	2000		
9E06054-MSD1	05/06/09 14:15	30	50	8090024	RSE0050-09	2000		
RSE0050-01	05/06/09 14:15	30	50	Color: /		Clarity:/	Texture:	
7470 Tot - Mercury								
RSE0050-02	05/06/09 14:15	30	50	Color: /		Clarity:/	Texture:	
7470 Tot - Mercury								
RSE0050-09	05/06/09 14:15	30	50	Color: /		Clarity:/	Texture:	
7470 Tot - Mercury	*245.1 Tot - Mercury*							
RSE0050-12	05/06/09 14:15	30	50	Color: /		Clarity:/	Texture:	
7470 Tot - Mercury								
RSE0050-13	05/06/09 14:15	30	50	Color: /		Clarity:/	Texture:	
7470 Tot - Mercury								
RSE0050-14	05/06/09 14:15	30	50	Color: /		Clarity:/	Texture:	
7470 Tot - Mercury								
RSE0122-04	05/06/09 14:15	30	50	Color: /		Clarity:/	Texture:	A00067,GE02
245.1 Tot - Mercury								
RSE0122-05	05/06/09 14:15	30	50	Color: /		Clarity:/	Texture:	A00067,GE02
245.1 Tot - Mercury								

Page 1 of 2



PREPARATION BENCH SHEET

9E06054

TestAmerica Buffalo

Matrix: Water **Prepared using: Metals - 7470A** **Printed: 5/8/2009 3:49:18PM**

Lab Number	Prepared	Initial (mL)	Final (mL)	Spike ID	Source ID	ul Spike	Comments

Eppendorfs used: HGL-4, HGL-5
Hot Block Temp: 110/B
Sample Temp: 98
Digestive cup lot: A811LS095
2 micron Filtermate:

Reagent	
9011677	HG Sulfuric Acid
9011681	HG Nitric Acid
9031215	HG Persulfate Soln
9041664	HG Permanganate Soln
9041671	HG HyHy Soln
9041685	HG Stannous Chloride Soln

Sediment in samples RSE0050-02,12,13,14
L4; GE02

Criteria:		
Initial Calibration/Second Source Criteria Met?	Y	N
CCV/CCB Criteria Met?	Y	N
Method Blank Criteria Met?	Y	N
LCS Criteria Met?	Y	N
MS/SD Criteria Met?	Y	N
ICSA ICSAB LCV Criteria Met?	Y	N

Dig. Analyst: _______ Date: _______

Analyst: _______ Date: _______

Entry: _______ Date: _______

Review: _______ Date: _______

Comments: ____________________

Work Orders

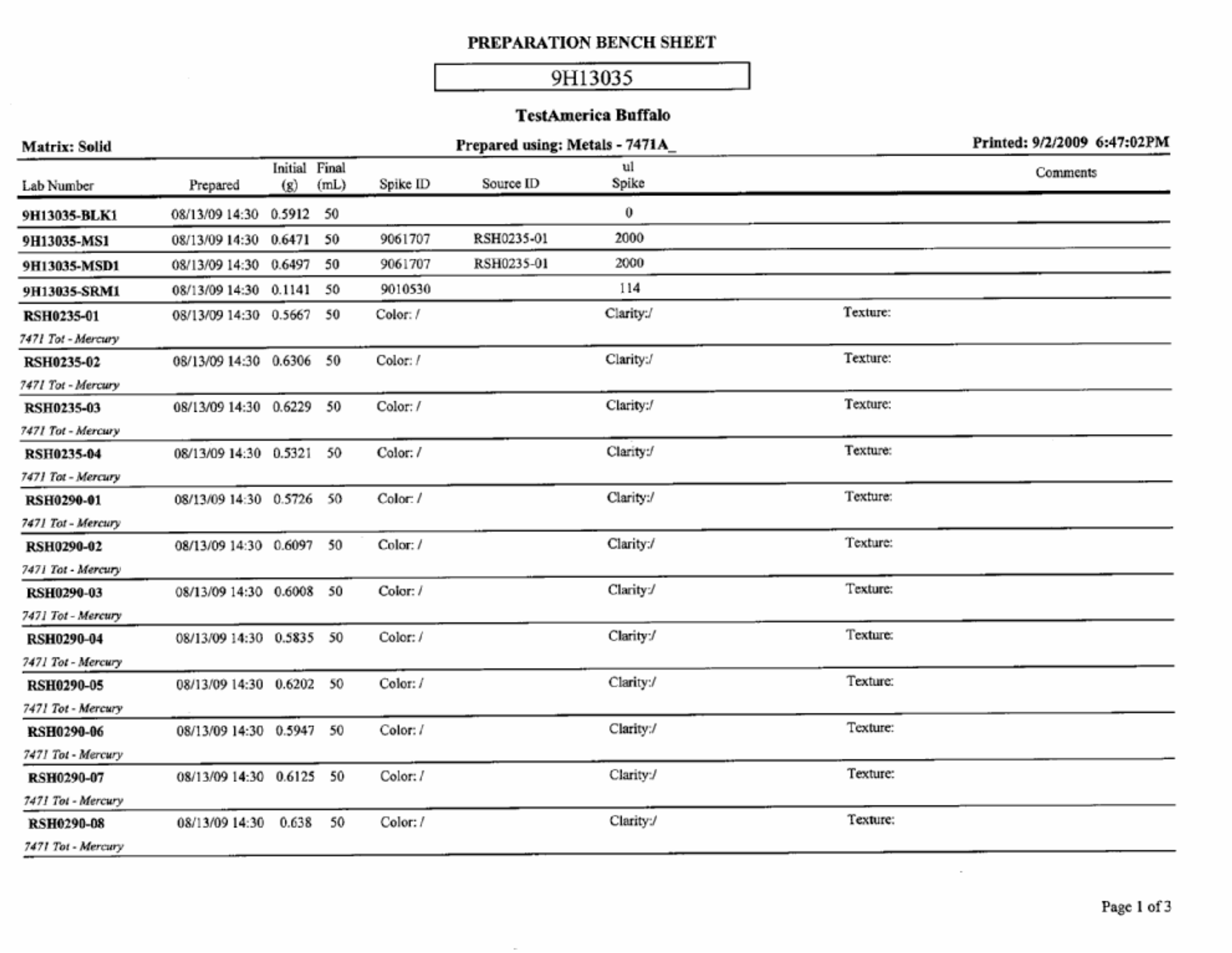
RSE0050

RSE0122

Page 2 of 2

Attachment 3

Sample Soil Digestion Batch Bench Sheet



PREPARATION BENCH SHEET

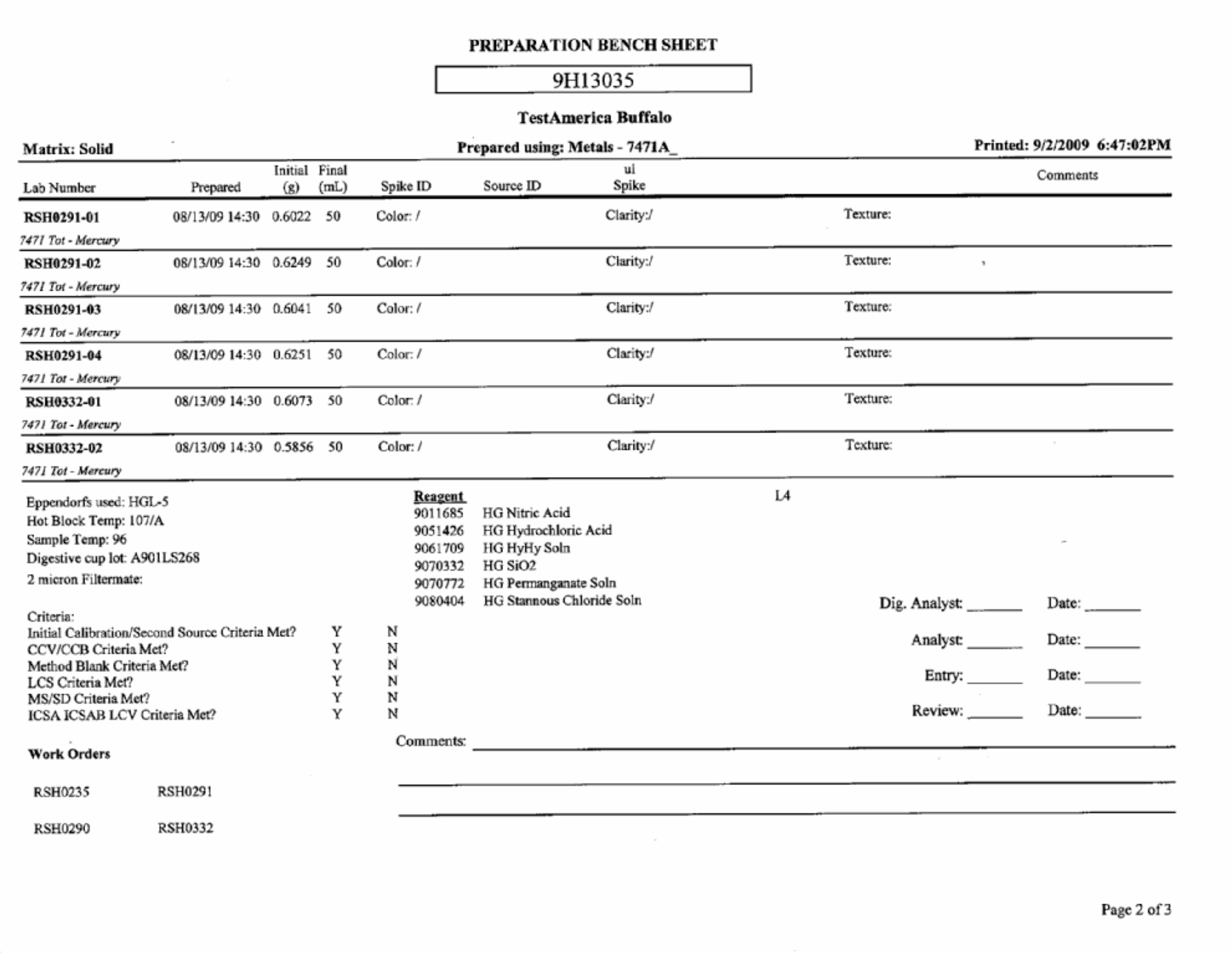
9H13035

TestAmerica Buffalo

Matrix: Solid **Prepared using: Metals - 7471A_** **Printed: 9/2/2009 6:47:02PM**

Lab Number	Prepared	Initial (g)	Final (mL)	Spike ID	Source ID	ul Spike	Comments
9H13035-BLK1	08/13/09 14:30	0.5912	50			0	
9H13035-MS1	08/13/09 14:30	0.6471	50	9061707	RSH0235-01	2000	
9H13035-MSD1	08/13/09 14:30	0.6497	50	9061707	RSH0235-01	2000	
9H13035-SRM1	08/13/09 14:30	0.1141	50	9010530		114	
RSH0235-01 *7471 Tot - Mercury*	08/13/09 14:30	0.5667	50	Color: /		Clarity:/	Texture:
RSH0235-02 *7471 Tot - Mercury*	08/13/09 14:30	0.6306	50	Color: /		Clarity:/	Texture:
RSH0235-03 *7471 Tot - Mercury*	08/13/09 14:30	0.6229	50	Color: /		Clarity:/	Texture:
RSH0235-04 *7471 Tot - Mercury*	08/13/09 14:30	0.5321	50	Color: /		Clarity:/	Texture:
RSH0290-01 *7471 Tot - Mercury*	08/13/09 14:30	0.5726	50	Color: /		Clarity:/	Texture:
RSH0290-02 *7471 Tot - Mercury*	08/13/09 14:30	0.6097	50	Color: /		Clarity:/	Texture:
RSH0290-03 *7471 Tot - Mercury*	08/13/09 14:30	0.6008	50	Color: /		Clarity:/	Texture:
RSH0290-04 *7471 Tot - Mercury*	08/13/09 14:30	0.5835	50	Color: /		Clarity:/	Texture:
RSH0290-05 *7471 Tot - Mercury*	08/13/09 14:30	0.6202	50	Color: /		Clarity:/	Texture:
RSH0290-06 *7471 Tot - Mercury*	08/13/09 14:30	0.5947	50	Color: /		Clarity:/	Texture:
RSH0290-07 *7471 Tot - Mercury*	08/13/09 14:30	0.6125	50	Color: /		Clarity:/	Texture:
RSH0290-08 *7471 Tot - Mercury*	08/13/09 14:30	0.638	50	Color: /		Clarity:/	Texture:

Page 1 of 3



PREPARATION BENCH SHEET

9H13035

TestAmerica Buffalo

Matrix: Solid **Prepared using: Metals - 7471A_** **Printed: 9/2/2009 6:47:02PM**

Lab Number	Prepared	Initial (g)	Final (mL)	Spike ID	Source ID	ul Spike	Comments
RSH0291-01 *7471 Tot - Mercury*	08/13/09 14:30	0.6022	50	Color: /		Clarity:/	Texture:
RSH0291-02 *7471 Tot - Mercury*	08/13/09 14:30	0.6249	50	Color: /		Clarity:/	Texture:
RSH0291-03 *7471 Tot - Mercury*	08/13/09 14:30	0.6041	50	Color: /		Clarity:/	Texture:
RSH0291-04 *7471 Tot - Mercury*	08/13/09 14:30	0.6251	50	Color: /		Clarity:/	Texture:
RSH0332-01 *7471 Tot - Mercury*	08/13/09 14:30	0.6073	50	Color: /		Clarity:/	Texture:
RSH0332-02 *7471 Tot - Mercury*	08/13/09 14:30	0.5856	50	Color: /		Clarity:/	Texture:

Eppendorfs used: HGL-5
Hot Block Temp: 107/A
Sample Temp: 96
Digestive cup lot: A901LS268
2 micron Filtermate:

Reagent	
9011685	HG Nitric Acid
9051426	HG Hydrochloric Acid
9061709	HG HyHy Soln
9070332	HG SiO2
9070772	HG Permanganate Soln
9080404	HG Stannous Chloride Soln

L4

Criteria:

Initial Calibration/Second Source Criteria Met?	Y	N
CCV/CCB Criteria Met?	Y	N
Method Blank Criteria Met?	Y	N
LCS Criteria Met?	Y	N
MS/SD Criteria Met?	Y	N
ICSA ICSAB LCV Criteria Met?	Y	N

Dig. Analyst: _______ Date: _______
Analyst: _______ Date: _______
Entry: _______ Date: _______
Review: _______ Date: _______

Comments: ____________________

Work Orders

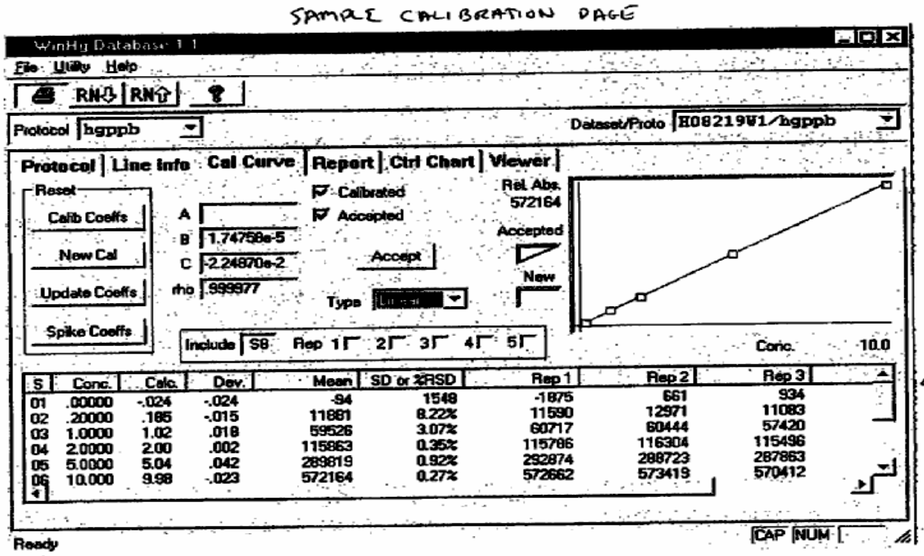
RSH0235	RSH0291
RSH0290	RSH0332

Page 2 of 3

Attachment 4

Example Instrument Calibration Page

SAMPLE CALIBRATION PAGE



Protocol: hgppb

Dataset/Proto: H08219W1/hgppb

A:
B: 1.74758e-5
C: -2.24870e-2
rho: .999977

☑ Calibrated
☑ Accepted

Type: Linear

Rel. Abs. 572164

Include: S8 Rep 1☐ 2☐ 3☐ 4☐ 5☐

S	Conc.	Calc.	Dev.	Mean	SD or %RSD	Rep 1	Rep 2	Rep 3
01	.00000	-.024	-.024	-94	1548	-1875	661	934
02	.20000	.185	-.015	11881	8.22%	11590	12971	11083
03	1.0000	1.02	.018	59526	3.07%	60717	60444	57420
04	2.0000	2.00	.002	115863	0.35%	115786	116304	115496
05	5.0000	5.04	.042	289819	0.92%	292874	288723	287863
06	10.000	9.98	-.023	572164	0.27%	572662	573419	570412

8-21-2009

L2

9-219-K
-E
-F
-G
-H
-I

9061710
-11
-12
-13
-14
-15

Cal: H08219C1
Run: H08219W1

Batches: 9H20039
9H20040
9H20041

Data Review: 8/21/09
2nd Review: 8/24/09 UMH

Reactivity
(Method Sect. 7.3)

Once printed, this is considered an uncontrolled document

Approvals (Signature/Date):

Lisa R Matecki 01/27/10
Lisa R. Matecki Date
Department Manager

Kenneth E. Kasperek 01/27/10
Kenneth Kasperek Date
Health & Safety Manager / Coordinator

Peggy Gray-Erdmann 01/27/10
Peggy Gray-Erdmann Date
Quality Assurance Manager

01/27/10
Chris Spencer Date
Laboratory Director

Facility Distribution No. ___________ **Distributed To:**________________________

1.0 Scope and Application

1.1 Analytes, Matrix(s), and Reporting Limits

1.1.1 This method is taken from EPA solid waste methods; Section 7.3, 9012, 9014 and 9034. On occasion clients may request modifications to this SOP. These modifications are handled following the procedures outlined in the Quality Assurance Manual.

1.1.2 This method is applicable to both aqueous and solid wastes

1.1.3 The reporting limit is 10.0 mg/kg.

1.1.4 This method is applicable to all wastes, with the condition that wastes that are combined with acids do not form explosive wastes.

1.1.5 This method provides a way to determine the specific rates of release of hydrocyanic acid and hydrogen sulfide upon contact with a weak aqueous acid.

1.1.6 This test measures only the hydrocyanic acid and hydrogen sulfide evolved using the specific test conditions. It is not intended to measure forms of cyanide or sulfide other than those evolved under the test conditions.

2.0 Summary of Method

An aliquot of acid is added to a fixed weight of waste in a closed system. The generated gas is swept into a scrubber. The concentrations of hydrocyanic acid and hydrogen sulfide are determined by titration.

3.0 Definitions

Standard definitions can be found in the TestAmerica Laboratory Quality Manual.

4.0 Interferences

Interferences are undetermined.

5.0 Safety

Employees must abide by the policies and procedures in the Corporate Safety Manual, Radiation Safety Manual and this document.

This procedure may involve hazardous material, operations and equipment. This SOP does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of the method to follow appropriate safety, waste disposal and health practices under the assumption that all samples and reagents are potentially hazardous. Safety glasses, gloves, lab coats and closed-toe, nonabsorbent shoes are a minimum.

5.1 Specific Safety Concerns or Requirements

5.1.1 Potassium Cyanide will form Hydrogen Cyanide (HCN) gas when combined with strong acids. Breathing HCN gas may result in death.

5.1.2 Sodium Sulfide will form Hydrogen Sulfide (HS) gas if combined with strong acids. Inhalation of HS gas may be fatal.

5.2 Primary Materials Used

The following is a list of the materials used in this method, which have a serious or significant hazard rating. **NOTE: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the MSDS for each of the materials listed in the table.** A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the MSDS for each material before using it for the first time or when there are major changes to the MSDS.

Material (1)	**Hazards**	**Exposure Limit (2)**	**Signs and symptoms of exposure**
Acetone	Flammable	1000 ppm-TWA	Inhalation of vapors irritates the respiratory tract. May cause coughing, dizziness, dullness, and headache.
Potassium Cyanide	Poison Corrosive	5 Mg/M3 TWA as CN	**This material will form Hydrogen Cyanide (HCN) gas when combined with strong acids. Breathing HCN gas may result in death.** Corrosive to the respiratory tract. May cause headache, weakness, dizziness, labored breathing nausea and vomiting, which can be followed by weak and irregular heart beat, unconsciousness, convulsions, coma and death. Solutions are corrosive to the skin and eyes, and may cause deep ulcers, which heal slowly. May be absorbed through the skin, with symptoms similar to those noted for inhalation. Symptoms may include redness, pain, blurred vision, and eye damage.
Sulfuric Acid	Corrosive Oxidizer Dehydrator	1 mg/m^3	This material will cause burns if comes into contact with the skin or eyes. Inhalation of vapors will cause irritation of the nasal and respiratory system.

Sodium Hydroxide	Corrosive	2 Mg/M3-Ceiling	Severe irritant. Effects from inhalation of dust or mist vary from mild irritation to serious damage of the upper respiratory tract, depending on severity of exposure. Symptoms may include sneezing, sore throat or runny nose. Contact with skin can cause irritation or severe burns and scarring with greater exposures. Causes irritation of eyes, and with greater exposures it can cause burns that may result in permanent impairment of vision, even blindness.
Sodium Sulfide	Corrosive	10 ppm-TWA 15 ppm-STEL	Will form Hydrogen Sulfide gas if combined with strong acids. Inhalation of HS gas may be fatal. Symptoms include painful conjunctivitis, headache, nausea, dizziness, coughing and, in extreme cases, pulmonary edema and possible death. Irritant. Contact with skin can produce serious caustic burns with painful inflammation and possible destruction of tissue. Inflammation, tearing and pain may be expected. Severe contact can cause destruction of tissue.
Sodium Hydroxide	Corrosive	2 Mg/M3-Ceiling	Severe irritant. Effects from inhalation of dust or mist vary from mild irritation to serious damage of the upper respiratory tract, depending on severity of exposure. Symptoms may include sneezing, sore throat or runny nose. Contact with skin can cause irritation or severe burns and scarring with greater exposures. Causes irritation of eyes, and with greater exposures it can cause burns that may result in permanent impairment of vision, even blindness.
1 – Always add acid to water to prevent violent reactions.			
2 – Exposure limit refers to the OSHA regulatory exposure limit.			

6.0 Equipment and Supplies

6.1 Supplies

6.1.1 3-neck round bottom flasks (500ml capacity)

6.1.2 Ring stands

6.1.3 Stir plates

6.1.4 500 ml capacity scrubbers

6.1.5 Stir bars (approx. 1" length)

6.1.6 Flexible Tygon tubing for connections

6.1.7 Nitrogen gas source with flow meter

6.1.8 Copper tubing

6.1.9 Addition funnels (250 ml capacity)

6.1.10 Rubber stoppers for 3-neck flask

6.1.11 500 ml capacity jars

6.1.12 Fume hood

6.1.13 Analytical Balance

6.1.14 Buret

7.0 Reagents and Standards

7.1 Sulfuric Acid (0.01N), H_2SO_4: Add 70ul concentrated H_2SO_4 into the 250ml addition funnels of reagent water.

7.2 Sodium Hydroxide Solution (0.25N), NaOH: Purchased pre-made from Ricca.

7.3 Cyanide Reference Solution, (1000mg/L): 1000mg/L Free Cyanide standard purchased pre-made.

7.4 Sulfide Reference Solution, (570mg/L): Dissolve 4.02g of Na_2S $9H_2O$ in approximately 900ml of reagent water and dilute to 1 liter.

7.5 Standard Iodine Solution, (0.0250N): Dissolve 3.16g Iodine and 23.0g KI in approximately 900ml reagent water and dilute to 1 liter.

7.6 Starch Indicator: Dissolve 10.0g of soluble starch in 1 liter hot reagent water. Solution should be boiled until all of the starch is dissolved.

7.7 Sodium Thiosulfate Titrant (0.025N): Dissolve 6.21g sodium thiosulfate ($Na_2S_2O_3$ $5H_2O$)

and 0.4g sodium hydroxide (NaOH) into 1 liter of distilled water. This titrant must be standardized before use as described in 13.2 and again monthly.

7.8 1:1 HCL: 500ml HCL into 1 liter of reagent water.

7.9 Sodium Phosphate Buffer: 138g Sodium Phosphate Monobasic – Monohydrate into 1L DiH2O. This solution should be made every three months. Refrigerate and store in a dark bottle.

7.10 Pryidine-Barbituric Acid: Place 15g of Barbituric acid into a 250ml volumetric flask, add just enough reagent water to wash the sides of the flask and wet the barbituric acid. Add 75mls of pyridine and mix. Add 15mls of concentrated HCL, mix, and cool to room temp. Dilute to 250mls with DiH2O and mix. This solution should be made monthly. Refrigerate and store in a dark bottle.

7.11 Chloramine-T solution: Dissolve 1.0g Cloramine-T into 100mls of DiH2O. This solution should be made monthly. Refrigerate and store in a dark bottle.

8.0 Sample Collection, Preservation, Shipment and Storage

Sample container, preservation techniques and holding times may vary and are dependent on sample matrix, method of choice, regulatory compliance, and/or specific contract or client requests. Listed below are the holding times and the references that include preservation requirements.

Matrix	Sample Container	Min. Sample Size	Preservation	Holding Time[1]	Reference
Waters	HDPE	50 mLs	NaOH, pH>12; Cool 4 $\underline{+}$ 2°C	180 Days	SW846 3rd Edition, Sect 7.3
Soils	Glass	3 grams	Cool 4 $\underline{+}$ 2°C	180 Days	N/A

[1] Inclusive of digestion and analysis.

9.0 Quality Control

9.1 **Sample QC** - The following quality control samples are prepared with each batch of samples.

Quality Controls	Frequency	Control Limit
Method Blank (MB)	Digested every 20 or fewer samples, analyzed every 10 samples or fewer	< Rpt. Limit
Laboratory Control Sample (LCS)	Digested every 20 or fewer samples, analyzed every 10 samples or fewer	Statistical Limits [3]
Sample Matrix Duplicate (MD)	Digested every 20 or fewer samples, analyzed every 20 samples or fewer	Statistical Limits [3]

[1]The sample selection for MS/MSD is randomly selected, unless specifically requested by a client.

[2] Analytical and QC samples (MB, LCS, MS/MSD)
[3] Statistical control limits are updated annually and are updated into LIMS.

9.2 Instrument QC

9.2.1 Laboratory Control Standard (LCS): Prepare separate calibration standards using 10.0 ml of the 1000 mg/L cyanide reference solution (7.3) and 10.0 ml of 570-ppm sulfide reference solution (7.4). This must be carried through the entire distillation procedure at one per twenty samples. Begin the analytical procedure by analyzing one LCS. Obtained values must be 10-100% of the true value for HCN and 10-100% of the true value for H2S. LCS samples are digested at a frequency of once per twenty samples or fewer and analyzed once per ten samples or fewer.

9.2.2 Method Blank (MBLK): To determine freedom from contamination, prepare a method blank. This must be carried through the entire distillation procedure at one per twenty samples. Begin the analytical procedure by analyzing one method blank along with the LCS. The blank consists of 10.0 ml reagent water that gets the same treatment as the samples and standards. The method blank must exhibit values less than the reporting limit.MBLK samples are digested at a frequency of once per twenty samples or fewer and analyzed once per ten samples or fewer.

9.2.3 Matrix Duplicate (MD): A sample duplicate must be digested and analyzed once in every group of twenty or fewer samples. The RPD between duplicate analyses should be less than 20%.

10.0 Procedure

10.1 Sample Preparation

10.1.1 Add 500 ml 0.25N sodium hydroxide to a calibrated scrubber.

10.1.2 Assemble the system and adjust the flow rate of nitrogen to 60 ml/min.

10.1.3 Add 10.0 grams of the waste to be tested to the round bottom 3-neck flask.

10.1.4 Add approximately 250 ml 0.01N sulfuric acid to the dropping funnel.

10.1.5 With the nitrogen flowing, begin stirring with a magnetic stir bar on a stir plate. Begin the drop wise addition of sulfuric acid from the dropping funnel. NOTE: The stirring speed should remain constant and should not be fast enough to create a vortex.

10.1.6 After 30 minutes, close off the nitrogen and disconnect the scrubber. Determine the amount of reactive cyanide and sulfide in the scrubber as described below.

10.2 Sample Analysis

10.2.1 Cyanide determination (Method 335.4)

10.2.1.1 Refer to BF-WC-015 Total Cyanide Methods, Section 10.12 Automated Analysis System Start-Up Procedure

10.2.1.2 If the concentration of cyanide exceeds the calibration curve for any sample, a dilution is required. A bench dilution may be prepared by pipetting an appropriate volume of distilled sample into a flask and dilution with 0.25 N NaOH. Repeat 10.2.1.1 for the colorimetric determination.

10.2.2 Sulfide determination (Method 376.1)

10.2.2.1 Into a 500ml flask place 1 ml of standard iodine solution. Bring the volume up to approximately 20 ml with reagent water.

10.2.2.2 Add 5ml 6N HCl to the flask.

10.2.2.3 Pipette 100 ml of the scrubber solution into the flask, keeping the tip of the pipette below the surface of the iodine solution.

10.2.2.4 If the iodine color remains, record the amount used up to this point. If the yellow color disappears, add more iodine solution in 1 ml increments until the color remains. Record the total amount of iodine solution used.

10.2.2.5 Add enough starch indicator for the solution to turn dark blue and titrate with 0.025N sodium thiosulfate until the blue disappears. Record the volume of titrant used.

10.2.2.6 Final results should be reported as Total Releasable H_2S (mg/kg). Calculate as shown in 11.2.

11.0 Calculations / Data Reduction

11.1 **Cyanide** (mg/kg): $$\frac{(\text{ml } AgNO_3 \text{ used for sample} - \text{ml } AgNO_3 \text{ used for blank})(\text{N } AgNO_3)(52.04)(2{,}000)}{\text{sample weight (grams)}}$$

11.2 **Sulfide** (mg/kg): $$\frac{[(\text{ml I2})(\text{N I2})] - [(\text{ml titrant})(\text{N titrant})]\ (16{,}030)}{200 \text{ mls}} * \frac{500\text{mls}}{\text{grams of sample}}$$

11.3 **LCS % Recovery** = $$\frac{\text{observed concentration}}{\text{known concentration}} \times 100$$

11.4 **Precision (RPD)** = $$\frac{|\text{orig. sample value} - \text{dup. sample value}|}{[(\text{orig. sample value} + \text{dup. sample value})/2]} \times 100$$

NOTE: All dry weight corrections are made in LIMS at the time the final report is prepared.

12.0 Method Performance

12.1 Method Detection Limit Study (MDL)

Method Detection Limit: A valid method detection limit for each analyte of interest must be generated. The MDL must be below the reporting limit for each analyte. The procedure for determination of the method detection limit is given in 40 CFR Part 136, Appendix B. Current TestAmerica Buffalo MDLs are maintained the QA department and are easily viewed in the laboratory LIMs system.

12.2 Demonstration of Capabilities

A one–time initial demonstration of performance for each individual method for both soils and water matrices must be generated.

12.2.1 This requires quadruplicate analysis of a mid–level check standard containing all of the standard analytes for the method using the same procedures used to analyze samples, including sample preparation.

12.2.2 Calculate the average recovery and standard deviation of the recovery for each analyte of interest.

12.2.3 Compare these results with the acceptance criteria given in the Method or to laboratory historical limits (if available).

12.2.4 Repeat the test for any analyte that does not meet the acceptance criteria. Repeated failure for any analyte indicates the need for the laboratory to evaluate the analytical procedure and take corrective action.

12.3 Training Requirements

12.3.1 The supervisor has the responsibility to ensure that this procedure is performed by an analyst who has been properly trained in its use and has the required experience.

12.3.2 The following analyst validation information is maintained for this method in the laboratory QA files.

12.3.2.1 The analyst must complete the laboratory safety orientation training that includes, but is not limited to, chemicals, PPE requirements, and electrical safety.

12.3.2.2 The analyst must read and understand this SOP.

12.3.2.3 The analyst must read and understand the Method used as reference for this SOP

12.3.2.4 The analyst must complete a DOC or successfully analyze PT samples annually.

12.3.2.5 The analyst must complete the STL Quality Assurance Training.

13.0 Pollution Control

All waste will be disposed of in accordance with Federal, State and Local regulations. Where reasonably feasible, technological changes have been implemented to minimize the potential for pollution of the environment. Employees will abide by this method and the policies in section 13 of the Corporate Safety Manual for “Waste Management and Pollution Prevention.”

14.0 Waste Management

The following waste streams are produced when this method is carried out.

- Acidic sample waste generated by sample digestion and disposed of in the “A” waste containers.
- Alkaline sample waste remaining in scrubbers is disposed of in the “D” waste containers.

15.0 References / Cross-References

EPA Test Methods for Evaluation Solid Waste, Physical/Chemical Methods (SW-846), Third Edition, Update III, December 1996; Section 7.3, 9012, 9014 and 9034.

16.0 Method Modifications:

Item	Method	Modification
N/A	Sect 7.3	N/A

17.0 Attachments

17.1 Distillation Sequence
17.2 Analytical Sequence
17.3 Analytical Batch for Reactive CN
17.4 Analytical Batch for Reactive Sulfide
17.5 Wet Chemistry Batch Summary

18.0 Revision History

- Revision 0, dated 18 February 2008
    - Integration for TestAmerica operations.
    - Department Manager, QA Manager change, signature updated
    - Updated attachments 17.2-17.6

- Revision 1, January 27, 2010
    - Updated attachments
    - Section 10.2.1- updated CN method from 335.2 to 335.4.

Attachment 17.1

Distillation Sequence

LCS – 1000 ppm CN and 570 ppm H_2S
MBLK
Sample
Sample
Sample
Sample
Sample
Sample
Sample
Sample
Sample
Sample
Sample
Sample
Sample
Sample
Sample
Sample
Sample
Sample
Sample
Sample Matrix Duplicate
LCS - 1000 ppm CN and 570 ppm H_2S
MBLK

Attachment 17.2
Analytical Sequence

LCS – 1000 ppm CN
MBLK
Sample
Sample
Sample
Sample
Sample
Sample
Sample
Sample
Sample
Sample Matrix Duplicate
LCS – 1000 ppm CN
MBLK

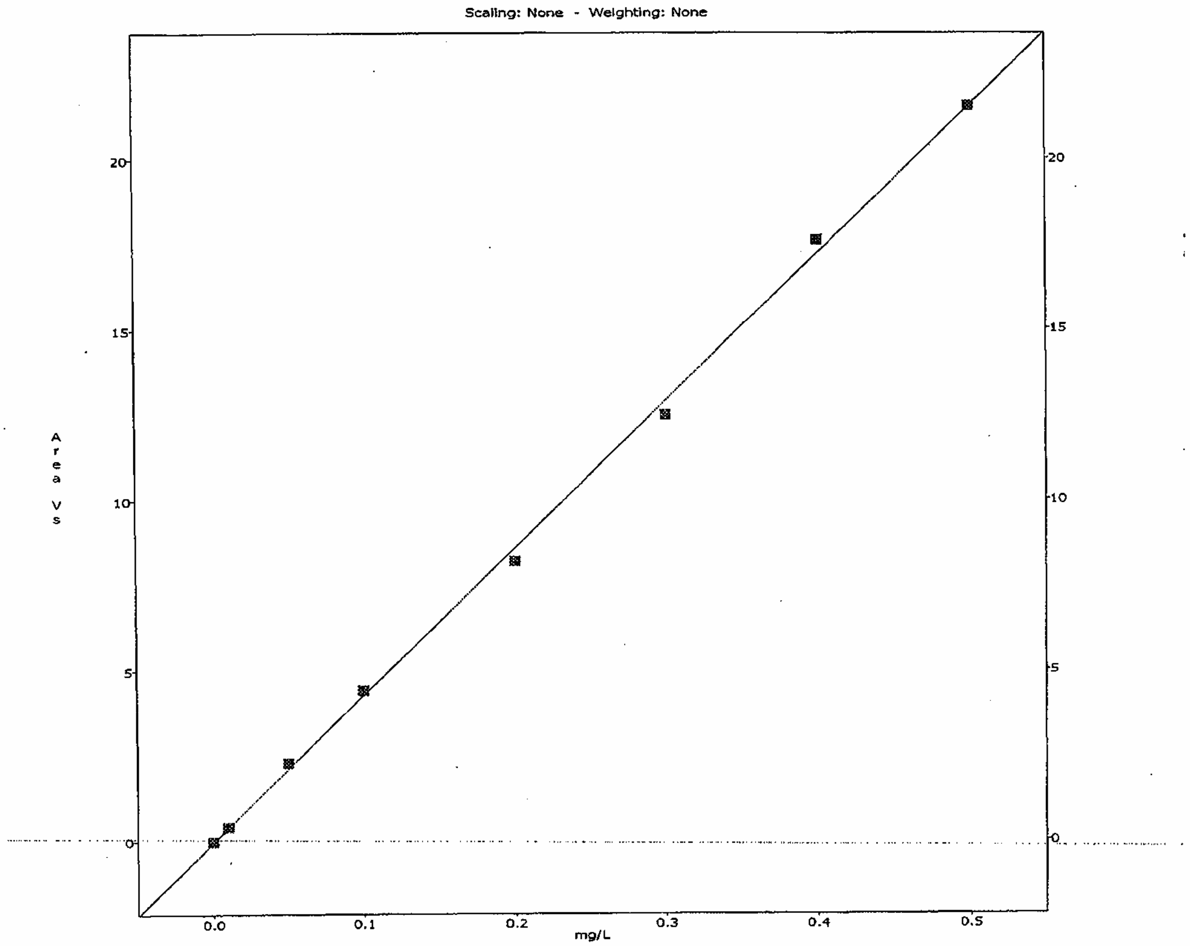
LCS – 570 ppm H2S
MBLK
Sample
Sample
Sample
Sample
Sample
Sample
Sample
Sample
Sample
Sample Matrix Duplicate
LCS – 570 ppm H2S
MBLK

Attachment 17.3
Analytical Batch for Reactive CN

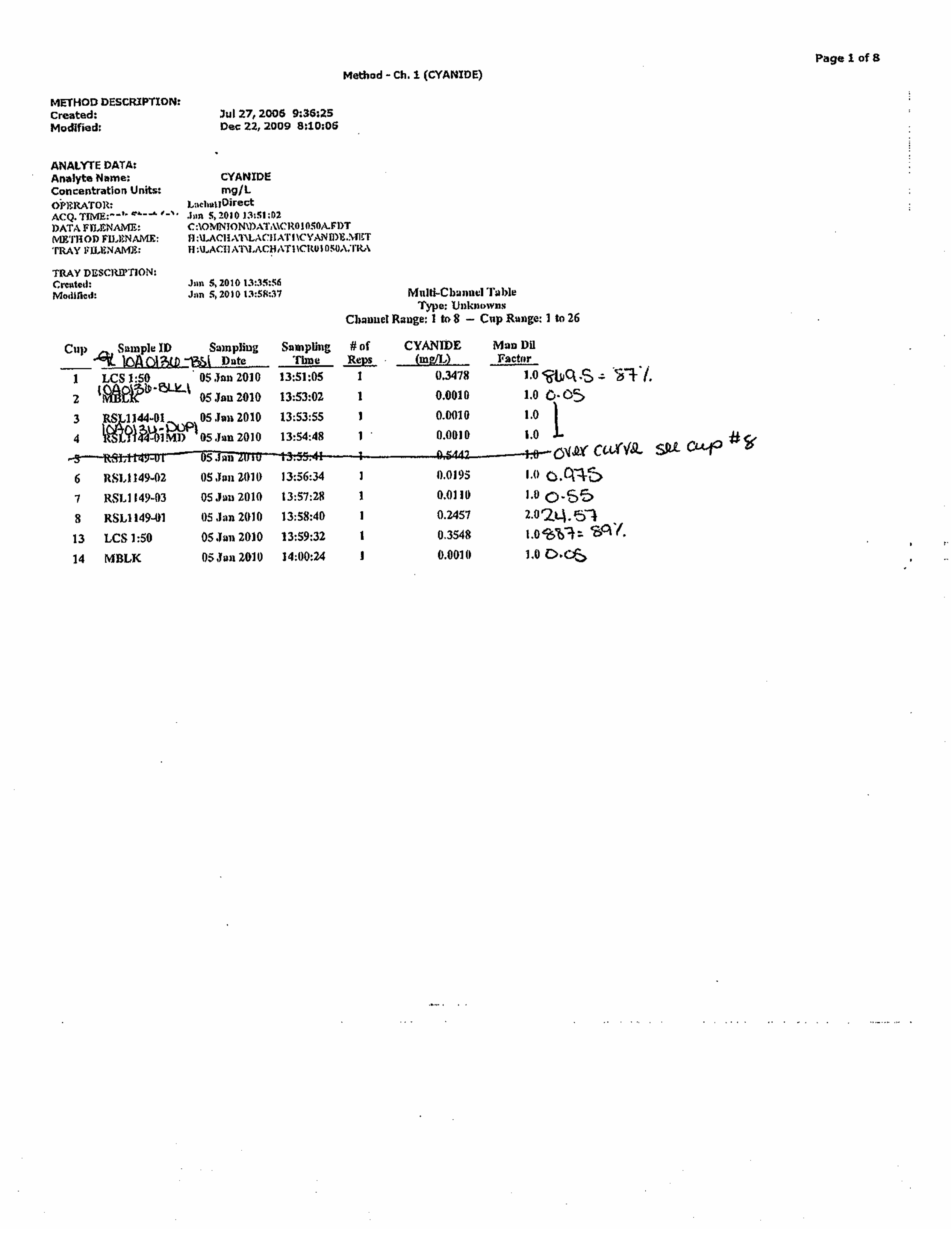
CYANIDE

Lvl	Area	mg/L	Rep 1	Rep 2	Rep 3	Rep 4	Rep 5	Replic STD	Replic % RSD	Residual 1st Poly
1	21544614	0.50	21544614					0.0	0.0	-0.1
2	17613428	0.40	17613428					0.0	0.0	-2.3
3	12490381	0.30	12490381					0.0	0.0	3.2
4	8208512	0.20	8208512					0.0	0.0	4.4
5	4419367	0.10	4419367					0.0	0.0	-3.4
6	2278707	0.05	2278707					0.0	0.0	-7.6
7	418573	0.01	418573					0.0	0.0	-6.9
8	0	0.00	0					0.0	0.0	
9		0.00						0.0	0.0	

1st Order Poly
Conc = 2.317e-008 Area + 9.921e-004
r = 0.9994



Printed: Tuesday, January 05, 2010 - 02:02 PM



Method - Ch. 1 (CYANIDE)

METHOD DESCRIPTION:
Created: Jul 27, 2006 9:36:25
Modified: Dec 22, 2009 8:10:06

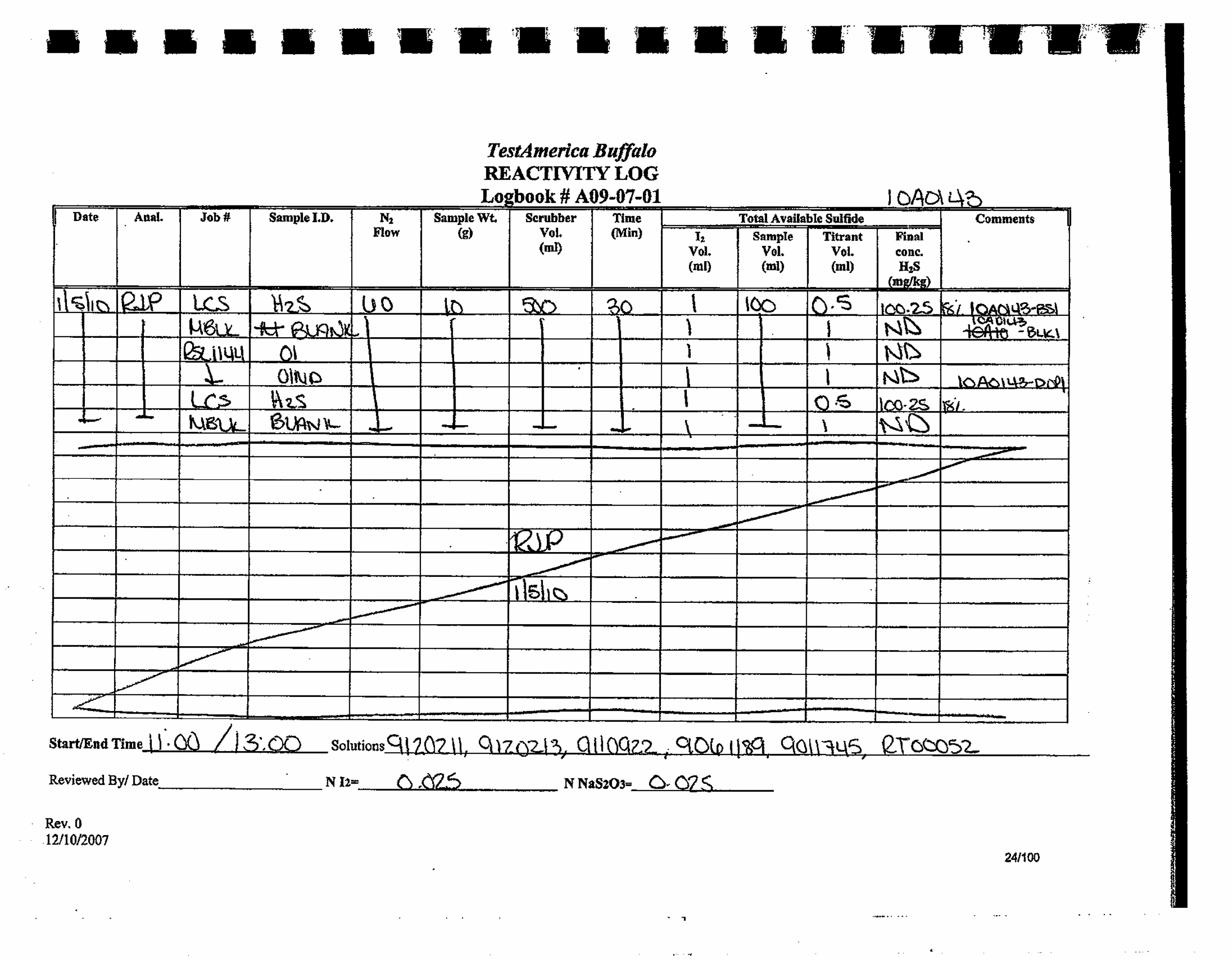
ANALYTE DATA:
Analyte Name: CYANIDE
Concentration Units: mg/L
OPERATOR: LachatDirect
ACQ. TIME: Jan 5, 2010 13:51:02
DATA FILENAME: C:\OMNION\DATA\CR01050A.FDT
METHOD FILENAME: H:\LACHAT\LACHAT1\CYANIDE.MET
TRAY FILENAME: H:\LACHAT\LACHAT1\CR01050A.TRA

TRAY DESCRIPTION:
Created: Jan 5, 2010 13:35:56
Modified: Jan 5, 2010 13:58:37

Multi-Channel Table
Type: Unknowns
Channel Range: 1 to 8 — Cup Range: 1 to 26

Cup	Sample ID	Sampling Date	Sampling Time	# of Reps	CYANIDE (mg/L)	Man Dil Factor	
1	QC 10A0136-BS1 LCS 1:50	05 Jan 2010	13:51:05	1	0.3478	1.0	869.5 = 87%
2	10A0136-BLK1 MBLK	05 Jan 2010	13:53:02	1	0.0010	1.0	0.05
3	RSL1144-01	05 Jan 2010	13:53:55	1	0.0010	1.0	
4	10A0136-DUP1 RSL1144-01MD	05 Jan 2010	13:54:48	1	0.0010	1.0	
~~5~~	~~RSL1149-01~~	~~05 Jan 2010~~	~~13:55:41~~	~~1~~	~~0.5442~~	~~1.0~~	over curve see cup #8
6	RSL1149-02	05 Jan 2010	13:56:34	1	0.0195	1.0	0.975
7	RSL1149-03	05 Jan 2010	13:57:28	1	0.0110	1.0	0.55
8	RSL1149-01	05 Jan 2010	13:58:40	1	0.2457	2.0	24.57
13	LCS 1:50	05 Jan 2010	13:59:32	1	0.3548	1.0	887 = 89%
14	MBLK	05 Jan 2010	14:00:24	1	0.0010	1.0	0.05

Attachment 17.4
Analytical Batch for Reactive Sulfide



TestAmerica Buffalo
REACTIVITY LOG
Logbook # A09-07-01

10A0143

Date	Anal.	Job #	Sample I.D.	N_2 Flow	Sample Wt. (g)	Scrubber Vol. (ml)	Time (Min)	Total Available Sulfide				Comments
								I_2 Vol. (ml)	Sample Vol. (ml)	Titrant Vol. (ml)	Final conc. H_2S (mg/kg)	
1/5/10	RJP	LCS	H2S	40	10	500	30	1	100	0.5	100.25	100% 10A0143-BS1
		MBLK	~~H~~ BLANK					1		1	ND	~~10A10~~ 10A0143 -BLK1
		RSL1144	01					1		1	ND	
		↓	01ND					1		1	ND	10A0143-DUP1
		LCS	H2S					1		0.5	100.25	100%
		MBLK	BLANK					1		1	ND	
						RJP						
						1/5/10						

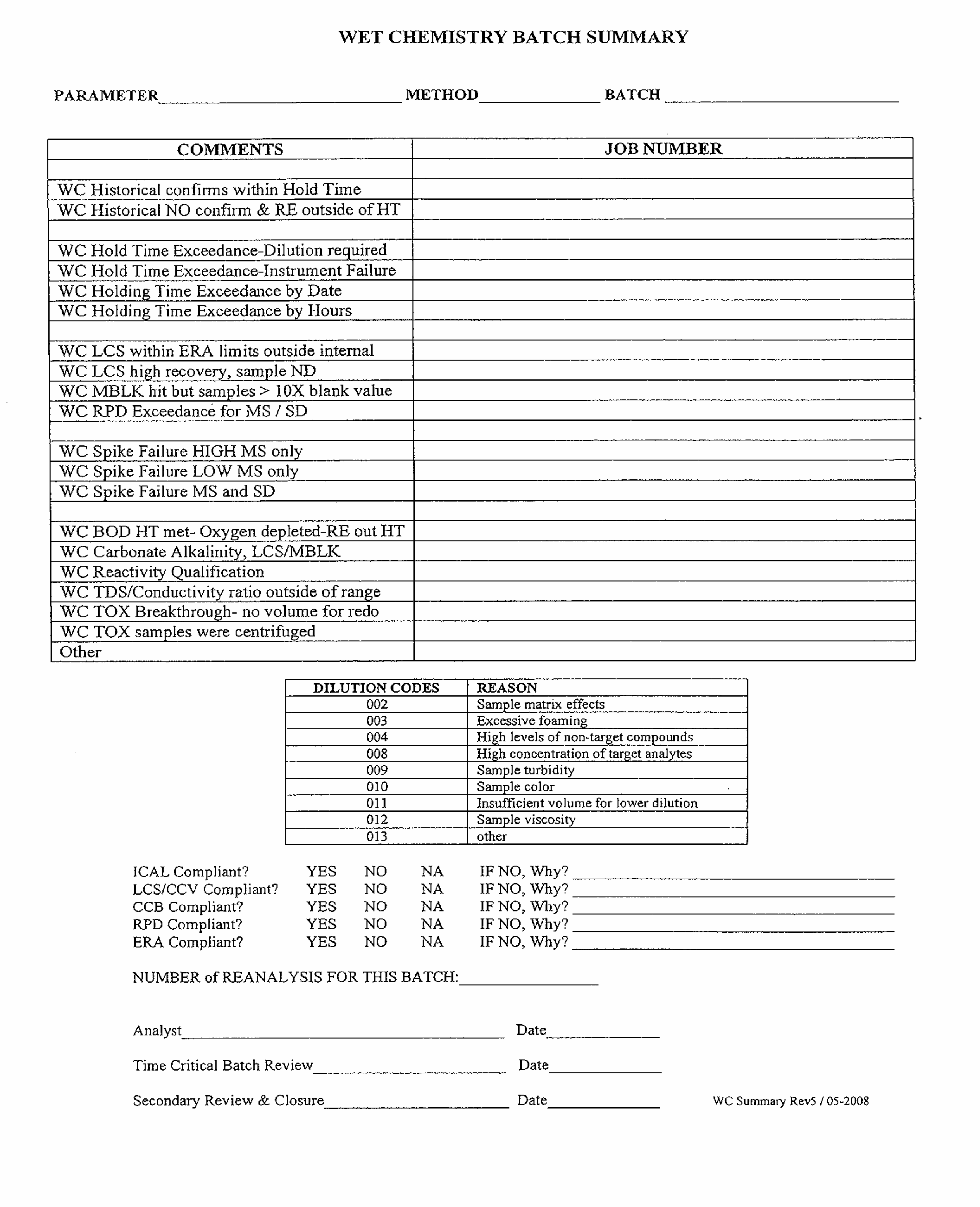
Start/End Time 11:00 / 13:00 Solutions 9120211, 9120213, 9110922, 9061189, 9011745, RT00052

Reviewed By/ Date________ N I_2= 0.025 N NaS_2O_3= 0.025

Rev. 0
12/10/2007

24/100

Attachment 17.5
Wet Chemistry Batch Summary



WET CHEMISTRY BATCH SUMMARY

PARAMETER______________________________ METHOD_______________ BATCH _____________________________

COMMENTS	JOB NUMBER
WC Historical confirms within Hold Time	
WC Historical NO confirm & RE outside of HT	
WC Hold Time Exceedance-Dilution required	
WC Hold Time Exceedance-Instrument Failure	
WC Holding Time Exceedance by Date	
WC Holding Time Exceedance by Hours	
WC LCS within ERA limits outside internal	
WC LCS high recovery, sample ND	
WC MBLK hit but samples > 10X blank value	
WC RPD Exceedance for MS / SD	
WC Spike Failure HIGH MS only	
WC Spike Failure LOW MS only	
WC Spike Failure MS and SD	
WC BOD HT met- Oxygen depleted-RE out HT	
WC Carbonate Alkalinity, LCS/MBLK	
WC Reactivity Qualification	
WC TDS/Conductivity ratio outside of range	
WC TOX Breakthrough- no volume for redo	
WC TOX samples were centrifuged	
Other	

DILUTION CODES	REASON
002	Sample matrix effects
003	Excessive foaming
004	High levels of non-target compounds
008	High concentration of target analytes
009	Sample turbidity
010	Sample color
011	Insufficient volume for lower dilution
012	Sample viscosity
013	other

ICAL Compliant? YES NO NA IF NO, Why? ________________________________
LCS/CCV Compliant? YES NO NA IF NO, Why? ________________________________
CCB Compliant? YES NO NA IF NO, Why? ________________________________
RPD Compliant? YES NO NA IF NO, Why? ________________________________
ERA Compliant? YES NO NA IF NO, Why? ________________________________

NUMBER of REANALYSIS FOR THIS BATCH:_______________

Analyst____________________________________ Date_____________

Time Critical Batch Review_____________________ Date_____________

Secondary Review & Closure____________________ Date_____________ WC Summary Rev5 / 05-2008

Title: METHOD 3050B: ACID DIGESTION OF SEDIMENTS, SLUDGES, AND SOILS

Once printed, this is considered an uncontrolled document

Approvals (Signature/Date):

E. Scott Wagner 02/08/10
E. Scott Wagner Date
Department Manager

Kenneth E. Kasperek 02/08/10
Kenneth Kasperek Date
Health & Safety Manager

Peggy Gray-Erdmann 02/08/10
Peggy Gray-Erdmann Date
Quality Assurance Manager

02/08/10
Chris Spencer Date
Laboratory Director

Facility Distribution No. ____________ **Distributed To:**__________________________

1.0 Scope and Application

This method is an acid digestion procedure used to prepare sediment, oil, waste, sludge, and soil samples for analysis by inductively coupled plasma atomic emission spectrometry (ICP-AES) and inductively coupled plasma-mass spectroscopy (ICP-MS). This method may be digested using The Environmental Express Auto Block or by manually adding reagents and heating on hot blocks.

1.1 Analytes, Matrix(s), and Reporting Limits

Samples prepared by this method may be analyzed for the following metals in soils, sludges, or sediments. However, not all analytes are analyzed by each method.

Aluminum
Antimony
Arsenic
Barium
Beryllium
Boron
Calcium
Cadmium
Chromium
Cobalt
Copper
Iron
Lead
Magnesium
Manganese
Molybdenum
Nickel
Potassium
Silver
Sodium
Thallium
Selenium
Titanium
Vanadium
Zinc
Tin
Lithium

See SOP BF-ME-009 Section 22.3 for soil detection limits for all analytes listed above.

2.0 Summary of Method

A representative sample (0.5 g - wet weight) is digested in Nitric acid and Hydrogen Peroxide. The digestate is then refluxed with Hydrochloric acid as the final reflux. All digestates are diluted to a final volume of 50 ml with laboratory reagent water. A separate sample aliquot shall be dried for a total solids determination.

3.0 Definitions

3.1 Total Metals --The concentration determined on filtered sample following digestion. Note that this method is designed to determine total *environmentally available* metals.

4.0 Interferences

4.1 Sludge samples can contain diverse matrix types, each of which may present an analytical challenge. Spiked samples and any relevant standard reference material are processed in accordance with the quality control requirements to aid in determining whether this method is applicable to a given waste.

4.2 Boron from glassware will leach into the sample solution during and following sample processing. For critical low level determinations of Boron, only quartz and/or plastic labware is used.

4.3 Allowing samples to boil or go dry during digestion may result in the loss of volatile metals. If this occurs, the sample must be re-prepared. Antimony (Sb) is easily lost by volatilization from hydrochloric media.

5.0 Safety

Employees must abide by the policies and procedures in the Corporate Safety Manual, Radiation Safety Manual and this document.

This procedure may involve hazardous material, operations and equipment. This SOP does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of the method to follow appropriate safety, waste disposal and health practices under the assumption that all samples and reagents are potentially hazardous. Safety glasses, gloves, lab coats and closed-toe, nonabsorbent shoes are a minimum.

5.1 Specific Safety Concerns or Requirements

5.1.1 Laboratory coats, gloves and safety glasses shall be worn during all stages of the digestion procedure.

5.1.2 Nitrile gloves shall be used when handling all standards and samples. Safety glasses must be worn at all times. Extra care must be taken when dispensing concentrated acids. Concentrated acids must be dispensed only in the fume hood.

5.1.3 Samples that contain high concentrations of carbonates or organic material or samples that are at elevated pH can react violently when acids are added.

5.1.4 Hydrogen peroxide (H_2O_2) is a strong oxidizer and is corrosive. The digestion must be cooled sufficiently before the addition of H_2O_2 to avoid a reaction and possible violent effervescence, or boiling over of the digestate.

5.1.5 Many of the metallic elements analyzed for in this method are known to be hazardous to health. Care must be taken in the handling and disposing of all standards and samples. See section 20.0 for procedures on the disposal of standard and sample waste.

5.2 Primary Materials Used

The following is a list of the materials used in this method, which have a serious or significant hazard rating. **Note: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the MSDS for each of the materials listed in the table.** A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the MSDS for each material before using it for the first time or when there are major changes to the MSDS.

Material (1)	**Hazards**	**Exposure Limit (2)**	**Signs and symptoms of exposure**
Hydrochloric Acid	Corrosive Poison	5 ppm-Ceiling	Inhalation of vapors can cause coughing, choking, inflammation of the nose, throat, and upper respiratory tract, and in severe cases, pulmonary edema, circulatory failure, and death. Can cause redness, pain, and severe skin burns. Vapors are irritating and may cause damage to the eyes. Contact may cause severe burns and permanent eye damage.
Nitric Acid	Corrosive Oxidizer Poison	2 ppm-TWA 4 ppm-STEL	Nitric acid is extremely hazardous; it is corrosive, reactive, an oxidizer, and a poison. Inhalation of vapors can cause breathing difficulties and lead to pneumonia and pulmonary edema, which may be fatal. Other symptoms may include coughing, choking, and irritation of the nose, throat, and respiratory tract. Can cause redness, pain, and severe skin burns. Concentrated solutions cause deep ulcers and stain skin a yellow or yellow-brown color. Vapors are irritating and may cause damage to the eyes. Contact may cause severe burns and permanent eye damage.
Hydrogen Peroxide	Oxidizer Corrosive	1 ppm-TWA	Vapors are corrosive and irritating to the respiratory tract. Vapors are very corrosive and irritating to the eyes and skin.
1 – Always add acid to water to prevent violent reactions.			
2 – Exposure limit refers to the OSHA regulatory exposure limit.			

6.0 Equipment and Supplies

- *Environmental Express* Auto Block/with PDA and Digestion Blocks
- 50 ml Polypropylene digestion cups with reflux caps from Environmental Express
- Sample racks-polycarbonate
- Filter Mate Teflon press filter
- Membrane disk filters 0.45 um 47mm Pall Life Sciences
- Eppendorf pipettes and pipette tips
- Analytical balance capable of accurate weighing to 0.01 g.
- Drying oven maintained at 105±4°C.
- NIST Certified Thermometer - That covers range of 0-150°C.
- Silicon (IV) Oxide beads
- **Maintenance Supplies for the Autoblock from Environmental Express**:
- The PDA will issue a maintenance indicator message informing analyst to schedule this maintenance:
- HEPA Filter Replacement---every 12 months
- Peristaltic Pump Tube---Flexible tubing-every 12 months
- Reagent Injection Kit---Tubing Assembly from Splitter to Sample Cup,6 lines-every 12 months
- Reagent Uptake Line---25 feet of reagent uptake line for 5 bottle-to-inlet lines-as required

6.1 Instrumentation

Environmental Express Auto-Block w/PDA

7.0 Reagents and Standards

7.1 Reagent grade chemicals are used in all tests. Unless otherwise indicated, it is intended that all reagents confirm the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available. Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

7.2 Laboratory Reagent Water. See SOP No. BF-GP-002

7.3 Trace metal grade concentrated nitric acid (HNO_3) and hydrochloric acid (HCl). The certificate of analysis is directly on the acid bottle. Whenever the purity of the acid is suspected, the acid is analyzed via ICPMS to determine levels of impurities. If impurity concentrations are at such levels that method blanks are <MDL, the acid can be used.

7.4 1:1 HNO_3, is prepared by mixing equal volume of reagent water and concentrated nitric acid. Pour concentrated acid to water; never pour water to concentrated acid.

7.5 30% Hydrogen Peroxide, H_2O_2, un-stabilized 30% Hydrogen Peroxide used if analysis requires Tin.

7.6 Spike standards:

7.6.1 10 µg/ml of Ag is prepared by filling a class "A" volumetric flask half way with laboratory reagent water and adding four mls of Nitric Acid. Pipette 1.0 ml of 1000 µg/ml Ag stock standard to the 100 ml volumetric flask and fill to the line with laboratory reagent water. Spike standard to be verified via ICP-AES prior to use.

7.6.2 40 µg/ml of Sn is prepared by filling a class A volumetric flask half way with laboratory reagent water and adding four mls of Nitric Acid. Pipette 4.0 ml of 1000 µg/ml Sn stock standard to a 100 ml volumetric flask and fill to the line with laboratory reagent water. Spike standard to be verified via ICP-AES prior to use.`

7.6.3 ICP-AES spikes: ICUS-1370, ICUS-574, 10 µg/mL Ag and 40 µg/mL Sn. See Table 1 for details.

7.6.4 ICP-MS spikes: See Table 1.

7.6.5 The SRM sample is purchased from Environmental Resource Associates. The concentration of each analyte is different from lot to lot. The certified concentrations are stored in the binder with the Certificates of Analysis.

8.0 Sample Collection, Preservation, Shipment and Storage

Sample container, preservation techniques and holding times may vary and are dependent on sample matrix, method of choice, regulatory compliance, and/or specific contract or client requests. Listed below are the holding times and the references that include preservation requirements.

Matrix	Sample Container	Min. Sample Size	Preservation	Holding Time [1]	Reference
Waters	poly	300 mls	HNO_3, pH < 2;	180 Days	40 CFR Part 136.3
Soil	Glass	3 grams	Cool 4 $\underline{+}$ 2^oC	180 Days	N/A

[1] Inclusive of digestion and analysis.

9.0 Quality Control

9.1 For each batch of samples (not to exceed 20 samples), a preparation blank (method blank, BLK) is carried throughout the entire sample preparation and analytical process. This blank is useful in monitoring any contamination.

9.2 For each batch of samples (not to exceed 20 samples), a Standard Reference Material (SRM) must be employed to demonstrate proper implementation of the method.

9.3 For each batch of samples (not to exceed 20 samples), a matrix spike (MS) is processed on a routine basis. The MS will be used to determine matrix effects on digestion and detection.

9.4 For each batch of samples (not to exceed 20 samples), a replicate sample is processed on a routine basis. Replicate samples are either method duplicates (DUP) or spike duplicates (MSD) depending on the clients' request, but are usually spike duplicates. Replicate samples will be used to determine precision. DUP is another aliquot of the selected sample. MSD is another MS that is processed through the preparation and analysis procedure.

<u>Sample QC</u> - The following quality control samples are prepared with each batch of samples.

Quality Controls	Frequency	Control Limit
Method Blank (BLK)	1 in 20 or fewer samples	< Rpt. Limit or < 2.2MDL for MCAWW
Standard Reference material (SRM)	1 in 20 or fewer samples	Specified by manufacturer of SRM
Matrix Spike (MS)[1]	1 in 20 or fewer samples	75-125%
MS Duplicate (MSD)[1]	1 in 20 or fewer samples	75-125%
Matrix Duplicate (DUP)[1]	1 in 20 depending on method/client request	20%

[1] The sample selection for MS/MSD/DUP is determined by the extraction lab unless specifically requested by a client.

10.0 Procedure

10.1 Method Blank (BLK): For each digestion batch of 20 samples or less, weigh 0.5 grams of Silicon Oxide to a digestion cup. Record the weight on the batch sheet. The Silicon Oxide is used as a soil substitute. Next, transfer 10 mls of laboratory reagent water to the digestion cup and carry it through the entire analytical process.

10.2 Standard Reference material (SRM): For each digestion batch of 20 samples or less, weigh 0.5 g of LCS to a digestion cup and carry it through the entire digestion process.

10.3 Matrix Spike (MS) and Matrix Spike Duplicate (MSD): For each digestion batch of 20 samples or less, prepare one sample in triplicate and fortify two aliquots with appropriate amount of spikes according to the determination methods. These three samples are ‘Sample’, ‘MS’, and ‘MSD’.

ICP-AES and ICP-MS: Fortify with 0.50 mL ICUS-1370, 0.50 ml ICUS-574, 0.50 mL 10 μg/ml Ag, and 0.50 mL 40 μg/ml Sn spiking solutions. (Table 1)

10.4 Matrix Duplicate: For each digestion batch of 20 samples or less, prepare one sample in triplicate and fortify one of the aliquots with appropriate amount of spikes according to the determination methods described in 10.03. These three samples are treated as ‘Sample’, ’DUP‘ and ‘MS’. A matrix duplicate is not routinely prepared. It is only done on the basis of the clients’ requests.

10.5.0 Sample Digestion via manual method

10.5.1 Mix the sample thoroughly to achieve homogeneity. Weigh out 0.5 grams of sample ±0.05 grams to a 50ml digestion cup.

10.5.2 Weigh out 0.5 grams of SRM ±0.001 grams to a digestion cup.

10.5.3 Spike the Matrix Spike and Spike Duplicate as stated in 10.3

10.5.4 Add 10.0mls of laboratory reagent water to each digestion cup

10.5.5 Add 5.0mls of 1:1 Nitric Acid and heat for 15 minutes with a reflux cap.

10.5.6 Add 2.5mls of concentrated Nitric Acid and heat for 30 minutes with a reflux cap.

10.5.7 Cool for 5 minutes.

10.5.8 Add 3.0 mls of reagent water, wait 1 minute, and add 1.0 ml of Hydrogen Peroxide.

10.5.9 Wait 5 minutes.

10.5.10 Add 1.0ml of Hydrogen Peroxide.

10.5.11 Heat for 8 minutes with a reflux cap.

10.5.12 Cool for 5 minutes.

10.5.13 Add 1.0 ml of Hydrogen Peroxide

10.5.14 Wait 3 minutes

10.5.15 Add 2.0 mls of Hydrogen Peroxide

10.5.16 Wait 3 minutes.

10.5.17 Heat for 8 minutes.

10.5.18 Cool for 5 minutes.

10.5.19 Add 3.0mls of reagent water and 2.5mls of concentrated Hydrochloric Acid.

10.5.20 Heat for 15 minutes with a reflux cap.

10.5.21 Allow sample to cool. Wash down the digestion cup walls and reflux cap with reagent water. Bring the final volume to 50mls with reagent water.

10.5.22 Filter each sample with a filter.

10.6 <u>Sample digestion via Auto-block</u>

10.6.1 Verify that temperature is set to 115° in the temperature control block.

10.6.2 Prepare samples as instructed under the manual method sections 10.5.1 through 10.5.4

10.6.3 Place samples in auto-block. Make sure that the last row is completely filled or reagents will be added to empty block tubes.

10.6.4 Tap the “Select Mode” button in the Manual Mode window.

10.6.5 Tap the “Select Mode” button in the Service Mode window.

10.6.6 Verify that the method selected is 3050_coolingtest.rom

10.6.7 Tap the “Start” button

10.6.8 At the cannot Verify Reagent H2O2 message, tap “OK”

10.6.9 Verify that all reagents have adequate volume, then tap “OK”

10.6.10 Select the correct number of rows for the run, then tap the “Apply” button.

10.7 Calibration

10.7.1 Environmental Express digestion cups arrive with a volume certification these certificates are kept in the digestion lab. Each lot of cups is verified at the 50mL mark and recorded.

10.7.2 Analytical balances are checked and calibrated using NIST Class "1" Certified weights (See SOP BF-GP-002) daily and entered into a logbook.

10.7.3 Hot block/Auto-Block temperatures are to be checked daily and entered into the digestion log. This is done using a NIST certified thermometer. Acceptance range is 95 degrees ±3 degrees.

10.7.4 Pipettes/ Eppendorf's are verified daily and calibrated every 3 months by a delivery of reagent water on a Certified Balance (See SOP BF-GP-001). This information is logged into a spreadsheet.

10.7.5 Auto-block Reagent pump calibration: all 6 lines are calibrated quarterly to insure accuracy of volumes injected. Per manufacturer ±2% at 10mls is acceptable. These are logged in a spreadsheet.

10.7 Sample Analysis

Refer to Analytical Sop’s BF-ME-009 and BF-ME-010.

11.0 Calculations / Data Reduction

11.1 The concentrations determined are to be reported on the basis of the actual weight of the sample. If a dry weight analysis is desired, then the percent solids of the sample must also be provided.

11.2 If percent solid is desired, a separate determination of percent solids must be performed on a homogeneous aliquot of the sample. See SOP #BF-GP-004.

11.3 Accuracy

ICV / CCV, SRM % Recovery = $\frac{\text{observed concentration}}{\text{known concentration}} \times 100$

MS % Recovery = $\frac{\text{(spiked sample) - (unspiked sample)}}{\text{spiked concentration}} \times 100$

11.4 Precision (RPD)

Matrix Duplicate (DUP) = $\dfrac{|\text{orig. sample value - dup. sample value}|}{[(\text{orig. sample value + dup. sample value})/2]} \times 100$

11.5 **Concentration** = mg/kg or L = $\dfrac{C \times V \times D}{W}$

Where:
C = sample concentration in extract (ppm)
V = Volume of extract (mL)
D = Dilution Factor

W = Weight/Volume of sample aliquot extracted (grams or mLs)

NOTE: All dry weight corrections are made in LIMS at the time the final report is prepared.

11.6 **Method Performance** On an annual basis, Method Detection Limit studies are performed in accordance with 40 CFR 136, Appendix B.

11.7 Demonstration of Capabilities

Doc's are performed on each method from each analyst once a year and approved by the laboratory manager as well as the Quality Manager.

11.8 Training Requirements

The QA Manual or the SOP will be referenced for training requirements.

12.0 Pollution Control

All samples, reagents, and laboratory wastes must be handled with caution. Appropriate safety measures should be employed as detailed in TestAmerica's Laboratory Safety Manual and Chemical Hygiene Plan. All waste will be disposed of in accordance with Federal, State and Local regulations. Where it is reasonably feasible, technological changes have been implemented to minimize the potential for pollution of the environment. Employees will abide by this method and the policies in section 13 of the Corporate Safety Manual for "Waste Management and Pollution Prevention."

It is TestAmerica's policy to evaluate each method and look for opportunities to minimize waste generated (i.e., examine recycling options, ordering chemicals based on quantity needed, preparation of reagents based on anticipated usage and reagent stability).

13.0 Waste Management

Waste management practices are conducted consistent with all applicable rules and regulations. Excess reagents, samples and method process wastes are disposed of in an accepted manner. Waste description rules and land disposal restrictions are followed. Waste disposal procedures are incorporated by reference to TestAmerica's Safety Manual.

14.0 References / Cross-References

Test Methods for Evaluating Solid Waste, Physical/Chemical Methods; SW846, Third Edition; Final Update II; September 1994.

.

15.0 Method Modifications:

Item	Method	Modification
1) VOLUMES	3050B	INITIAL WT 0.5 G : FINAL VOL 50MLS (ALL ACID VOLUMES ARE ADJUSTED ACCORDINGLY)

16.0 Attachments:

Attachment 1: Spike concentrations
Attachment 2: Digestion Batch sheet

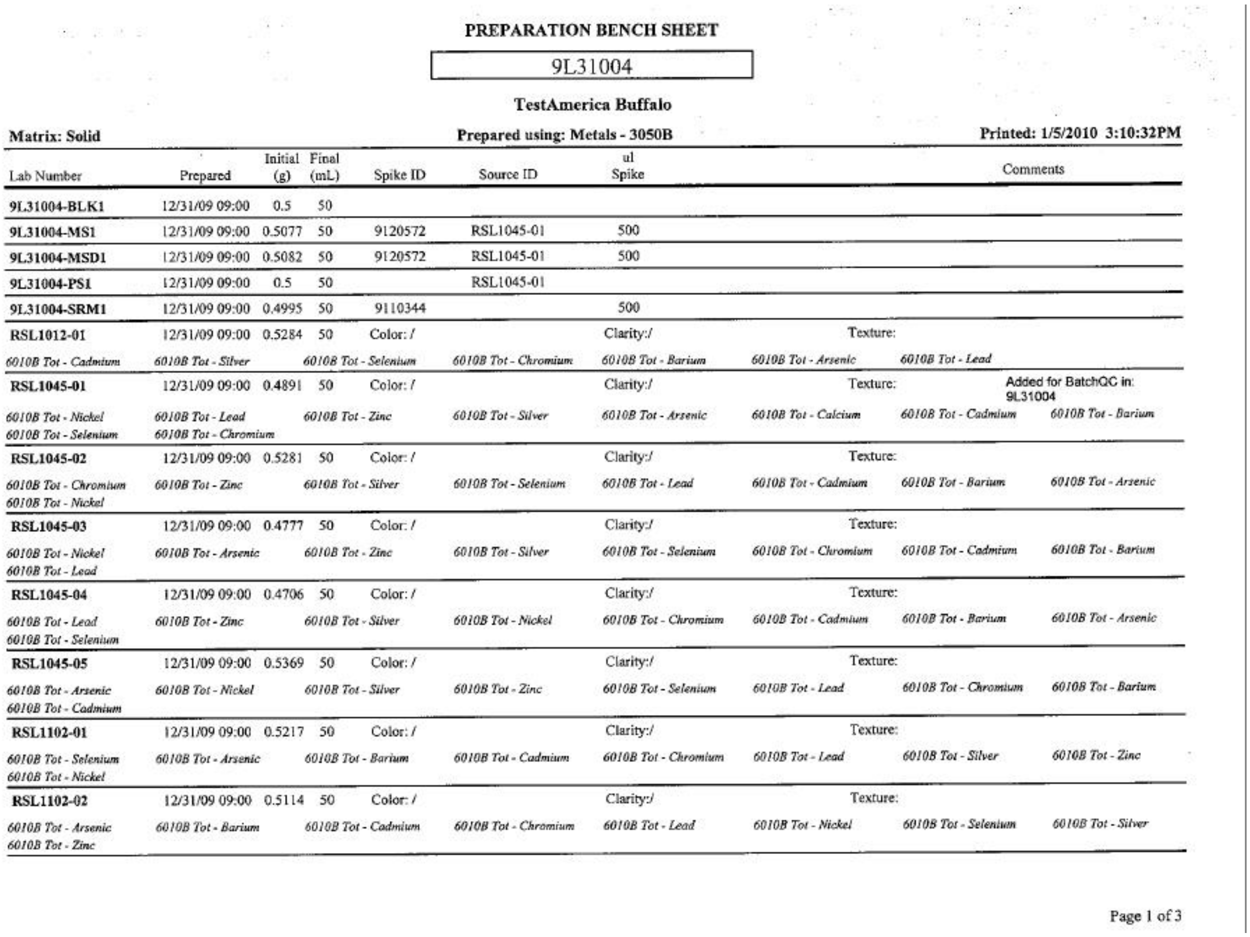
17.0 Revision History

- Revision 0, dated January 25, 2008
    - Integration for TestAmerica and STL operations.
    - Section 6.0: Corrected drying oven temperature range
    - Section 10.03: Deleted ICPMS spike
    - Section 10.1.1: Corrected from 0.2 g to 0.5 g
    - Table 2: Deleted
- Revision 1, dated February 8, 2010
    - Updated for Element
    - Auto-block method separated and updated

Attachment 1

(Table 1) Soil spikes

Analyte	ICUS-1370 (µg/mL)	ICUS-574 (µg/mL)	10 µg/Ml Ag Stock (µg/mL)	40 µg/mL Sn Stock (µg/mL)	Final Conc. In Digestate (ug/mL)	Final Conc. In Soil Sample (mg/kg)
Aluminum		2000			10	1
Antimony	40				.2	.02
Arsenic	40				.2	.02
Barium		40			.2	.02
Beryllium	40				.2	.02
Boron		40			.2	.02
Cadmium	40				.2	.02
Calcium	2000				10	1
Chromium	40				.2	.02
Cobalt	40				.2	.02
Copper	40				.2	.02
Iron	2000				10	1
Lead	40				.2	.02
Magnesium	2000				10	1
Manganese	40				.2	.02
Molybdenum	40				.2	.02
Nickel	40				.2	.02
Potassium		2000			10	1
Selenium	40				.2	.02
Silver			10		.05	.005
Sodium		2000			10	1
Thallium	40				.2	.02
Tin				40	.2	..02
Vanadium	40				.2	.02
Zinc	40				.2	.02
Titanium	40				.2	.02



PREPARATION BENCH SHEET

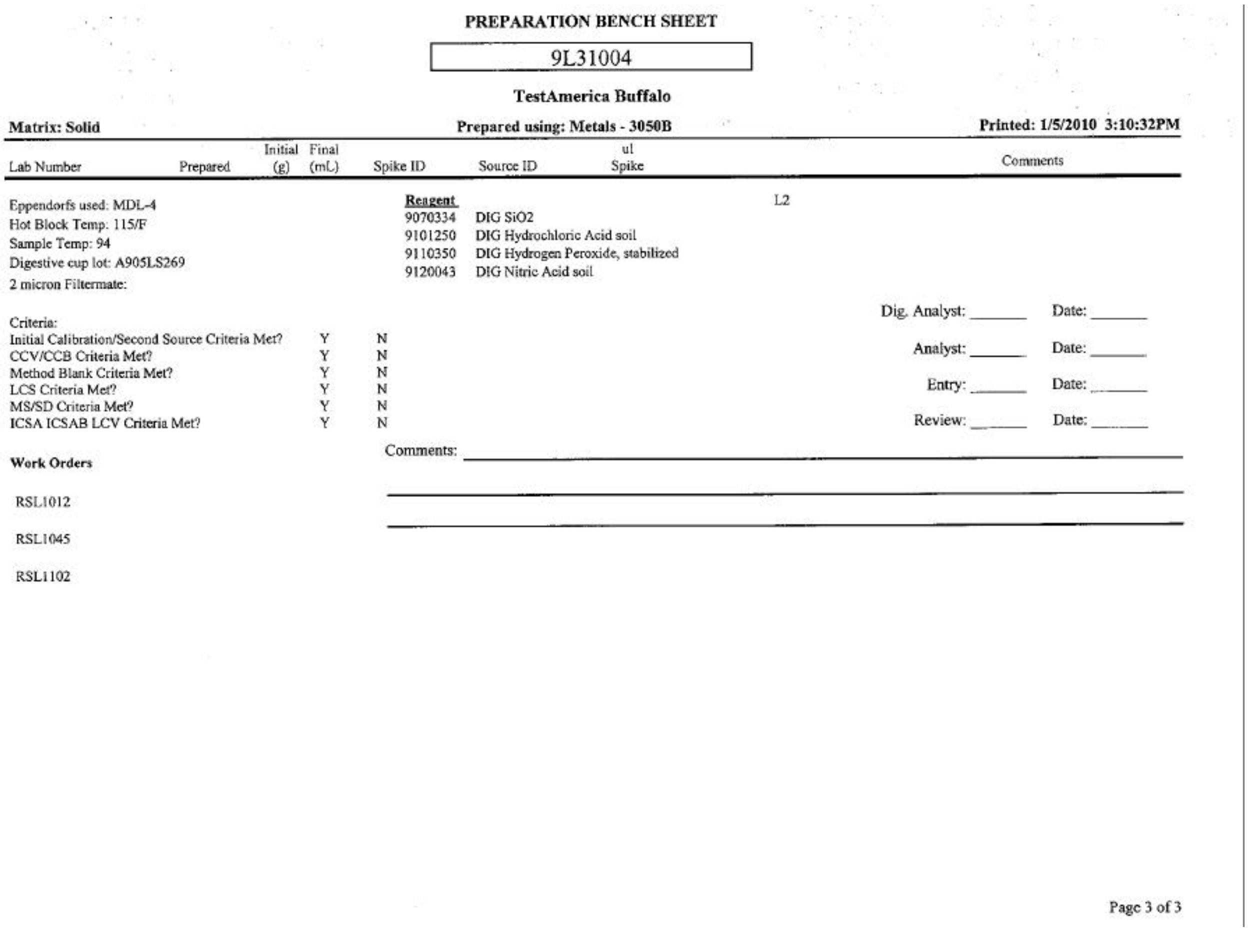
9L31004

TestAmerica Buffalo

Matrix: Solid **Prepared using: Metals - 3050B** **Printed: 1/5/2010 3:10:32PM**

Lab Number	Prepared	Initial (g)	Final (mL)	Spike ID	Source ID	ul Spike	Comments
9L31004-BLK1	12/31/09 09:00	0.5	50				
9L31004-MS1	12/31/09 09:00	0.5077	50	9120572	RSL1045-01	500	
9L31004-MSD1	12/31/09 09:00	0.5082	50	9120572	RSL1045-01	500	
9L31004-PS1	12/31/09 09:00	0.5	50		RSL1045-01		
9L31004-SRM1	12/31/09 09:00	0.4995	50	9110344		500	
RSL1012-01	12/31/09 09:00	0.5284	50	Color: /		Clarity:/	Texture:
6010B Tot - Cadmium	*6010B Tot - Silver*	*6010B Tot - Selenium*	*6010B Tot - Chromium*	*6010B Tot - Barium*	*6010B Tot - Arsenic*	*6010B Tot - Lead*	
RSL1045-01	12/31/09 09:00	0.4891	50	Color: /		Clarity:/	Texture: Added for BatchQC in: 9L31004
6010B Tot - Nickel	*6010B Tot - Lead*	*6010B Tot - Zinc*	*6010B Tot - Silver*	*6010B Tot - Arsenic*	*6010B Tot - Calcium*	*6010B Tot - Cadmium*	*6010B Tot - Barium*
6010B Tot - Selenium	*6010B Tot - Chromium*						
RSL1045-02	12/31/09 09:00	0.5281	50	Color: /		Clarity:/	Texture:
6010B Tot - Chromium	*6010B Tot - Zinc*	*6010B Tot - Silver*	*6010B Tot - Selenium*	*6010B Tot - Lead*	*6010B Tot - Cadmium*	*6010B Tot - Barium*	*6010B Tot - Arsenic*
6010B Tot - Nickel							
RSL1045-03	12/31/09 09:00	0.4777	50	Color: /		Clarity:/	Texture:
6010B Tot - Nickel	*6010B Tot - Arsenic*	*6010B Tot - Zinc*	*6010B Tot - Silver*	*6010B Tot - Selenium*	*6010B Tot - Chromium*	*6010B Tot - Cadmium*	*6010B Tot - Barium*
6010B Tot - Lead							
RSL1045-04	12/31/09 09:00	0.4706	50	Color: /		Clarity:/	Texture:
6010B Tot - Lead	*6010B Tot - Zinc*	*6010B Tot - Silver*	*6010B Tot - Nickel*	*6010B Tot - Chromium*	*6010B Tot - Cadmium*	*6010B Tot - Barium*	*6010B Tot - Arsenic*
6010B Tot - Selenium							
RSL1045-05	12/31/09 09:00	0.5369	50	Color: /		Clarity:/	Texture:
6010B Tot - Arsenic	*6010B Tot - Nickel*	*6010B Tot - Silver*	*6010B Tot - Zinc*	*6010B Tot - Selenium*	*6010B Tot - Lead*	*6010B Tot - Chromium*	*6010B Tot - Barium*
6010B Tot - Cadmium							
RSL1102-01	12/31/09 09:00	0.5217	50	Color: /		Clarity:/	Texture:
6010B Tot - Selenium	*6010B Tot - Arsenic*	*6010B Tot - Barium*	*6010B Tot - Cadmium*	*6010B Tot - Chromium*	*6010B Tot - Lead*	*6010B Tot - Silver*	*6010B Tot - Zinc*
6010B Tot - Nickel							
RSL1102-02	12/31/09 09:00	0.5114	50	Color: /		Clarity:/	Texture:
6010B Tot - Arsenic	*6010B Tot - Barium*	*6010B Tot - Cadmium*	*6010B Tot - Chromium*	*6010B Tot - Lead*	*6010B Tot - Nickel*	*6010B Tot - Selenium*	*6010B Tot - Silver*
6010B Tot - Zinc							

Page 1 of 3



PREPARATION BENCH SHEET

9L31004

TestAmerica Buffalo

Matrix: Solid
Prepared using: Metals - 3050B
Printed: 1/5/2010 3:10:32PM

Lab Number	Prepared	Initial (g)	Final (mL)	Spike ID	Source ID	ul Spike	Comments

Eppendorfs used: MDL-4
Hot Block Temp: 115/F
Sample Temp: 94
Digestive cup lot: A905LS269
2 micron Filtermate:

Reagent	
9070334	DIG SiO2
9101250	DIG Hydrochloric Acid soil
9110350	DIG Hydrogen Peroxide, stabilized
9120043	DIG Nitric Acid soil

L2

Criteria:

Initial Calibration/Second Source Criteria Met?	Y	N
CCV/CCB Criteria Met?	Y	N
Method Blank Criteria Met?	Y	N
LCS Criteria Met?	Y	N
MS/SD Criteria Met?	Y	N
ICSA ICSAB LCV Criteria Met?	Y	N

Dig. Analyst: _______ Date: _______

Analyst: _______ Date: _______

Entry: _______ Date: _______

Review: _______ Date: _______

Comments: ____

____

____

Work Orders

RSL1012

RSL1045

RSL1102

Page 3 of 3

Title: Sulfide
Method 376.2, SM 4500S-2 D

Once printed, this is considered an uncontrolled document

Approvals (Signature/Date):

Lisa R Matecki
01/27/10
Lisa R. Matecki Date
Department Manager

Kenneth E. Kasperek
01/27/10
Kenneth E. Kasperek Date
Health & Safety Manager

Peggy Gray-Erdmann
01/27/10
Peggy Gray-Erdmann Date
Quality Assurance Manager

01/27/10
Christopher A. Spencer Date
Laboratory Director

Facility Distribution No. ____________ **Distributed To:**_________________________

1.0 Scope and Application

1.1 Analytes, Matrix(s), and Reporting Limits

1.1.1 This method is applicable to drinking, surface and saline waters domestic and industrial wastes.

1.1.2 This method is suitable for the measurement of total and dissolved sulfide in concentrations up to 20 mg/L. Acid insoluble sulfides are not measured by the use of this test. Copper sulfide is the only common sulfide in this class.

1.1.3 On occasion clients may request modifications to this SOP. These modifications are handled following the procedures outlined in the Quality Assurance Manual.

2.0 Summary of Method

Sulfide reacts with dimethyl-p-phenylenediamine (p-aminodimethyl aniline) to form methylene blue. The intensity of the blue color is proportional to the sulfide concentration. Test results are measured at 625 nm.

3.0 Definitions

Refer to the TestAmerica Lab Quality Manual.

4.0 Interferences

4.1 Strong reducing agents, such as thiosulfate and sulfite, interfere by preventing formation of the blue color. Thiosulfate at concentrations about 10 mg/L may retard or prevent it.

4.2 Ferrocyanide produces a blue color

4.3 Sulfide itself prevents reaction if its concentration is very high, in the range of several hundred milligrams per liter. To avoid the possibility of false negative results, the antimony test may be used to obtain a qualitative result in industrial wastes likely to contain sulfur but giving no color by the Methylene Blue method

4.4 Sulfide may be volatilized by aeration therefore samples must be taken with a minimum of aeration.

4.5 Any oxygen added to the sample may convert the sulfide to an immeasurable form.

4.6 If the sample is not preserved with zinc acetate and sodium hydroxide the analysis of dissolved sulfide must be commenced immediately.

5.0 Safety

Employees must abide by the policies and procedures in the Corporate Safety Manual, Radiation Safety Manual and this document.

This procedure may involve hazardous material, operations and equipment. This SOP does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of the method to follow appropriate safety, waste disposal and health practices under the assumption that all samples and reagents are potentially hazardous. Safety glasses, gloves, lab coats and closed-toe, nonabsorbent shoes are a minimum.

5.1 Specific Safety Concerns or Requirements

Sodium Sulfide will form Hydrogen Sulfide (HS) gas if combined with water moisture or strong acids. **Inhalation of HS gas may be fatal**.

5.2 Primary Materials Used

The following is a list of the materials used in this method, which have a serious or significant hazard rating. **Note: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the MSDS for each of the materials listed in the table.** A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the MSDS for each material before using it for the first time or when there are major changes to the MSDS.

Material [1]	**Hazards**	**Exposure Limit [2]**	**Signs and symptoms of exposure**
Sulfide 1 Reagent	Harmful if inhaled, Causes severe burns, Carcinogen		Avoid contact with eyes and skin. Do not breathe. Wash thoroughly after handling. Use with adequate ventilation. Symptoms include irritation, abdominal pain, vomiting, dizziness, fever, coma and liver damage.
Sulfide 2 Reagent	Harmful if inhaled, Causes severe burns, Carcinogen		Avoid contact with eyes and skin. Do not breathe. Wash thoroughly after handling. Use with adequate ventilation. Symptoms include irritation, abdominal pain, vomiting, dizziness, fever, coma and liver damage.
Sulfide Standard	Corrosive	10 ppm-TWA 15 ppm-STEL	Will form Hydrogen Sulfide (HS) gas if combined with strong acids. **Inhalation of HS gas may be fatal**. Symptoms include painful conjunctivitis, headache, nausea, dizziness, coughing and, in extreme cases, pulmonary edema and possible death. Irritant. Contact with skin can produce serious caustic burns with painful inflammation and possible destruction of tissue. Inflammation, tearing and pain may be expected. Severe contact can cause destruction of tissue.
1 – Always add acid to water to prevent violent reactions.			
2 – Exposure limit refers to the OSHA regulatory exposure limit.			

5.3 Spill Response

Any spills should be cleaned as soon as possible. Neutralize acid spills before cleaning.

6.0 Equipment and Supplies

6.1 Instrumentation

Hach Odyssey Spectrophotometer

6.2 Supplies

6.2.1 Hach Sample Cells with cap, 25 ml volume (cat no. 24019-06)
6.2.2 Eppendorfs capable of dispensing 100-1,000µL and 500-5,000µL

7.0 Reagents and Standards

7.1 Sulfide stock solution, 5000 µg/mL: Dissolve 6.40-g sodium sulfide in 200 mL of deionized water. **A commercially prepared and NIST certified solution may be used instead**. If the prepared solution is used, it is unnecessary to standardize.

7.2 Second source sulfide stock solution, purchased from separate vendor or if unavailable from a separate lot number.

7.3 HACH prepared Sulfide 1 reagent: contains Sulfuric Acid and demineralized water.

7.4 HACH prepared Sulfide 2 reagent: contains Potassium dichromate and demineralized water

8.0 Sample Collection, Preservation, Shipment and Storage

Sample container, preservation techniques and holding times may vary and are dependent on sample matrix, method of choice, regulatory compliance, and/or specific contract or client requests. Listed below are the holding times and the references that include preservation requirements.

Matrix	Sample Container	Min. Sample Size	Preservation	Holding Time	Reference
Waters	HDPE	50 mLs	Zinc Acetate and NaOH Cool 4 ± 2°C	7 Days	
Soils	Glass	20 grams	Cool 4 ± 2°C	7 Days from ASTM preparation	N/A

9.0 Quality Control

9.1 **Sample QC** - The following quality control samples are prepared with each batch of samples.

Quality Controls	Frequency	Control Limit
Method Blank (MB)	1 in 10 or fewer samples	< Rpt. Limit
Laboratory Control Sample (LCS)	1 in 10 or fewer samples	Statistical Limits [2]
Matrix Spike (MS)[1]	1 in 20 or fewer samples	Statistical Limits [2]
MS Duplicate (MSD)[1]	1 in 20 or fewer samples	Statistical Limits [2]

[1] The sample selected for MD/MS is done randomly, unless specifically requested by a client.
[2] Statistical control limits are updated annually and are entered into LIMS.

9.2 Instrument QC

9.2.1 Initial Calibration Verification (ICV) – Prepare an ICV using a separate source from the calibration curve. An ICV must be run after each new curve and found to be compliant to +/- 10% of the true value when compared against the new calibration data.

9.2.2 Laboratory Control Sample (LCS) – Prepare a LCS using a pre-made sulfide standard at a concentration of 1.0ppm. Analyze one LCS at the beginning of each analytical run. Values obtained must be within +/- 10% of the true value.

9.2.3 Continuing Calibration Verification (CCV) – Prepare a calibration standard from the pre-made sulfide standard at a concentration of 0.5 ppm. Analyze one CCV after every ten sample analyses. Values obtained must be within +/- 10% of the true value.

9.2.4 Method Blank (MBLK) – To determine freedom from contamination, analyze one method blank at the beginning and end of the analytical run and after every ten samples during the analysis. A method blank consists of reagent water carried through the entire analytical procedure.

9.2.5 Sample Duplicate (MD): Sample duplicates should be analyzed at least once for every twenty samples or less.

9.2.6 Sample Spike (MS): Sample spikes must be analyzed every twenty samples or less. Samples must fall within the established range that is calculated yearly and available in LIMS. Sample results associated with a spike recovery that fails should be evaluated to see if reanalysis is required.

9.2.7 Calibration Acceptance Summary

Step	***Standards***	***Type***	***Control Limit***	***Frequency***
Initial Cal	7 standards 0.10ppm, 0.25 ppm, 0.40 ppm, 0.50 ppm, 0.65 ppm, 0.80 ppm, 1.00 ppm	Linear	R $\geq$ 0.995	Performed not less than every three months
ICV	1.0ppm		90-110%	Once after each new curve
LCS	1.0ppm		90-110%	Once at the beginning of an analytical run
CCV	0.50 ppm			Every ten samples
MBLK	0 ppm		< Report Limit	Every ten samples
MD	0 ppm		< 20% RPD from base sample	Every 20 samples
MS	0.50 ppm		Statistical Limits	Every 20 samples

10.0 Procedure

10.1 Calibration

10.1.1 Curve standards: Prepare seven curve standards in the range from 0.10 ppm to 1.00 ppm from standardized sodium sulfite and a blank. Analyze curve standards according to the procedure herein.

10.1.2 A calibration curve must be run at a minimum every three months.

10.1.3 Curve correlation (r) must be 0.995 or better.

10.2 Sample Analysis

10.2.1 Set spectrophotometer to 625 nm.

10.2.2 Avoiding excess agitation of the sample, add 25mL of sample to a sample cell.

10.2.3 Measure 25mL of deionized water into a second sample cell.

10.2.4 Add 1mL of Sulfide 1 Reagent to each sample cell.

10.2.5 Add 1mL of Sulfide 2 Reagent to each cell.

10.2.6 Cap the cell and immediately invert to mix.

10.2.7 An orange/pink color will develop then the solution will turn blue if sulfide is present.

10.2.8 Wait 5-minutes for the reaction period before reading.

10.2.9 When 5 minutes is up, wipe the blank and place it in the cell holder.

10.2.10 Zero the instrument with the blank.

10.2.11 Wipe the prepared sample and place it into the cell holder.

10.2.12 Record the sample absorbency and compare against calibration curve.

11.0 Calculations / Data Reduction

11.1 Percent Recovery for Analyses Involving Spikes:

$$\%\ \text{Recovery} = \left[\frac{(\text{SSR - SR})}{\text{SA}}\right] \text{ x } 100$$

where:

SSR = spiked sample result
SR = sample result

11.2 Relative Percent Difference (RPD):

$$\text{RPD} = \frac{|x_1 - x_2|}{\left(\frac{x_1 + x_2}{2}\right)} \times 100$$

where:

x_1 = analytical % recovery
x_2 = replicate % recovery

11.3 **Concentration** = mg/kg or L $= \frac{C \times V \times D}{W}$

Where:
C = sample concentration in extract (ppm)
V = Volume of extract (mL)
D = Dilution Factor

W = Weight/Volume of sample aliquot extracted (grams or mLs)

NOTE: All dry weight corrections are made in LIMS at the time the final report is prepared.

12.0 Method Performance

12.1 Method Detection Limit Study (MDL)

The method detection limit (MDL) is to be performed yearly in accordance with the specifications in 40 CFR 136, appendix B, and must demonstrate the ability to quantitate at or below the reporting limit for each analyte. The current MDL is on file with the department supervisor and the QA Department.

12.2 Demonstration of Capabilities

12.2.1 A one–time initial demonstration of performance for each individual method for water matrices must be generated.

12.2.2 This requires quadruplicate analysis of a mid–level check standard containing all of the standard analytes for the method using the same procedures used to analyze samples, including sample preparation.

12.2.3 Compare these results with the acceptance criteria given in the Method or to laboratory historical limits (if available).

12.2.4 Repeat the test for any analyte that does not meet the acceptance criteria. Repeated failure for any analyte indicates the need for the laboratory to evaluate the analytical procedure and take corrective action.

12.3 Training Requirements

12.3.1 The supervisor has the responsibility to ensure that this procedure is performed by an analyst who has been properly trained in its use and has the required experience.

12.3.2 The following analyst validation information is maintained for this method in the laboratory QA files.

12.3.3 The analyst must complete the laboratory safety orientation training that includes, but is not limited to, chemicals, PPE requirements, and electrical safety.

12.3.4 The analyst must read and understand this SOP.

12.3.5 The analyst must read and understand the Method used as reference for this SOP.

12.3.6 The analyst must complete a DOC or successfully analyze PT samples annually.

12.3.7 The analyst must complete the TestAmerica Quality Assurance Training.

13.0 Pollution Control

All waste will be disposed of in accordance with Federal, State and Local regulations. Where reasonably feasible, technological changes have been implemented to minimize the potential for pollution of the environment. Employees will abide by this method and the policies in section 13 of the Corporate Safety Manual for “Waste Management and Pollution Prevention.”

14.0 Waste Management

Aqueous waste generated during procedure is disposed of in the “A” waste containers. Preserved samples and sulfide stock solutions must be disposed of in "D" waste containers.

15.0 References / Cross-References

15.1 Methods for Chemical Analysis of Water and Wastes, U.S. Environmental Protection Agency, 376.2-1.

15.2 Standard Methods for the Examination of Water and Wastewater, 4500-S2-D

16.0 Method Modifications:

Item	Method	Modification
1	4500-S2-D	Spectrophotometer is set to a wavelength of 625nm.
2	4500-S2-D	Reagents used are purchased pre-made and at known concentration and certified by the vendor.

17.0 Attachments

17.1 Analytical Run Log
17.2 Analytical Batch
17.3 Wet Chemistry Batch Summary Sheet

18.0 Revision History

- Revision 0, dated January 30, 2008
    - Integration for TestAmerica and STL operations.
    - Updated procedure for HACH reagents

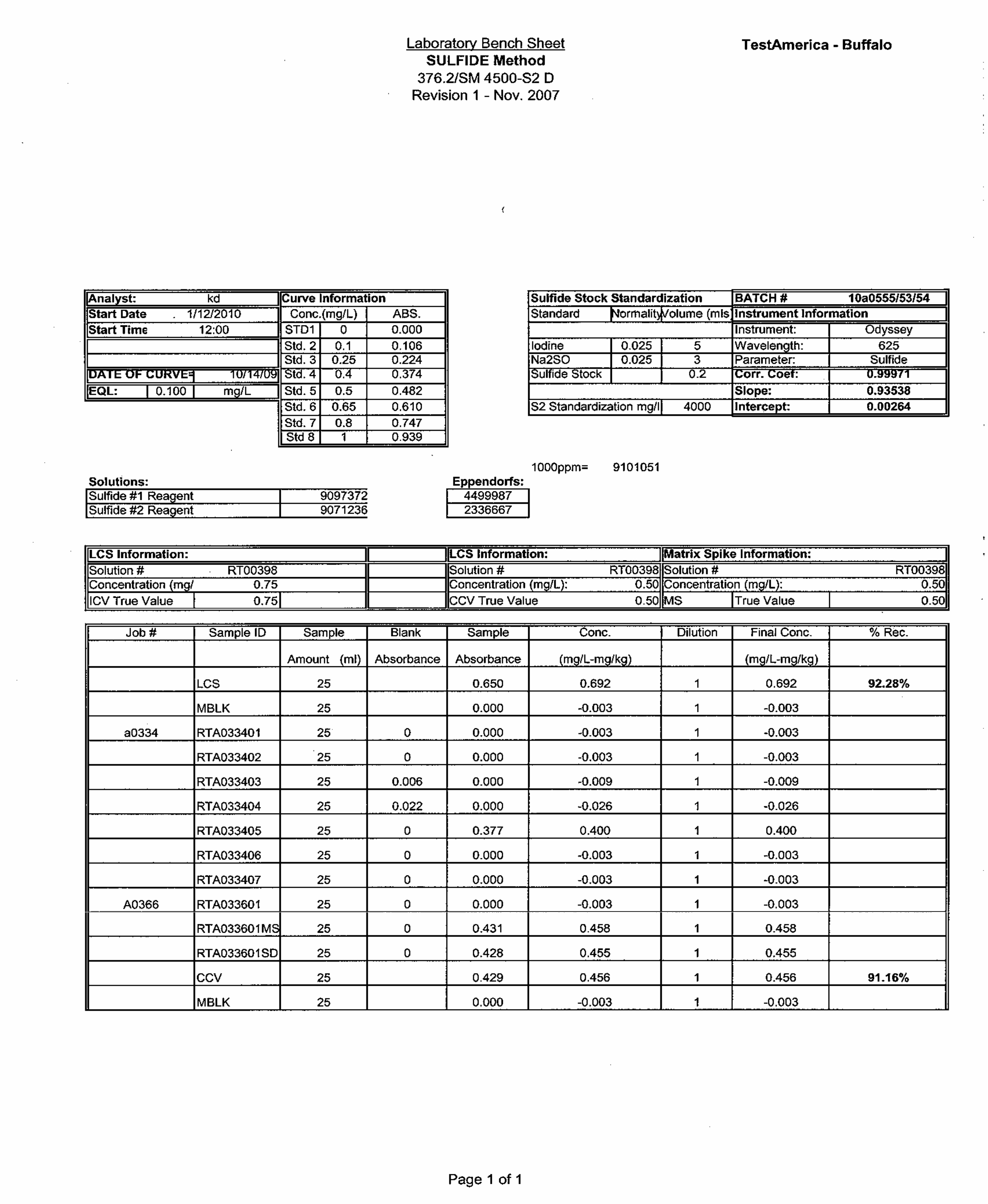
- Revision 1, dated January 27, 2010
    - Updated attachments

Attachment 17.1

Analytical Run Log

LCS
MBLK
Sample
Sample
Sample
Sample
Sample
Sample
Sample
Sample
Sample
Sample
CCV
CCB
Sample
Sample
Sample
Sample
Sample
Sample
Sample
Sample
Sample Duplicate (MD)
Sample Spike (MS)
CCV
CCB

Attachment 17.2
Analytical Batch



Laboratory Bench Sheet
SULFIDE Method
376.2/SM 4500-S2 D
Revision 1 - Nov. 2007

TestAmerica - Buffalo

Analyst:	kd	
Start Date	1/12/2010	
Start Time	12:00	
DATE OF CURVE=	10/14/09	
EQL:	0.100	mg/L

Curve Information		
Conc.(mg/L)		ABS.
STD1	0	0.000
Std. 2	0.1	0.106
Std. 3	0.25	0.224
Std. 4	0.4	0.374
Std. 5	0.5	0.482
Std. 6	0.65	0.610
Std. 7	0.8	0.747
Std 8	1	0.939

Sulfide Stock Standardization		
Standard	Normality	Volume (mls
Iodine	0.025	5
Na2SO	0.025	3
Sulfide Stock		0.2
S2 Standardization mg/l		4000

BATCH #	**10a0555/53/54**
Instrument Information	
Instrument:	Odyssey
Wavelength:	625
Parameter:	Sulfide
Corr. Coef:	**0.99971**
Slope:	**0.93538**
Intercept:	**0.00264**

1000ppm= 9101051

Solutions:

Sulfide #1 Reagent	9097372
Sulfide #2 Reagent	9071236

Eppendorfs:

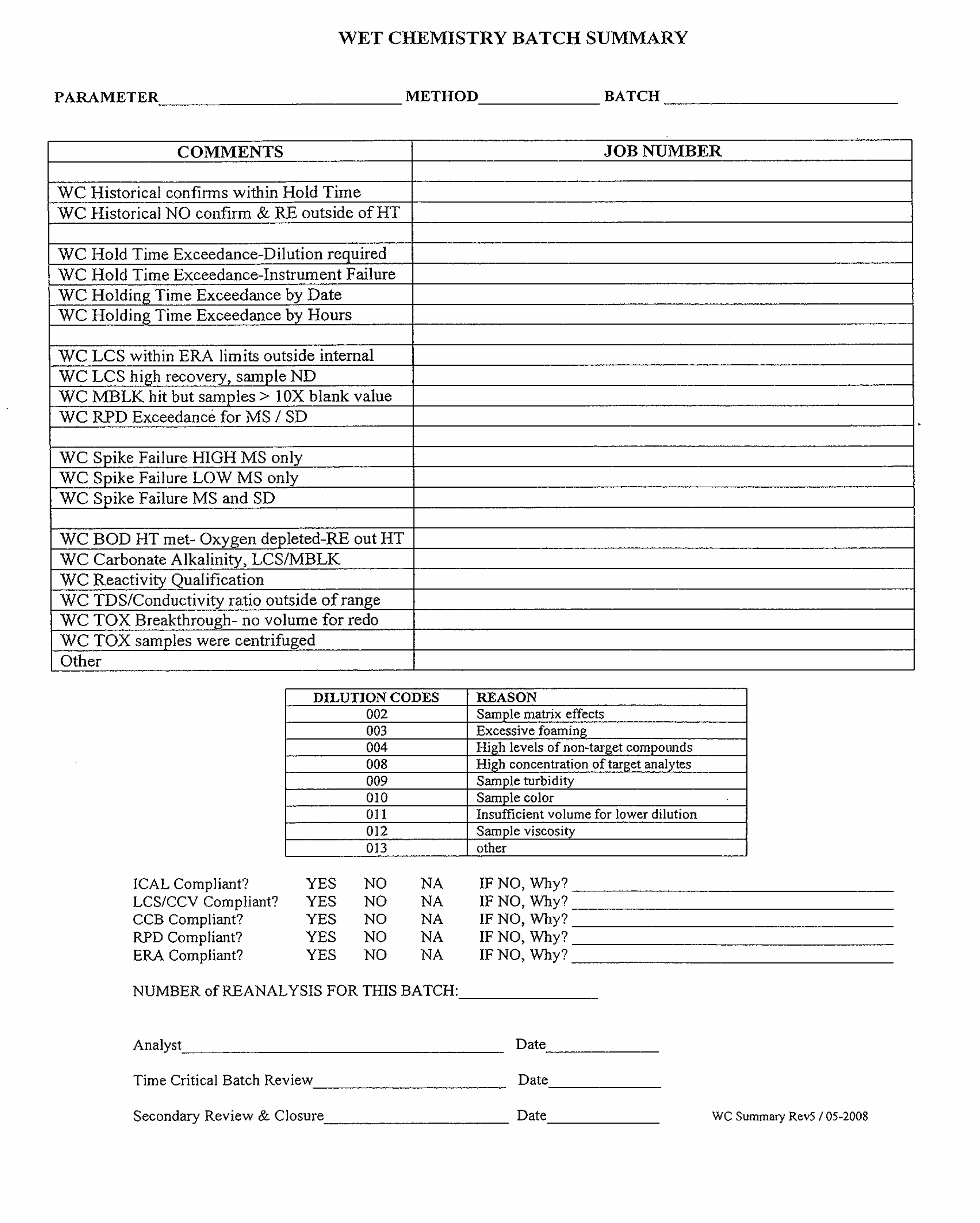
4499987
2336667

LCS Information:			**LCS Information:**		**Matrix Spike Information:**		
Solution #	RT00398		Solution #	RT00398	Solution #		RT00398
Concentration (mg/	0.75		Concentration (mg/L):	0.50	Concentration (mg/L):		0.50
ICV True Value	0.75		CCV True Value	0.50	MS	True Value	0.50

Job #	Sample ID	Sample Amount (ml)	Blank Absorbance	Sample Absorbance	Conc. (mg/L-mg/kg)	Dilution	Final Conc. (mg/L-mg/kg)	% Rec.
	LCS	25		0.650	0.692	1	0.692	**92.28%**
	MBLK	25		0.000	-0.003	1	-0.003	
a0334	RTA033401	25	0	0.000	-0.003	1	-0.003	
	RTA033402	25	0	0.000	-0.003	1	-0.003	
	RTA033403	25	0.006	0.000	-0.009	1	-0.009	
	RTA033404	25	0.022	0.000	-0.026	1	-0.026	
	RTA033405	25	0	0.377	0.400	1	0.400	
	RTA033406	25	0	0.000	-0.003	1	-0.003	
	RTA033407	25	0	0.000	-0.003	1	-0.003	
A0366	RTA033601	25	0	0.000	-0.003	1	-0.003	
	RTA033601MS	25	0	0.431	0.458	1	0.458	
	RTA033601SD	25	0	0.428	0.455	1	0.455	
	CCV	25		0.429	0.456	1	0.456	**91.16%**
	MBLK	25		0.000	-0.003	1	-0.003	

Page 1 of 1

Attachment 17.3
Wet Chemistry Batch Summary Sheet



WET CHEMISTRY BATCH SUMMARY

PARAMETER______________________ METHOD____________ BATCH ______________________

COMMENTS	JOB NUMBER
WC Historical confirms within Hold Time	
WC Historical NO confirm & RE outside of HT	
WC Hold Time Exceedance-Dilution required	
WC Hold Time Exceedance-Instrument Failure	
WC Holding Time Exceedance by Date	
WC Holding Time Exceedance by Hours	
WC LCS within ERA limits outside internal	
WC LCS high recovery, sample ND	
WC MBLK hit but samples > 10X blank value	
WC RPD Exceedance for MS / SD	
WC Spike Failure HIGH MS only	
WC Spike Failure LOW MS only	
WC Spike Failure MS and SD	
WC BOD HT met- Oxygen depleted-RE out HT	
WC Carbonate Alkalinity, LCS/MBLK	
WC Reactivity Qualification	
WC TDS/Conductivity ratio outside of range	
WC TOX Breakthrough- no volume for redo	
WC TOX samples were centrifuged	
Other	

DILUTION CODES	REASON
002	Sample matrix effects
003	Excessive foaming
004	High levels of non-target compounds
008	High concentration of target analytes
009	Sample turbidity
010	Sample color
011	Insufficient volume for lower dilution
012	Sample viscosity
013	other

ICAL Compliant? YES NO NA IF NO, Why? ______________________
LCS/CCV Compliant? YES NO NA IF NO, Why? ______________________
CCB Compliant? YES NO NA IF NO, Why? ______________________
RPD Compliant? YES NO NA IF NO, Why? ______________________
ERA Compliant? YES NO NA IF NO, Why? ______________________

NUMBER of REANALYSIS FOR THIS BATCH:____________

Analyst____________________________ Date__________

Time Critical Batch Review__________________ Date__________

Secondary Review & Closure__________________ Date__________

WC Summary Rev5 / 05-2008

Title: Method 3010A Acid Digestion of Aqueous Samples and Extracts for Total Metals Analysis by ICP-AES

Once printed, this is considered an uncontrolled document

Approvals (Signature/Date):

E. Scott Wagner 02/08/10
E. Scott Wagner Date
Department Manager

Kenneth E. Kasperek 02/08/10
Kenneth Kasperek Date
Health & Safety Manager

Peggy Gray-Erdmann 02/08/10
Peggy Gray-Erdmann Date
Quality Assurance Manager

02/08/10
Christopher Spencer Date
Laboratory Director

Facility Distribution No. ___________ **Distributed To:**________________________

1.0 Scope and Application

1.1. This digestion procedure is used for the preparation of aqueous samples, TCLP and mobility-procedure extracts, and wastes that contain suspended solids for analysis, by inductively coupled plasma atomic emission spectroscopy (ICP-AES). This procedure is used to determine total metals.

2.0 Analytes, Matrix(s), and Reporting Limits

2.1 Aqueous samples, TCLP and mobility-procedure extracts, and wastes that contain suspended solids for total metals analysis

2.2 Reporting Limit: N/A

2.3 Samples prepared by Method 3010A may be analyzed by ICP-AES for the following elements:

Aluminum
Antimony
Arsenic
Barium
Beryllium
Boron
Cadmium
Calcium
Chromium
Cobalt
Copper
Iron
Lead
Magnesium
Manganese
Molybdenum
Nickel
Potassium
Selenium
Silver
Sodium
Thallium
Tin
Titanium
Vanadium
Zinc

2.4 On occasion clients may request modifications to this SOP. These modifications are handled following the procedures outlined in the Quality Assurance Manual.

3.0 Summary of Method

3.1 A mixture of Nitric acid and the sample is refluxed in a digestion cup. This step is repeated with additional portions of Nitric acid until the digestate is light in color or until its color has stabilized. After the digestate has been brought to a low volume between 5 and 10 ml, it is refluxed with Hydrochloric acid and finally brought up to the final volume of 50 ml. If the sample should go to dryness, it must be discarded and the sample re-prepared.

4.0 Definitions

4.1 Total Metals – The concentration determined on an unfiltered acidified sample following vigorous digestion.

4.2 Trace ICP – an ICP with the viewing angle along the long axis of the torch.

5.0 Interferences

5.1 Potential sources of trace metals contamination include: metallic or metal-containing labware (e.g., talc gloves which contain high levels of zinc), containers, impure reagents, dirty glassware, improper sample transfers, dirty work areas, atmospheric inputs such as dirt and dust, etc. Be aware of potential sources of contamination and take appropriate measures to minimize or avoid them.

5.2 Physical interference effects may contribute to inaccuracies in the determination of trace elements. Oils, solvents and other matrices may not be digested using this method if they are not soluble with acids. If physical interferences are present, they should be documented.

5.3 Visual interferences or anomalies (such as foaming, emulsions, precipitates, etc.) must be documented on the digestion log.

5.4 Allowing samples to boil or go dry during digestion may result in the loss of volatile metals. If this occurs the sample must be re-prepared. Antimony is easily lost by volatilization from hydrochloric acid media.

5.5 Precipitation of silver chloride (AgCl) may occur when chloride ions and high concentrations of silver (i.e., greater than 1 mg/L) are present in the sample. Samples containing more than 1 mg/L of silver can be diluted, re-digested and reanalyzed to produce more accurate results upon project manager/client request.

6.0 Safety

6.1 Employees must abide by the policies and procedures in the Corporate Safety Manual, Radiation Safety Manual and this document.

6.2 Nitrile Gloves are to be used when handling all standards and samples. Safety glasses must be worn at all times. Extra care is taken when dispensing concentrated acids. Concentrated acids are to be dispensed only in the fume hood.

6.3 Specific Safety Concerns or Requirements

Samples that contain high concentrations of carbonates, organic material, or samples that are at an elevated pH can react violently when acids are added.

6.4 Primary Materials Used

The following is a list of the materials used in this method, which have a serious or significant hazard rating. **Note: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the MSDS for each of the materials listed in the table.** A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the MSDS for each material before using it for the first time or when there are major changes to the MSDS.

Material (1)	**Hazards**	**Exposure Limit (2)**	**Signs and symptoms of exposure**
Hydrochloric Acid	Corrosive Poison	5 ppm-Ceiling	Inhalation of vapors can cause coughing, choking, inflammation of the nose, throat, and upper respiratory tract, and in severe cases, pulmonary edema, circulatory failure, and death. Can cause redness, pain, and severe skin burns. Vapors are irritating and may cause damage to the eyes. Contact may cause severe burns and permanent eye damage.
.2 Nitric Acid	Corrosive Oxidizer Poison	2 ppm-TWA 4 ppm-STEL	Nitric acid is extremely hazardous; it is corrosive, reactive, an oxidizer, and a poison. Inhalation of vapors can cause breathing difficulties and lead to pneumonia and pulmonary edema, which may be fatal. Other symptoms may include coughing, choking, and irritation of the nose, throat, and respiratory tract. Can cause redness, pain, and severe skin burns. Concentrated solutions cause deep ulcers and stain skin a yellow or yellow-brown color. Vapors are irritating and may cause damage to the eyes. Contact may cause severe burns and permanent eye damage.
1 – Always add acid to water to prevent violent reactions.			
– Exposure limit refers to the OSHA regulatory exposure limit.			

7.0 Equipment and Supplies

7.1 *Environmental Express* Hot Blocks
7.2 *Environmental Express* 50 ml Polypropylene digestion cups
7.3 Eppendorf pipettes and pipette tips
7.4 NIST Certified Thermometer
7.5 Filters

8.0 Reagents and Standards

8.1 Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available. Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination

8.2 Laboratory Reagent Water – produced by a Millipore de-ionized system. The maximum allowed conductivity is 1.0 ohms-cm at 25°C. The reagent water must be free of the analytes of interest as demonstrated through the analysis of method blanks.

8.3 Nitric Acid (HNO3), concentrated, trace metal grade or better

8.4 Hydrochloric acid (HCl), concentrated, trace metal grade or better

8.4.1 The certificates of analysis for the concentrated acids are listed on the bottle. Whenever the purity of the acid is suspect, the acid should be analyzed by ICP-MS to determine levels of impurities. If impurity concentrations are at such levels that method blanks are <MDL, the acid may be used.

8.5 1:1 HCl prepared by mixing equal volume of reagent water and concentrated Hydrochloric acid. Pour concentrated acid to water; never pour water to concentrated acid.

8.6 Spike standards

8.6.1 Silver (Ag) - 10 μg/ml in 2% HNO3: Add 1.0 ml of 1000 μg/ml Ag stock standard to 50 ml reagent water in a 100 ml volumetric flask. Add 4 ml concentrated HNO3. Dilute to volume with reagent water

8.6.2 Tin (Sn) - 40 μg/ml in 2% HNO3: Add 4.0 ml of 1000 μg/ml Sn stock standard to 50 ml reagent water in a 100 ml volumetric flask. Add 4 ml concentrated HNO3. Dilute to volume with reagent water.

8.6.3 ICP-AES spikes: ICUS-1370, ICUS-574, 10 μg/ml Ag, 40 μg/ml Sn and ICUS-1454 (this spike is used for all TCLP's). See Table 1 for details.

8.6.4 The Certificates of Analysis for these standards are kept by analysts in the Digestion Lab.

9.0 <u>Sample Collection, Preservation, Shipment and Storage</u>

9.1 Aqueous wastewaters must be acidified to a pH of < 2 with concentrated HNO_3. Refrigeration is not required.

9.2 Sample holding time for metals is 180 days from the date of collection to the date of analysis.

9.3 If Boron is to be determined, collection into a plastic container is preferred.

9.4 If samples are received unpreserved, the Project Manager must be contacted immediately so that the client can be informed.

9.5 The matrix spike solution must be added to TCLP leachates before the samples are acidified.

10.0 Quality Control

10.1 Sample QC

10.1.1 Method Blank (BLK) is a volume of reagent water processed through the sample preparation and analysis procedure. For each batch of samples (not to exceed 20 samples), a Method Blank must be employed. This blank is useful in monitoring any contamination.

10.1.2 Blank Spike (BS) is a volume of reagent water spiked with known concentrations of analytes and carried through the preparation and analysis procedure. For each batch of samples (not to exceed 20 samples), a BS must be employed to determine analyte recovery.

10.1.3 A Matrix Spike and/or Matrix Spike Duplicate is an aliquot of sample that has been fortified with known concentrations of analytes and carried through the preparation and analysis procedure. For each batch of samples (not to exceed 20 samples), a Matrix Spike (MS) should be processed on a routine basis to determine biases in the analytical results due to sample matrix. Matrix Spike Duplicates (SD) samples may also be used to determine matrix effects on digestion and detection.

10.1.4 For each batch of samples (not to exceed 20 samples), replicate samples should be processed on a routine basis. Replicate samples are either Matrix Duplicate (MD) or Matrix *Spike* Duplicate (SD) depending on the clients' request, but are usually Matrix Spike Duplicates. Replicate samples will be used to determine precision. MD is just another aliquot of the selected sample. SD is just another MS that is processed through the preparation and analysis procedure.

10.2 Instrument QC

10.2.1 Reference SOP BF-ME-009

11.0 Procedure

11.1 Sample Preparation

11.1.1 One time procedural variations are allowed only if deemed necessary in the professional judgment of supervision to accommodate variation in sample matrix, chemistry, sample size or other parameters. Any variation in procedure shall be completely documented using a Job Exception Form. The Job Exception is routed to the Metals supervisor and then to the lab Project Manager and QA staff for possible client notification. The Job Exception should be placed in the project file. Any unauthorized deviations from this procedure must also be documented as a nonconformance, with cause and corrective addition described.

11.1.2 Method Blank (BLK): For each digestion batch of 20 samples or less, transfer 50 ml of laboratory reagent water to a digestion cup and carry through the entire analytical process.

11.1.3 Blank Spike (BS): For each digestion batch of 20 samples or less, transfer 50 ml of laboratory reagent water to a digestion cup, fortify with the following spike solutions and carry through the entire analytical process.

Spike Standards	**Volume**
ICUS-1370	0.25 ml
ICUS-574	0.25 ml
Ag (10 ug/ml)	0.25 ml
Sn (40ug/ml)	0.25 ml
ICUS-1454 *use for TCLP's	2.0 ml / 400ml

11.1.4 Matrix Spike (MS) and Matrix Spike Duplicate (SD): For each digestion batch of 20 samples or less, prepare one sample in triplicate. Analyze one aliquot and fortify two aliquots with the same spiking solutions as listed in 11.1.3 for the BS. These three samples are Sample, MS and SD.

11.1.5 Matrix Duplicate (MD): For each digestion batch of 20 samples or less, prepare one sample in triplicate and fortify one aliquot with the spiking solutions indicated above for the MS. Analyze the other two aliquots. These three samples are treated as sample, MD and MS. MD is not routinely prepared. It is only done on the basis of the clients' requests.

11.1.6 Digestion Procedure:

11.1.6.1 Transfer a 50 ml representative aliquot of the well mixed sample to a 50 ml digestion cup.

11.1.6.2 Add 3.0 ml of concentrated HNO_3.

11.1.6.3 Place the cup on a hot block (sample temperature 95±3°C) and cautiously evaporate to a low volume of approximately 5 ml, making certain that the sample does not boil and that no portion of the bottom of the digestion cup is allowed to go dry.

11.1.6.3.1 NOTE: If a sample is allowed to go to dryness, low recoveries will result. Should this occur, discard the sample and re-prepare.

11.1.6.3.2 NOTE: If samples are evaporated unevenly, reagent water might be added to bring all samples to the same volume and continue the evaporation. This note is also applicable to other evaporation steps.

11.1.6.4 Continue heating, adding additional HNO_3 (3 ml as an example) as necessary, until the digestion is complete (generally indicated when the digestate is light in color or does not change in appearance with continued refluxing).

11.1.6.5 Evaporate to a low volume (approximately 5 ml), not allowing any portion of the bottom of the digestion cup to go dry. Cool the digestion cup.

11.1.6.6 Add 5.0 ml 1:1 HCL (equal parts of concentrated Hydrochloric acid and blank water)

11.1.6.7 Reflux for an additional 15 minutes to dissolve any precipitate or residue resulting from evaporation.

11.1.6.8 Wash down the cup walls. Bring to final volume of 50 ml with reagent water.

11.1.6.9 Turbid samples are filtered with 2 µm Teflon filters or .45um membrane disk filters

11.2 Calibration

11.2.1 The Environmental Express digestion cups are Class-A-calibrated. The certificates are kept in digestion lab. The lot number of the cups used is recorded in the comment section on the digestion log. Each lot is verified at the 50ml final volume mark.

11.2.2 Analytical balances should be checked and calibrated using NIST Class "1" Certified weights (See SOP BF-GP-002) daily. These weights are recorded in a weight calibration logbook stored in the digestion laboratory.

11.2.3 Hot block temperatures are to be checked daily and documented in the digestion hot block temp logbook. The Hot block temperature is verified by measuring the temperature of a tube of reagent water placed in the apparatus

11.2.3 Pipettes /Eppendorf's are verified daily and calibrated quarterly by a delivery of reagent water on a Certified Balance (See SOP BF-GP-001). These results are entered into a QA approved spreadsheet; copies of these spreadsheets are in the digestion laboratory.

11.3 Sample Analysis

11.3.1 Refer to SOP BF-ME-009.
:

12.0 Calculations / Data Reduction: NA

13.0 Method Performance

13.1 Method Detection Limit Study (MDL)

13.1.1 On an annual basis, Method Detection Limit studies are performed in accordance with 40 CFR 136, Appendix B.

13.2 Demonstration of Capabilities

13.2.1 Initial Demonstration of Capability is done once per method upon the completion of training. This allows the analyst to perform the method without trainer supervision.

13.2.2 Continuing Demonstration of Capability is done once a year per method. This ensures that the analyst has remained proficient in performing the method and no retraining is necessary.

13.3 Training Requirements: Refer to QA Manual for specifics.

14.0 Pollution Control

14.1 All samples, reagents, and laboratory wastes must be handled with caution. Appropriate safety measures should be employed as detailed in Test America's Laboratory Safety Manual and Chemical Hygiene Plan. All waste will be disposed of in accordance with Federal, State and Local regulations, where reasonably feasible, technological changes have been implemented to minimize the potential for pollution of the environment. Employees will abide by this method and the policies in section 13 of the Corporate Safety Manual for "Waste Management and Pollution Prevention."

15.0 Waste Management

15.1 Waste management practices are conducted consistent with all applicable rules and regulations. Excess reagents, samples and method process wastes are disposed of in an accepted manner. Waste description rules and land disposal restrictions are followed. Waste disposal procedures are incorporated by reference to TestAmerica Buffalo's Laboratory Safety Manual. The following waste streams are produced when this method is carried out.

15.1.1 All acidic waste generated should be disposed of as HNO3 waste in an "AN" waste container. All laboratory wastes and used samples must be disposed in an "AN" waste container as detailed in TestAmerica's Laboratory Safety Manual, Chemical Hygiene Plan, and SOP AWM-HazMg-01.

16.0 References/Cross References

16.1 *Method 3010A*; Test Methods for Evaluating Solid Waste, Physical/Chemical Methods; SW846, Third Edition; 9/86 with all applicable updates (I-7/92; II-9/94; IIA-8/93; IIB-1/95; III-12/96; IIIA-4/98)

17.0 Method Modifications: NA

18.0 Attachments

Table 1:	ICP-AES Spikes
Diagram 1:	Digestion log

19.0 Revision History

- Revision 0, dated June 25, 2008
    - Integration for TestAmerica operations
    - Quality Manager change, signature updated

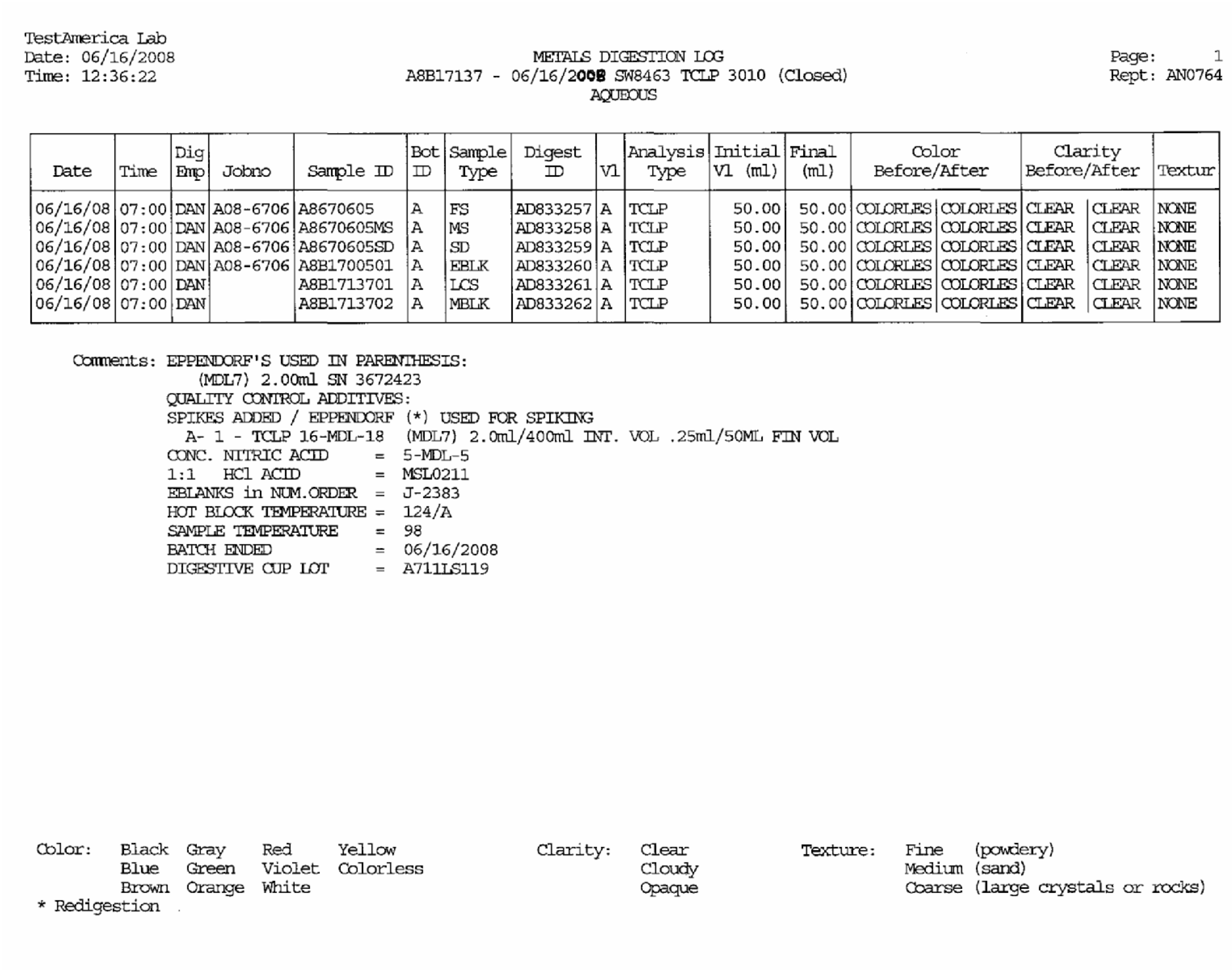
- Revision 1, dated February 8, 2010
    - Changed Spike standard preparation for Silver (Ag) and Tin (Sn) to reflect currently used procedures. Sections 8.6.1 and 8.6.2
    - Changed Method Blank/Prep Blank (MB, PB) to Method Blank (BLK). Sections 10.1.1 and 11.1.2
    - Changed Laboratory Fortified Blank (LFB) to Blank Spike (BS). Sections 10.1.2, 11.1.3, 11.1.4
    - Section 11.2.3: Changed weekly verification to daily

Attachment 1

Table 1: Spike Solutions and Final Concentrations for ICP-AES

Analyte	**ICUS-1370** (μg/mL)	***ICUS-574*** (μg/mL)	10 ug/mL Ag Stock (ug/mL)	10 μg/mL Sn Stock (μg/mL)	**ICUS-1454** *(ug/mL)*	Final Conc. In Digestate if using **ICUS-1454** (ug/mL)	Final Conc. In Digestate if using **ICUS-1370** and **ICUS-574** (ug/mL)
Aluminum		2000					10
Antimony	40				200	1.0	0.2
Arsenic	40				200	1.0	0.2
Barium		40			200	1.0	0.2
Beryllium	40				200	1.0	0.2
Boron		40					0.2
Cadmium	40				200	1.0	0.2
Calcium	2000						10
Chromium	40				200	1.0	0.2
Cobalt	40				200	1.0	0.2
Copper	40				200	1.0	0.2
Iron	2000						10
Lead	40				200	1.0	0.2
Magnesium	2000						10
Manganese	40				200	1.0	0.2
Molybdenum	40				200	1.0	0.2
Nickel	40				200	1.0	0.2
Potassium		2000					10
Selenium	40				200	1.0	0.2
Silver			10		200	1.0	0.05
Sodium		2000					10
Thallium	40				200	1.0	0.2
Tin				40			0.2
Vanadium	40				200	1.0	0.2
Zinc	40				200	1.0	0.2
Titanium	40						0.2

Attachment 2. Diagram 1: Digestion Log



TestAmerica Lab
Date: 06/16/2008
Time: 12:36:22

METALS DIGESTION LOG
A8B17137 - 06/16/2008 SW8463 TCLP 3010 (Closed)
AQUEOUS

Page: 1
Rept: AN0764

Date	Time	Dig Emp	Jobno	Sample ID	Bot ID	Sample Type	Digest ID	V1	Analysis Type	Initial V1 (ml)	Final (ml)	Color Before/After		Clarity Before/After		Textur
06/16/08	07:00	DAN	A08-6706	A8670605	A	FS	AD833257	A	TCLP	50.00	50.00	COLORLES	COLORLES	CLEAR	CLEAR	NONE
06/16/08	07:00	DAN	A08-6706	A8670605MS	A	MS	AD833258	A	TCLP	50.00	50.00	COLORLES	COLORLES	CLEAR	CLEAR	NONE
06/16/08	07:00	DAN	A08-6706	A8670605SD	A	SD	AD833259	A	TCLP	50.00	50.00	COLORLES	COLORLES	CLEAR	CLEAR	NONE
06/16/08	07:00	DAN	A08-6706	A8B1700501	A	EBLK	AD833260	A	TCLP	50.00	50.00	COLORLES	COLORLES	CLEAR	CLEAR	NONE
06/16/08	07:00	DAN		A8B1713701	A	LCS	AD833261	A	TCLP	50.00	50.00	COLORLES	COLORLES	CLEAR	CLEAR	NONE
06/16/08	07:00	DAN		A8B1713702	A	MBLK	AD833262	A	TCLP	50.00	50.00	COLORLES	COLORLES	CLEAR	CLEAR	NONE

```
Comments: EPPENDORF'S USED IN PARENTHESIS:
             (MDL7) 2.00ml SN 3672423
          QUALITY CONTROL ADDITIVES:
          SPIKES ADDED / EPPENDORF (*) USED FOR SPIKING
            A- 1 - TCLP 16-MDL-18  (MDL7) 2.0ml/400ml INT. VOL .25ml/50ML FIN VOL
          CONC. NITRIC ACID       =  5-MDL-5
          1:1   HCl ACID          =  MSL0211
          EBLANKS in NUM.ORDER    =  J-2383
          HOT BLOCK TEMPERATURE   =  124/A
          SAMPLE TEMPERATURE      =  98
          BATCH ENDED             =  06/16/2008
          DIGESTIVE CUP LOT       =  A711LS119
```

```
Color:    Black  Gray    Red     Yellow          Clarity:   Clear          Texture:   Fine    (powdery)
          Blue   Green   Violet  Colorless                  Cloudy                    Medium  (sand)
          Brown  Orange  White                              Opaque                    Coarse  (large crystals or rocks)
* Redigestion
```

Title: TOC Lloyd Kahn Method

Approval Signatures:

William S. Cicero Laboratory Director	Jessica Holzschuh Department Manager
Kirstin McCracken Kirstin L. McCracken Quality Assurance Manager	Bryce E. Stearns Technical Director
Dan Helfrich Health & Safety Coordinator	

Approval Date: March 17, 2010

Facility Distribution No. Electronic **Distributed To: Facility Intranet**

The controlled copy of this SOP is the PDF copy of the SOP that is posted to the laboratory's SOP Directory. Printed copies of this SOP or electronic copies of this SOP distributed outside the facility are considered uncontrolled.

1.0 Scope and Application

This SOP describes the laboratory procedure for the determination of total organic carbon (TOC) and black carbon in soils, sediments and other solids.

The procedure for TOC in soils and sediments is provided in the main body of this SOP. The procedure for the determination of TOC in marine sediment high in inorganic carbon is provided in Appendix B and the procedure for black carbon is provided in Appendix D.

1.1 Analytes, Matrix(s), and Reporting Limits

This procedure may be used to determine percent dry weight in soil and solid materials.

The routine reporting limit is 1000 mg/kg based on an initial sample weight of 10 mg. Additional weight of sample may be used (up to 25 mg) to achieve as low a reporting limit as 500 mg/kg.

2.0 Summary of Method

A 10 mg aliquot of sample is transferred to a tin capsule, treated with phosphoric acid and dried in an oven at a temperature 105°C for 30 minutes to one hour in order to separate the organic carbon from inorganic carbonates and bicarbonates. The sample is analyzed on an instrument where it is pyrolyzed in an inductive type furnace. The carbon is converted to carbon dioxide and measured by a differential thermal conductivity detector.

This procedure is based on the following reference documents:

- EPA Region II Document Determination of Total Organic Carbon in Sediment, July 27, 1998, authored by Lloyd Kahn, Quality Assurance Specialist.

- Dixon, Wilfrid J., and Massey, Frank J. Jr.: Introduction to Statistical Analysis (fourth edition). Edited by Wilfrid J. Dixon. McGraw-Hill Book Company, New York, 1983. P377 and P548.

If the laboratory's SOP has been modified from the above referenced document, a list of modifications is provided in Section 16.0 of this SOP.

3.0 Definitions

A list of general laboratory terms and definitions are provided in Appendix A.

4.0 Interferences

Volatile organics in the sediments may be lost in the decarbonation step resulting in a low bias.

Maintaining the sample at 4°C, analyzing the sample within the specified holding time, and analyzing the wet sample, may minimize bacterial decomposition and volatilization of the organic compounds.

5.0 Safety

Employees must abide by the policies and procedures in the Corporate Environmental Health and Safety Manual (CW-E-M-001) and this document. This procedure may involve hazardous material, operations and equipment. This SOP does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of the method to follow appropriate safety, waste disposal and health practices under the assumption that all samples and reagents are potentially hazardous. Safety glasses, gloves, lab coats and closed-toe, nonabsorbent shoes are a minimum.

5.1 Specific Safety Concerns or Requirements

None

5.2 Primary Materials Used

Table 1 lists those materials used in this procedure that have a serious or significant hazard rating along with the exposure limits and primary hazards associated with that material as identified in the MSDS. The table does not include all materials used in the procedure**.** A complete list of materials used can be found in section 7.0. Employees must review the information in the MSDS for each material before using it for the first time or when there are major changes to the MSDS. Any questions regarding the safe handling of these materials should be directed to the laboratory’s Environmental Health and Safety Coordinator.

6.0 Equipment and Supplies

- Drying Oven: Capable of maintaining a temperature of 105 ±2°C.
- Carlo Erba Elemental Analyzer Model EA1108 and Model NA 1500 or equivalent.
- Costech Elemental Analyzer: Model 4010 or equivalent.
- Analytical Balance: Capable of weighing to the nearest 0.001mg.
- Aluminum Weigh Boats.
- Tweezers
- 5mm X 9mm tin capsules
- Quartz Columns: Costech Analytical or equivalent.
- Quartz wool: for segregating and containing column materials
- Copper Wire, Reduced: Costech Analytical or equivalent.
- Tungsten on Alumina: Costech Analytical or equivalent.
- High Temperature Gloves
- Clear Plastic Sample Trays: Costech Analytical or equivalent.

7.0 Reagents and Standards

7.1 Reagents

- Reagent water
- Phosphoric Acid, Concentrated: Reagent Grade, J.T. Baker recommended.

Phosphoric Acid Solution (1:19): Add approximately 100 mL of reagent water to a 200 mL volumetric flask. Add 18.34 g of concentrated phosphoric acid to the volumetric flask then adjust to volume with reagent water. Mix the solution well then transfer the solution to a 250 mL polyethylene bottle. Assign an expiration date of six months from date made and store the solution at room temperature.

7.2 Standards

- Acetanilide Crystals of known Carbon percentage: Purchased from Costech Analytical. Used to check instrument calibration.
- Sulfanilamide Crystals (41.84% Carbon): Purchased from Costech Analytical. This material is used to calibrate the instruments.
- Laboratory Control Samples (LCS) Material, Organic Material of known Carbon percentage: Purchased from LECO Corporation.
- Matrix Spike Material, 1632B trace elements in coal (76.86% Carbon)

8.0 Sample Collection, Preservation, Shipment and Storage

The laboratory does not perform sample collection so sampling procedures are not included in this SOP. Sampling requirements may be found in the published reference method.

Listed below are the recommended minimum sample size, preservation and holding time requirements:

Matrix	Sample Container	Minimum Sample Size	Preservation	Holding Time[1]	Reference
Solids	Amber glass	10 g	Chilled to ≤ 4°C	14 Days	TOC by Lloyd Kahn

[1] Holding time is determined from date of collection.

Unless otherwise specified by client or regulatory program, after analysis, samples and extracts are retained for a minimum of 30 days after provision of the project report and then disposed of in accordance with applicable regulations.

9.0 Quality Control

9.1 Sample QC

The laboratory prepares the following quality control samples with each batch of samples.

QC Item	Frequency	Acceptance

		Criteria
Method Blank (MB)	1 in 20 or fewer samples	< RL
Laboratory Control Sample (LCS)	1 in 20 or fewer samples	%R (85-115)
Sample Duplicate (DP)	Client Request	RPD (≤ 20)
Matrix Spikes (MS)	Client Request	%R (85-115)

9.2 Instrument QC

The laboratory analyzes the following instrument check standards:

QC Item	Frequency	Acceptance Criteria
Initial Calibration (ICAL)	Initial Method Set-Up, after combustion chamber is changed (approx. every 200 drops)	Correlation coefficient must be >0.995
Calibration Verification (Acetanilide)	Every 20 drops and at the end of the analytical sequence	%R (85-115)
Calibration Blank (CCB)	After every acetanilide	<RL

10.0 Procedure

10.1 Calibration

Analyze a calibration curve each time the combustion column is changed. Change the column after 200 drops or when you experience result issues or odd peak shapes or baseline issues. The column change procedure is provided in Appendix C.

The recommended formulations for each calibration level are provided in the following table:

Calibration Standard Sulfanimide	Weight[1] (mg)	% Carbon	Carbon (mg)
Calibration Level 1	0.100	41.84	0.0418
Calibration Level 2	0.500	41.84	0.2092
Calibration Level 3	1.00	41.84	0.4184
Calibration Level 4	1.50	41.84	0.6276
Calibration Level 5	1.75	41.84	0.7322

[1]These weights are approximate. Enter the actual weight used into the software program.

Measure a single drop for each calibration point. The instrument software system plots peak area against mg of Carbon and calculates a correlation coefficient using standard linear regression. The correlation coefficient (r) must be $\geq$ 0.995 for the calibration to be considered acceptable. If it is not, repeat the calibration prior to further analysis.

1.0 Troubleshooting

- Calibration passes at > 0.995 correlation, but LCS fails abnormally low: Re-calibrate. Calibration usually needs to be > 0.999 correlation.

- Carbon peak “maxes out” at instrument 1200mv (peak has flat top): Reanalyze sample at lower weight.

- No peaks on any chromatograms, no results: Gases to instrument may be off. Turn on all gasses at valve manifold.

- Autosampler will not work at all: Gasses to instrument may be off. Turn on all gasses at valve manifold.

- Single chromatogram shows results at bottom of page, but no peak or baseline in chromatogram window: Re-print single chromatogram.

- Some or all chromatograms show carbon peak at same retention time as Acetanilide, but peak is not identified as carbon, or is identified as another element: Retention time shifted. Adjust retention time in calibration window, and reprint chromatograms.
- Upon recalibration, peaks are not being identified as carbon: In calibration window, general tab, adjust retention time to match peaks. Starting at level 1, “Open Standard”, open level1 curve pt. in calibration directory, click “Add Peak” button, click on peak itself. Increase level #, opening standard for each curve pt and add each peak. Carbon Tab should have all five calibration points on curve, if done correctly.
- Peaks in chromatograms identified as carbon, but all results in summary table below chromatogram are zero: Current calibration not associated with run when started. Open current calibration, copy first two columns for all points (5 rows) in small table in general tab. Then, open calibration that was associated with run (should be empty) and paste into table in calibration tab. Reprint all chromatograms on run.
- Software crashes during analysis: Boot up software normally. Chromatograms already printed/analyzed are ok, but, sample that was analyzing during shutdown is lost. Restart table at next sample by un-checking “run” box for samples already run and sample that was lost.

- Autosampler error causes few samples to remain in autosampler tray after run has finished: Identify samples that got stuck. Create a new run and analyze stuck samples (with initial weights) with bracketing QC. No PBS/LCS needed.
- Autosampler error causes many sequential samples to remain in autosampler tray after run has finished (usually end of run): Add rows onto existing table. Identify samples that did not get analyzed and repeat Ids and weights into added rows. Restart table. All analyzed samples’ status should be blue (analyzed), added rows should be green (not analyzed yet).
- Various result issues or odd peak shapes or baseline issues: Column may be leaking or cracked. Change column, recalibrate.

10.3 Sample Preparation

Using tweezers, and working directly from the box, place a tin capsule on the analytical balance and tare the balance. Using the small sample scoop, add approximately 10 mg (or the project specified sample weight) of sample to the capsule. Record the actual sample weight used on sample preparation log. Remove the capsule from the balance and place into one of the aluminum holding trays. Weigh two additional portions of sample into two separate tin capsules for each field sample.

To prepare the method blank, set two empty tin capsules into an aluminum holding tray.

To prepare the LCC, weigh ~9 mg of the LECO LCS material into two separate tin capsules and set them in sequence in an aluminum holding tray.

For the matrix spike, weigh out an additional sample aliquot and record its weight. Add 0.3 – 0.7 mg of matrix spike material and record this weight.

For the sample duplicate, weigh out an additional sample aliquot. Prepare two aliquots for both the matrix spike and the sample duplicate.

Add two drops of 1:19 phosphoric acid to each tin capsule. Place the aluminum trays into a drying oven set to a temperature of 105ºC for 30-60 minutes or until all samples appear dry.

Using tweezers pinch the top of each tin capsule closed and compress the capsule around the material inside. Work carefully so as not to tear the capsule, but crush it down to the smallest size. Set the prepared samples in line in a clear plastic sample tray for storage, or place directly into an autosampler tray for analysis. For the latter, leave positions open for the acetanilide check standards and associated calibration blanks.

Prepare the acetanilide standard and blanks as follows:

For each acetanilide spike, weigh ~0.5 mg of acetanilide material into a tin capsule. Fold the capsule up and compress down to the smallest size possible. Prepare enough acetanilide to ensure a frequency of every 20 drops and the end of the analytical sequence. For each associated calibration blank, leave an empty position in the autosampler tray.

Software Set-up and Analysis

If the column has been changed generate a new calibration curve. If not, use the existing calibration curve for analysis. Each column will analyze approximately 200 individual sample drops. When the counter on the instrument approaches 200, watch the instrument data for signs that the column is deteriorating; poor peak resolution, trailing baselines, extraneous peaks. If a column change is necessary, refer to Appendix C for the procedure. After changing the column, generate a new calibration curve.

Select the appropriate channel: Channel 1 is the NA 1500, Channel 2 is the EA 1108, and Channel 3 is the Costech instrument, which has its own PC. At the main screen select the sample table icon. The last sample table that was run will be shown on the screen.

Open a new sample table, and select the appropriate number of sample positions for the analysis, then name the table with the date and a unique alpha designator (i.e. 061505a). In front of the %3r in the file name column of the sample table, add the sample table name to ensure that each individual chromatogram generated from this sample table has a unique filename associated with it.

If the combustion column has been changed and instrument needs to be calibrated, follow the procedure below:

Prepare a “bypass” drop to determine the retention time for carbon with the new column. The bypass is an aliquot of acetanilide. The weight is not needed. Drop the bypass into the instrument and initiate a singular analysis. Set the retention time for carbon in the software to match that of the bypass drop.

Identify the first five sample lines with the names Std1 through Std 5. Enter their respective weights in the weight column, assign them a level # in the level column (Std1 is level 1, Std2 is level 2, etc.) to alert the software the order in which to place the calibration standards. In the sample type column, use the drop down and select “standard” for each. Finally, use the drop down in the Standard name column and select “sulfanilamide” for each. Add the standards to the autosampler tray and hit “start” to run the calibration.

Sample Analysis:

Open a new sample tray and create a unique file name. When the instrument was last calibrated, the software creates a calibration file with the same name as the sample table in which it was run. Open this file and save it with the same name as the sample table about to be run to ensure that the analysis is calculated from the most recent calibration. To do this, click on the calibration icon (looks like a little calibration curve) and use the file option to open the calibration file last performed. Save this file with the same name as your sample table. Click on the sample table icon (looks like a little sample table) to get back to your sample table.

Enter each sample ID and their respective weights and save the sample table. Enter a weight of 10 mg for the Method Blank (PBS) and instrument blanks.

An example analytical sequence follows:

Initial Calibration (calibration blank and 5 calibration standards)

Acetanilide	(1 drop)
Blank	(1 drop)
PBS	(2 individual drops)
LCS	(2 individual drops)
Sample	(2 individual drops)
Sample	(2 individual drops)
Sample	(2 individual drops)
Sample	(2 individual drops)
Sample	(2 individual drops)
Sample	(2 individual drops)
Sample	(2 individual drops)
Sample	(2 individual drops)
Acetanilide	(1 drop)
Blank	(1 drop)

Add the samples and acetanilides to the autosampler tray and set the tray into the autosampler carriage. Turn the autosampler tray until the number 1 position is behind the post, in front of the autosampler. The tray is now set to run.

Click the “start” icon to begin the analysis.

After analysis review the analytical results against the acceptance criteria given in Table 2, Section 18.0, and perform corrective action as necessary. Report results in mg/kg Carbon and corrected for % solids.

11.0 Calculations / Data Reduction

11.1 Calculations

11.2 Percent Carbon to mg/kg Carbon Conversion

% Carbon × 10,000 = mg/kg Carbon

11.3 LCS Percent Recovery (%R)

$$\%R = \frac{\text{LCS Result}}{\text{LCS True Value}} \times 100$$

11.4 MS Percent Recovery (%R)

$$\text{mg/Kg wet SA} = \frac{\text{Spike TV} \times \text{weight of MS added}}{\text{sample weight}} \times 1\,\text{million}$$

$$\text{mg/Kg dry SA} = \frac{\text{mg/Kg wet SA}}{\%\,\text{solid}} \times 100$$

$$\text{mg/Kg dry Carbon} = \frac{\text{mg/Kg wet Carbon (from instrument)}}{\%\,\text{solid}} \times 100$$

$$\%R = \frac{A - B}{C} \times 100$$

Where:
A= Average of two drops of MS sample result: mg/Kg dry carbon
B= Average of two drops of parent sample: mg/Kg dry carbon
C= Average of two drops of mg/Kg dry SA
SA= spike added (mg/Kg)
Spike TV= 0.7686 (mg/Kg)

11.5 Relative Percent Difference (RPD)

$$\text{RPD} = \frac{|D_1 - D_2|}{\frac{D_1 + D_2}{2}} \times 100$$

Where:

D_1 = First Sample Value

D_2 = Second Sample Value (duplicate)

11.6 Dixon Test (Use 3-7 results)

1. Sort all the results in ascending order (low values to high).
2. Calculate the tau statistic for the low and high values.
3. Compare the calculated tau statistics (low and high) to critical values listed below.
4. If either calculated tau is higher than the critical value, reject that value and repeat the test.

Tau statistic for lowest value = $\tau_L = (X_2 - X_1) / (X_k - X_1)$
Tau statistic for highest value = $\tau_H = (X_k - X_{k-1}) / (X_k - X_1)$

Where:
X_2 = Second lowest value in sorted list.
X_1 = Lowest value in sorted list.
X_k = Highest value in sorted list.
X_{k-1} = Second highest value in sorted list.

Number of observations, k	Critical Values
3	0.941
4	0.765
5	0.642
6	0.560
7	0.507

11.2 Data Review

11.2.1 Primary Data Review

Evaluate and QC samples against the acceptance criteria given in Table 2. Perform the recommended corrective action as necessary. If corrective action is not taken or is not successful, initiate a nonconformance report (NCR) to document the situation.

11.2.2 Secondary Data Review

Spot-check the calculations using the equations given in Section 11.1.

Verify that the performance criteria for the QC items listed in Table 2 were met. If the results do not fall within the established limits verify the recommended corrective actions were performed. If corrective action was not taken or is unsuccessful, ensure the situation is documented with a nonconformance report (NCR) and ensure data is qualified accordingly. Report the nonconformance in the narrative note program.

11.3 Data Reporting

Report analytical results above the adjusted reporting limit (RL) as the value found. Report analytical results less than the adjusted RL with a “U” data qualifier. Adjust the RL for sample dilution/concentration and dry weight. The laboratory’s routine reporting limit (RL) for TOC is 1000 mg/Kg based on a 10.0 mg sample mass, assuming 100% solids. Unless otherwise specified for the project, report all soils in dry weight.

Review project documents such as the environmental test request (ETR) analytical worksheets, Project Plan (PP), Project Memo or any other document/process used to communicate project requirements to ensure those project requirements were met. If project requirements were not met, immediately notify the project manager (PM) to determine an appropriate course of action.

Generate the data report in the deliverable format specified by the laboratory PM and release the report to report management.

Retain, manage and archive electronic and hardcopy data as specified in laboratory SOP BR-QA-014 Laboratory Records.

12.0 **Method Performance**

12.1 Method Detection Limit Study (MDL)

Perform a method detection limit (MDL) study at initial method set-up following the procedures specified in laboratory SOP BR-QA-005.

12.2 Demonstration of Capabilities (DOC)

Perform a method demonstration of capability at initial set-up and when time there is a significant change in instrumentation or procedure.

Each analyst that performs this procedure must complete an initial demonstration of capability (IDOC) prior to independent analysis of client samples. Each analyst must demonstrate on-going proficiency (ODOC) annually thereafter. DOC procedures are further described in the laboratory’s quality system manual (QAM) and in the laboratory SOP for employee training, BR-QA-011.

12.3 Training Requirements

Any employee that performs any portion of the procedure described in this SOP must have documentation in their employee training file that they have read this version of this SOP.

Instrument analysts, prior to independent analysis of client samples, must also have documentation of demonstration of initial proficiency (IDOC) and annual on-going proficiency (ODOC) in their employee training files.

13.0 **Pollution Control**

It is TestAmerica’s policy to evaluate each method and look for opportunities to minimize waste generated (i.e., examine recycling options, ordering chemicals based on quantity needed, preparation of reagents based on anticipated usage and reagent stability). Employees must abide by the policies in Section 13 of the Corporate Safety Manual for “Waste Management and Pollution Prevention.”

14.0 Waste Management

Waste management practices are conducted consistent with all applicable rules and regulations. Excess reagents, samples and method process wastes are disposed of in an accepted manner. Waste description rules and land disposal restrictions are followed. Waste disposal procedures are incorporated by reference to BR-EH-001 *Hazardous Waste*.

The following waste streams are produced when this method is carried out.

- Caustic waste – 2.5 L glass satellite container.
- Acidic Waste - 2.5L glass satellite container

The satellite containers are labeled "Hazardous Waste" along with the type of waste category generated. Authorized personnel routinely transfer the contents of the satellite containers to the hazardous waste storage room for future disposal in accordance with Federal, State and Local regulations.

15.0 References / Cross-References

- EPA Region II Document Determination of Total Organic Carbon in Sediment, July 27, 1998, authored by Lloyd Kahn, Quality Assurance Specialist.
- Dixon, Wilfrid J., and Massey, Frank J. Jr.: Introduction to Statistical Analysis (fourth edition). Edited by Wilfrid J. Dixon. McGraw-Hill Book Company, New York, 1983. P377 and P548.
- Corporate SOP CW-E-M-001 Corporate Environmental Health and Safety Manual
- Laboratory SOP BR-QA-005, Procedures for the Determination of Limits of Detection (LOD), Limits of Quantitation (LOQ) and Reporting Limits (RL).
- Laboratory SOP BR-QA-011 Employee Training
- Laboratory SOP BR-EH-011 Hazardous Waste
- Laboratory SOP BR-QA-014 Laboratory Records
- Laboratory Quality Assurance Manual (QAM)

16.0 Method Modifications

The laboratory procedure is modified from the reference method as follows:

Modification Number	Method Reference	Modification
1	TOC by Lloyd Kahn	The laboratory analyzes two drops per sample and if the RPD is greater than 40% the Dixon test is utilized.

17.0 Attachments

- Table 1: Primary Materials Used
- Table 2: QC Summary & Recommended Corrective Action
- Appendix A: Terms and Definitions
- Appendix B: TOC Procedure for High Concentration Marine Sediments (CITHON)
- Appendix C: Column change procedure
- Appendix D: Determination of Black Carbon in Sediment Procedure

18.0 Revision History

BR-WC-008, Rev. 12:

- Section 11.2: The procedure for evaluating data using the low-level spreadsheet was removed.

Table 1: Primary Materials Used

Material (1)	Hazards	Exposure Limit (2)	Signs and symptoms of exposure
Phosphoric Acid	Corrosive	1 Mg/M3 TWA	Inhalation is not an expected hazard unless misted or heated to high temperatures. May cause redness, pain, and severe skin burns. May cause redness, pain, blurred vision, eye burns, and permanent eye damage.
1 – Always add acid to water to prevent violent reactions.			
2 – Exposure limit refers to the OSHA regulatory exposure limit.			

Table 2: QC Summary, Frequency, Acceptance Criteria and Recommended Corrective Action

QC Item	Frequency	Acceptance Criteria	Recommended Corrective Action[1]
ICAL	Following each column change	correlation coefficient $\geq$ 0.995	Standards check, re-calibration
Acetanilide	Every 20 drops and at the end of the analytical run	%R (85-115)	Re-prepare and reanalyze samples not bracketed by passing standard.
Blank (paired with Acetanilide)	Following each Acetanilide	< RL	Re-prepare and reanalyze batch.
Method Blank (MB)	Once per batch of 20 samples	< RL DoD: ½ RL	Re-prepare and reanalyze batch.
LCS	Once per batch of 20 samples	%R (75-125)	Re-prepare and reanalyze batch.
Sample Duplicate (DP)	One per batch of 20 or less samples	RPD (≤ 20)	Discuss outlier in project narrative
MS/MSD	One per batch of 20 or less samples	%R (75-125)	Discuss outlier in project narrative
Sample precsion	Each sample is run in duplicate	%RPD<40%	Analyze 2 more replicates and perform Dixon test for high and low outliers. Include Dixon spreadsheet in the data package and narrative note results.

[1] The recommended corrective action may include some or all of the items listed in this column. The corrective action taken may be dependent on project data quality objectives and/or analyst judgment but must be sufficient to ensure that results will be valid. If corrective action is not taken or is not successful, data must be flagged with appropriate qualifiers.

Appendix A: Terms and Definitions

Batch: environmental samples, which are prepared and/or analyzed together with the same process, using the same lot(s) of reagents. A preparation/digestion batch is composed of one to 20 environmental samples of similar matrix, meeting the above criteria.

Calibration: the establishment of an analytical curve based on the absorbance, emission intensity or other measured characteristic of known standard.

Calibration Standards: a series of known standard solutions used to calibrate the instrument response with respect to analyte concentration. A standard containing the analyte in question (sulphanilimide) is prepared at varying weights and analyzed. This standard is a separate source from the LCS. The sulphanilimide is used to calibrate the instrument response with respect to analyte concentration.

Demonstration of Capability (DOC): procedure to establish the ability to generate acceptable accuracy and precision.

Holding Time: the maximum time that a sample may be held before preparation and/or analysis as promulgated by regulation or as specified in a test method.

Laboratory Control Sample (LCS): a blank matrix spiked with a known amount of analyte(s) processed simultaneously with and under the same conditions as samples through all steps of the procedure.

Matrix Duplicate (DP): duplicate aliquot of a sample processed and analyzed independently; under the same laboratory conditions; also referred to as Sample Duplicate.

Method Blank (MB): a blank matrix processed simultaneously with and under the same conditions as samples through all steps of the procedure. Also known as the preparation blank (PB).

Non-conformance: an indication, judgment, or state of not having met the requirements of the relevant specification, contract or regulation.

Preservation: refrigeration and/or reagents added at the time of sample collection to maintain the chemical, physical, and/or biological integrity of the sample.

Reporting Limit (RL): the level to which data is reported for a specific test method and/or sample.

Appendix B: Marine Sediments High in Inorganic Carbon

Sample Preparation

Transfer approximately 10 g of a thoroughly mixed sample to an aluminum weigh dish, and dry in the 105°C oven. Grind the sample with the pink mortar and pestle to a fine powder. Record the weight of a 250 mL Teflon beaker then transfer ~ 5 g of the ground sample to this beaker.

If the sample is to be spiked, weigh the beaker to the nearest 0.1mg and record the weight. Likewise determine and record the weight of the added sample. Add 0.1g of NIST 1632b Trace Elements in Coal (80.11% Carbon) to the sample. Record the weight added. Evenly distribute the spike over the sample and use a glass stir rod to mix the spike with the sample. Do not use that stir rod with any other sample.

Use Talc-free latex gloves from this point on to minimize the risk of acid burns. Add several drops of 1:1 HCL to each sample and stir each sample with its own glass stir rod. Carefully rinse the stir rod and beaker walls with DI water using a fine-tipped squirt bottle. Use only what is needed to bring the entire sample to the bottom of the beaker. ***When adding water to acid use necessary precautions to avoid splashing!*** Samples with high concentrations of inorganic carbon may effervesce to the point of overflowing the beaker, so take care to add the acid in small aliquots and stir vigorously. If the sample “boils over” it must be re-prepared. Continue to add 1:1 HCL in small aliquots until there is no further reaction, taking sample to dryness after each addition of acid in a 105-degree oven.

Dry the treated samples in the oven after each acid/water addition. Do not add more than a total of 200 mL of 1:1 HCL to any sample.

NOTE*: Samples are hydroscopic and will absorb water if they are exposed to air for too long.*

Weigh beaker with residue and record the residue weight measurement. After the sample is thoroughly dry, scrape the sample residue from the beaker and grind to a powder using the pink mortar and pestle. Transfer the ground sample to a clean, dry 40-mL vial reserved for this analysis.

NOTE: *Depending on the nature of the sample, it may be difficult to completely remove the dried residue from the beaker or to grind it to a homogenous powder. Where difficulties are encountered, make a note on the preparation worksheet.*

Analysis

Perform TOC analysis on processed sample material as outlined in section 10.0 of this SOP.

Appendix C: Column Change Procedure

Turn off the helium and oxygen supplies to the instrument.

Dial the left furnace temperature to a reading of 052 (this equates to 520˚C). Wait until the temperature drops below 600˚C to remove the column.

Remove the panel covering the furnace and unscrew the autosampler connection from the top of the column.

Unscrew the fitting at the bottom of the column and remove.

Lift the column up and out of the furnace using high temperature gloves.

CAUTION: The column will still be 500-600˚C. Do not touch the center portion of the column. Place the spent column in the metal can designated for this purpose.

Lay a new quartz column on the bench top, measure and mark off for the following:

- One inch up from the bottom and add a ½ inch plug of quartz wool. Note: pack the quartz wool tightly enough for it to stay in place.
- Pour in 2 ½ inches of copper wire
- Pack another ½ inch quartz wool plug on top of the copper
- Pour in 3 inches of tungsten
- Pack a final ½ inch quartz wool plug on top of the tungsten

Place the new column into the furnace and reconnect the top and bottom fittings. Snug these up, but don’t over tighten.

Replace the panel covering the furnace, dial the furnace temperature back to 102 (this equates to 1020˚C), and turn the helium and oxygen supplies back on.

When the instrument comes up to operating temperature, it is ready to calibrate.

Appendix D: Determination of Black Carbon in Sediment Procedure

1. Obtain a representative subsample of the sediment. Weight 10 grams of sample into a clean pre-tared aluminum drying pan or equivalent.

2. Dry the sample at 105°C for at least 12 hours.

3. Grind the sample using a mortar and pestle.

4. Sieve the sample using a number 35 sieve (500 um).

5. Treat the sample with phosphoric acid. Add acid drop wise until effervescence is no longer observed.

6. Dry the sample at 105°C for 1 hour.

7. Set aside an aliquot of the sample at this stage for direct TOC analysis, reported without correction for the IN623 percent solids. Continue with the sample for Black Carbon.

8. Place the dried sample into a clean crucible and cover the sample.

9. Bake the samples at 375°C in a muffle for 24 hours or until the LCS is +/- 50% of the true value.

10. Allow the samples to cool and transfer approximately 5.0 mg into each of two tin capsules.

11. Transfer the sample (in the tin capsules) to the TOC analyzer for analysis by the Lloyd Kahn Method.

12. The sample is pyrolyzed in an inductive type furnace, where the carbon is converted to carbon dioxide, which is measured using a differential thermal conductivity detector.

13. The results will be reported as mg/Kg Black Carbon.

Note: Black carbon LCS material: NIST Standard Reference Material 1944 New York-New Jersey Waterways Sediment.

References:

Orjan Gustafsson, Thomas D. Bucherli, Zofia Kukulska, Mette Andersson, Claude Largeau, Jean-Noel Rouzaud, Christopher M. Reddy and Timothy I. Eglinton (December 2001) Evaluation of a Protocol for the Quantification of Black Carbon in Sediments, Global Biogeochemical Cycles, Volume 15, pages 881-890.

Orjan Gustafsson, Farnaz Haghseta, Charmaine Chan, John MacFarlane & Philip M. Gschwend (1997) Quantification of the Dilute Sedimentary Soot Phase: Implications for PAH Speciation and Bioavailability, Environmental Science & Technology, Volume 31, pages 203-209.

ARCADIS

Attachment 2

TestAmerica Quality Manual

Cover Page:

Quality Assurance Manual

TestAmerica Buffalo
10 Hazelwood Drive
Amherst, New York 14228
716.504.9800
716.691.7991

www.testamericainc.com

Facility Distribution No. 34 Distributed To: Jo Ann Robertson Arcadis Syracuse, NY

CONTROLLED COPY

Title Page:

Quality Assurance Manual
Approval Signatures

Laboratory Director – Chris Spencer	Date 02/09/2009
Quality Assurance Manager - Peggy Gray-Erdmann Peggy Gray-Erdmann	Date 02/01/2009
Technical Director & EH&S - Kenneth Kasperek Kenneth E. Kasperek	Date 02/05/2009
Customer Service Manager - Mark Nemec	Date 02/06/2009
Operations Manager – Jennifer Pierce Jennifer Pierce	Date 02/05/2009

SECTION 2

TABLE OF CONTENTS

Section No.	Title	Page No.	Effective Date
-	COVER PAGE	COVER	02/02/2009
1.0	TITLE PAGE	1-1	02/02/2009
2.0	TABLE OF CONTENTS	2-1	02/02/2009
3.0	INTRODUCTION	3-1	02/02/2009
3.1	Introduction And Compliance References	3-1	02/02/2009
3.2	Terms And Definitions	3-2	02/02/2009
3.3	Scope / Fields Of Testing	3-2	02/02/2009
3.4	Management Of The Manual	3-2	02/02/2009
4.0	ORGANIZATION AND MANAGEMENT *(NELAC 5.4.1)*	4-1	02/02/2009
4.1	Overview	4-1	02/02/2009
4.2	Roles And Responsibilities	4-1	02/02/2009
4.3	Deputies	4-1	02/02/2009
5.0	QUALITY SYSTEM *(NELAC 5.4.2)*	5-1	02/02/2009
5.1	Quality Policy Statement	5-1	02/02/2009
5.2	Ethics And Data Integrity	5-1	02/02/2009
5.3	Quality System Documentation	5-2	02/02/2009
5.4	Qa/Qc Objectives For The Measurement Of Data	5-3	02/02/2009
5.5	Criteria For Quality Indicators	5-5	02/02/2009
5.6	Statistical Quality Control	5-5	02/02/2009
5.7	Quality System Metrics	5-6	02/02/2009
6.0	DOCUMENT CONTROL *(NELAC 5.4.3)*	6-1	02/02/2009
6.1	Overview	6-1	02/02/2009
6.2	Document Approval And Issue	6-1	02/02/2009
6.3	Procedures For Document Control Policy	6-2	02/02/2009
6.4	Obsolete Documents	6-2	02/02/2009
7.0	SERVICE TO THE CLIENT	7-1	02/02/2009
7.1	Overview	7-1	02/02/2009
7.2	Review Sequence And Key Personnel	7-2	02/02/2009
7.3	Documentation	7-3	02/02/2009
7.4	Special Services	7-4	02/02/2009
7.5	Client Communication	7-4	02/02/2009
7.6	Reporting	7-5	02/02/2009
7.6	Client Surveys	7-5	02/02/2009
8.0	SUBCONTRACTING OF TESTS *(NELAC 5.4.5)*	8-1	02/02/2009
8.1	Overview	8-1	02/02/2009
8.2	Qualifying And Monitoring Subcontractors	8-1	02/02/2009
8.3	Oversight And Reporting	8-3	02/02/2009

Section No.	Title	Page No.	Effective Date
8.4	Contingency Planning	8-4	02/02/2009
9.0	PURCHASING SERVICES AND SUPPLIES *(NELAC 5.4.6)*	9-1	02/02/2009
9.1	Overview	9-1	02/02/2009
9.2	Glassware	9-1	02/02/2009
9.3	Reagents, Standards & Supplies	9-1	02/02/2009
9.4	Purchase Of Equipment/Instruments/Software	9-4	02/02/2009
9.5	Services	9-4	02/02/2009
9.6	Suppliers	9-4	02/02/2009
10.0	COMPLAINTS (NELAC 5.4.8)	10-1	02/02/2009
10.1	Overview	10-1	02/02/2009
10.2	External Complaints	10-1	02/02/2009
10.3	Internal Complaints	10-2	02/02/2009
10.4	Management Review	10-2	02/02/2009
11.0	CONTROL OF NON-CONFORMING WORK *(NELAC 5.4.9)*	11-1	02/02/2009
11.1	Overview	11-1	02/02/2009
11.2	Responsibilities And Authorities	11-1	02/02/2009
11.3	Evaluation Of Significance And Actions Taken	11-2	02/02/2009
11.4	Prevention Of Nonconforming Work	11-2	02/02/2009
11.5	Method Suspension/Restriction (stop work procedures)	11-3	02/02/2009
12.0	CORRECTIVE ACTION *(NELAC 5.4.10)*	12-1	02/02/2009
12.1	Overview	12-1	02/02/2009
12.2	General	12-1	02/02/2009
12.3	Closed Loop Corrective Action Process	12-2	02/02/2009
12.4	Technical Corrective Actions	12-3	02/02/2009
12.5	Basic Corrections	12-3	02/02/2009
13.0	PREVENTIVE ACTION *(NELAC 5.4.11)*	13-1	02/02/2009
13.1	Overview	13-1	02/02/2009
13.2	Management Of Change	13-2	02/02/2009
14.0	CONTROL OF RECORDS *(NELAC 5.4.12)*	14-1	02/02/2009
14.1	Overview	14-1	02/02/2009
14.2	Technical And Analytical Records	14-4	02/02/2009
14.3	Laboratory Support Activities	14-5	02/02/2009
14.4	Administrative Records	14-6	02/02/2009
14.5	Records Management, Storage And Disposal	14-6	02/02/2009
15.0	AUDITS *(NELAC 5.4.13)*	14-1	02/02/2009
15.1	Overview	15-1	02/02/2009
15.2	External Audits	15-3	02/02/2009
15.3	Audit Findings	15-3	02/02/2009
16.0	MANAGEMENT REVIEWS *(NELAC 5.4.14)*	16-1	02/02/2009
16.1	Quality Assurance Report	16-1	02/02/2009
16.2	Annual Management Review	16-1	02/02/2009

Section No.	Title	Page No.	Effective Date
16.3	Potential Integrity Related Managerial Reviews	16-2	02/02/2009
17.0	PERSONNEL *(NELAC 5.5.2)*	17-1	02/02/2009
17.1	Overview	17-1	02/02/2009
17.2	Education And Experience Requirements For Technical Personnel	17-1	02/02/2009
17.3	Training	17-2	02/02/2009
17.4	Data Integrity And Ethics Training Program	17-3	02/02/2009
18.0	ACCOMMODATIONS AND ENVIRONMENTAL CONDITIONS *(NELAC 5.5.3)*	18-1	02/02/2009
18.1	Overview	18-1	02/02/2009
18.2	Environment	18-1	02/02/2009
18.3	Work Areas	18-2	02/02/2009
18.4	Floor Plan	18-2	02/02/2009
18.5	Building Security	18-2	02/02/2009
19.0	TEST METHODS AND METHOD VALIDATION *(NELAC 5.5.4)*	19-1	02/02/2009
19.1	Overview	19-1	02/02/2009
19.2	Standard Operating Procedures	19-1	02/02/2009
19.3	Laboratory Methods Manual	19-1	02/02/2009
19.4	Selection Of Methods	19-2	02/02/2009
19.5	Laboratory Developed Methods And Non-Standard Methods	19-5	02/02/2009
19.6	Validation Of Methods	19-6	02/02/2009
19.7	Method Detection Limits (Mdl)/ Limits Of Detection (Lod)	19-7	02/02/2009
19.8	Instrument Detection Limits (Idl)	19-8	02/02/2009
19.9	Verification Of Detection And Reporting Limits	19-8	02/02/2009
19.10	Retention Time Windows	19-8	02/02/2009
19.11	Evaluation Of Selectivity	19-8	02/02/2009
19.12	Estimation Of Uncertainty Of Measurement	19-9	02/02/2009
19.14	Control Of Data	19-10	02/02/2009
20.0	EQUIPMENT (AND CALIBRATIONS) *(NELAC 5.5.5)*	20-1	02/02/2009
20.1	Overview	20-1	02/02/2009
20.2	Preventive Maintenance	20-1	02/02/2009
20.3	Support Equipment	20-2	02/02/2009
20.4	Instrument Calibrations	20-5	02/02/2009
20.5	Tentatively Identified Compounds (Tics) – Gc/Ms Analysis	20-7	02/02/2009
20.6	Gc/Ms Tuning	20-7	02/02/2009
21.0	MEASUREMENT TRACEABILITY *(NELAC 5.5.6)*	21-1	02/02/2009
21.1	Overview	21-1	02/02/2009
21.2	Nist-Traceable Weights And Thermometers	21-1	02/02/2009
21.3	Reference Standards / Materials	21-2	02/02/2009

Section No.	Title	Page No.	Effective Date
21.4	Documentation And Labeling Of Standards, Reagents, And Reference Materials	21-2	02/02/2009
22.0	SAMPLING *(NELAC 5.5.7)*	22-1	02/02/2009
22.1	22.1 Overview	22-1	02/02/2009
22.2	Sampling Containers	22-1	02/02/2009
22.3	Definition Of Holding Time	22-1	02/02/2009
22.4	Sampling Containers, Preservation Requirements, Holding Times	22-2	02/02/2009
22.5	Sample Aliquots / Subsampling	22-2	02/02/2009
23.0	HANDLING OF SAMPLES *(NELAC 5.5.8)*	23-1	02/02/2009
23.1	Chain Of Custody **(Coc)**	23-1	02/02/2009
23.2	Sample Receipt	23-2	02/02/2009
23.3	Sample Storage	23-3	02/02/2009
23.4	Hazardous Samples And Foreign Soils	23-4	02/02/2009
23.5	Sample Shipping	23-4	02/02/2009
23.6	Sample Disposal	23-4	02/02/2009
24.0	ASSURING THE QUALITY OF TEST RESULTS *(NELAC 5.5.9)*	24-1	02/02/2009
24.1	Overview	24-1	02/02/2009
24.2	Controls	24-1	02/02/2009
24.3	Negative Controls	24-1	02/02/2009
24.4	Positive Controls	24-2	02/02/2009
24.5	Sample Matrix Controls	24-4	02/02/2009
24.6	Acceptance Criteria (Control Limits)	24-4	02/02/2009
24.7	Additional Procedures To Assure Quality Control	24-7	02/02/2009
25.0	REPORTING RESULTS *(NELAC 5.5.10)*	25-1	02/02/2009
25.1	Overview	25-1	02/02/2009
25.2	Test Reports	25-1	02/02/2009
25.3	Reporting Level Or Report Type	25-3	02/02/2009
25.4	Supplemental Information For Test	25-4	02/02/2009
25.5	Environmental Testing Obtained From Subcontractors	25-5	02/02/2009
25.6	Client Confidentiality	25-5	02/02/2009
25.7	Format Of Reports	25-6	02/02/2009
25.8	Amendments To Test Reports	25-6	02/02/2009
25.9	Policies On Client Requests For Amendments	25-6	02/02/2009

LIST OF TABLES

Table No.	Title	Page	Effective Date
12-1	Example- General Corrective Action Procedures	12-6	02/02/2009
14-1	Record Index	14-1	02/02/2009
14-2	Special Record Retention Requirements	14-3	02/02/2009
15-1	Audit Types and Frequency	15-1	02/02/2009
20-1	Example - Laboratory Equipment & Instrumentation	20-8	02/02/2009
20-2	Example – Schedule of Routine Maintenance	20-11	02/02/2009
20-3	Example – Periodic Calibration	20-13	02/02/2009
24-1	Negative Controls	24-1	02/02/2009
24-5	Sample Matrix Control	24-4	02/02/2009

LIST OF FIGURES

Figure No.	Title	Page	Effective Date
4-1	Corporate and Laboratory Organizational Charts	4-3	02/02/2009
8-1	Subcontracted Laboratory Approval Form	8-5	02/02/2009
12-1	Example - Corrective Action Report	12-5	02/02/2009
19-1	Demonstration of Capability Documentation	19-16	02/02/2009
23-1	Example – Chain of Custody	23-6	02/02/2009
23-2	Example – Sample Acceptance Policy	23-7	02/02/2009
23-3	Example – Cooler Receipt Form	23-10	02/02/2009

LIST OF APPENDICES

Appendix No.	Title	Page	Effective Date
1	Laboratory Floor Plan	Appendix 1-1	02/02/2009
2	Glossary / Acronyms	Appendix 2-1	02/02/2009
3	Laboratory Certifications, Accreditations, Validations	Appendix 3-1	02/02/2009

REFERENCED CORPORATE SOPs AND POLICIES

SOP/Policy Reference	Title
CA-Q-S-001	Solvent and Acid Lot Testing and Approval
CA-Q-S-002	Acceptable Manual Integration Practices
CA-Q-S-004	Method Compliance & Data Authenticity Audits
CA-Q-S-006	Detection Limits
CW-Q-S-001	Corporate Document Control and Archiving
CW-Q-S-002	Writing a Standard Operating Procedure (SOPs)
CA-L-S-001	Internal Investigation of Potential Data Discrepancies and Determination for Data Recall
CA-L-S-002	Subcontracting Procedures
CA-L-P-001	Ethics Policy
CA-L-P-002	Contract Compliance Policy
CW-F-P-002	Authorization Matrix
CW-F-P-004	Procurement and Contracts Policy
CA-C-S-001	Work Sharing Process
CA-T-P-001	Qualified Products List
CW-F-S-007	Controlled Purchases Policy
CW-F-S-018	Vendor Selection
CA-Q-M-002	Corporate Quality Management Plan

REFERENCED LABORATORY SOPs

SOP Reference	Title
BF-GP-001	Calibration of Autopipettes and Repipetters
BF-GP-002	Support Equipment: Maintenance, Record Keeping and Corrective Actions
BF-GP-005	Sample Homogenization and Subsampling
BF-GP-012	Technical Data Review
BF-GP-013	Manual Integration
BF-GP-015	Record Storage and Retention
BF-GP-018	Strict Internal Chain or Custody
BF-GP-019	Standard Traceability and Preparation
BF-GP-020	Thermometer Calibration
BF-PM-001	Project Information Requirements
BF-PM-003	Bottle Order Set-up
BF-PM-005	Correctness of Analysis
BF-QA-001	Determination of Method Detection Limits
BF-QA-002	Quality Control Limits
BF-QA-003	Procedure for Writing, Reviewing and Revising Controlled Documents
BF-QA-004	Laboratory Personnel Training
BF-QA-005	Preventative and Corrective Action
BF-QA-006	Data Quality Review
BF-SR-001	Cooler Shipping - Bottle Kits and Samples
BF-SR-002	Receipt of Analytical Samples

SECTION 3

INTRODUCTION
(NELAC 5.1 - 5.3)

3.1 INTRODUCTION AND COMPLIANCE REFERENCES

TestAmerica Buffalo's Quality Assurance Manual (QAM) is a document prepared to define the overall policies, organization objectives and functional responsibilities for achieving TestAmerica's data quality goals. The laboratory maintains a local perspective in its scope of services and client relations and maintains a national perspective in terms of quality.

The QAM has been prepared to assure compliance with the 2003 National Environmental Laboratory Accreditation Conference (NELAC) standards and ISO/IEC Guide 17025 (1999 or 2005 if you're an A2LA lab). In addition, the policies and procedures outlined in this manual are compliant with TestAmerica's Corporate Management Plan (CQMP) and the various accreditation and certification programs listed in Appendix 3. The CQMP provides a summary of TestAmerica's quality and data integrity system. It contains requirements and general guidelines under which all TestAmerica facilities shall conduct their operations.

The QAM has been prepared to be consistent with the requirements of the following documents:

- EPA 600/4-88/039, *Methods for the Determination of Organic Compounds in Drinking Water,* EPA, Revised July 1991.
- EPA 600/R-95/131, *Methods for the Determination of Organic Compounds in Drinking Water,* Supplement III, EPA, August 1995.
- EPA 600/4-79-019, *Handbook for Analytical Quality Control in Water and Wastewater Laboratories,* EPA, March 1979.
- EPA SW-846, *Test Methods for the Evaluation of Solid Waste, 3rd Edition,* September 1986; Update I, July 1992; Update II, September 1994; and Update III, December 1996.
- Federal Register, 40 CFR Parts 136, 141, 172, 173, 178, 179 and 261.
- USEPA Contract Laboratory Program. *Statement of Work for Inorganics Analysis. Multi-Media, Multi-Concentration*. Document ILM04.0/4.1/4.2
- USEPA Contract Laboratory Program. *Statement of Work for Inorganics Analysis. Multi-Media, Multi-Concentration*. Document ILM05.1/5.2/ 5.3.
- USEPA Contract Laboratory Program. *Statement of Work for Organics Analysis. Multi-Media, Multi-Concentration.* Document Number OLM03.1, August 1994.
- USEPA Contract Laboratory Program. *Statement of Work for Organics Analysis. Multi-Media, Multi-Concentration.* Document Number OLM04.0, August 1994 and updates
- New York State Analytical Services Protocol, July 2005
- APHA, *Standard Methods for the Examination of Water and Wastewater,* 18th Edition, 19th, 20th and 21st Edition.
- *U.S. Department of Energy* Order 414.1B, *Quality Assurance*, Approved April 29, 2004.
- *U.S. Department of Energy*, *Quality Systems for Analytical Services*, Revision 2.1, November 2005.

- Toxic Substances Control Act (TSCA).

3.2 TERMS AND DEFINITIONS

A Quality Assurance Program is a company-wide system designed to ensure that data produced by the laboratory conforms to the standards set by state and/or federal regulations. The program functions at the management level through company goals and management policies, and at the analytical level through Standard Operating Procedures (SOPs) and quality control. The TestAmerica program is designed to minimize systematic error, encourage constructive, documented problem solving, and provide a framework for continuous improvement within the organization.

Refer to Appendix 2 for the Glossary/Acronyms.

3.3 SCOPE / FIELDS OF TESTING

The laboratory analyzes a broad range of environmental and industrial samples every month. Sample matrices vary among air, drinking water, effluent water, groundwater, hazardous waste, sludge and soils. The Quality Assurance Program contains specific procedures and methods to test samples of differing matrices for chemical, physical and biological parameters. The Program also contains guidelines on maintaining documentation of analytical process, reviewing results, servicing clients and tracking samples through the laboratory. The technical and service requirements of all requests to provide analyses are thoroughly evaluated before commitments are made to accept the work. Measurements are made using published reference methods or methods developed and validated by the laboratory.

The methods covered by this manual include the most frequently requested methodologies needed to provide analytical services in the United States and its territories. The approach of this manual is to define the minimum level of quality assurance and quality control necessary to meet requirements. All methods performed by the laboratory shall meet these criteria as appropriate. In some instances, quality assurance project plans (QAPPs), project specific data quality objectives (DQOs) or local regulations may require criteria other than those contained in this manual. In these cases, the laboratory will abide by the requested criteria following review and acceptance of the requirements by the Laboratory Director/Manager and the Quality Assurance (QA) Manager. In some cases, QAPPs and DQOs may specify less stringent requirements. The Laboratory Director/Manager and the QA Manager must determine if it is in the lab’s best interest to follow the less stringent requirements.

3.4 MANAGEMENT OF THE MANUAL

3.4.1 Review Process

The manual is reviewed annually by senior laboratory management to assure that it reflects current practices and meets the requirements of the laboratory’s clients and regulators as well as the CQMP. Occasionally, the manual may need changes in order to meet new or changing regulations and operations. The QA Manager will review the changes in the normal course of business and incorporate changes into revised sections of the document. All updates will be reviewed by the senior laboratory management staff. The laboratory updates and approves such changes according to our Document Control & updating procedures (refer to BF-QA-003)

SECTION 4

ORGANIZATION AND MANAGEMENT
(NELAC 5.4.1)

4.1 OVERVIEW

TestAmerica Buffalo is a local operating unit of TestAmerica Laboratories, Inc.. The organizational structure, responsibilities and authorities of the corporate staff of TestAmerica Laboratories, Inc. are presented in the CQMP. TestAmerica Buffalo has day-to-day independent operational authority overseen by corporate officers (e.g., President, Chief Operating Officer, Corporate Quality Assurance, etc.). The TestAmerica Buffalo laboratory operational and support staff work under the direction of the Laboratory Director. The organizational structure for both Corporate & TestAmerica Buffalo is presented in Figure 4-1.

4.2 ROLES AND RESPONSIBILITIES

In order for the Quality Assurance Program to function properly, all members of the staff must clearly understand and meet their individual responsibilities as they relate to the quality program. The following descriptions briefly define each role in its relationship to the Quality Assurance Program.

4.2.1 Quality Assurance Program

The responsibility for quality lies with every employee of the laboratory. All employees have access to the QAM, are trained to this manual and are responsible for upholding the standards therein. Each person carries out his/her daily tasks in a manner consistent with the goals and in accordance with the procedures in this manual and the laboratory's SOPs. Role descriptions for corporate personnel are defined in the CQMP. This manual is specific to the operations of TestAmerica's Buffalo laboratory.

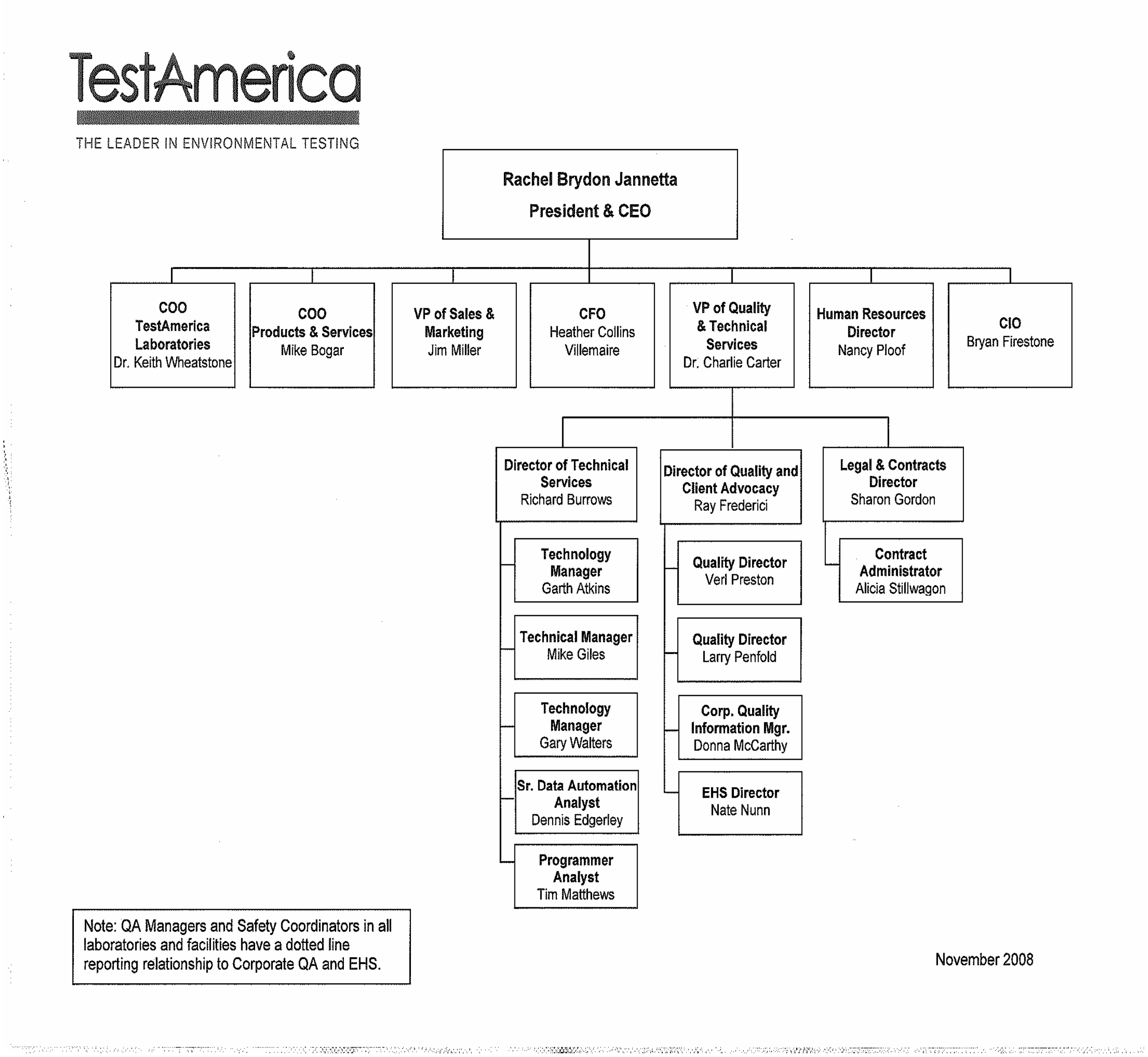
4.3 DEPUTIES

The following table defines who assumes the responsibilities of key personnel in their absence:

Key Personnel	Deputy	Comment
Laboratory Director	Operations Manager (1) Technical Director (2)	
QA Manager	QA Specialist (1) Operations Manager (2)	
Technical Director	Laboratory Director (1) Operations Manager (2)	
Operations Manager	Department Manager (1) Department Manager (2)	Selected based on availability
Customer Service Manager	Project Mng't Manager (1) Laboratory Director (2)	

Key Personnel	Deputy	Comment
Project Management Manager	Customer Srv. Manager (1) Project Manager (2)	(2) Selected based on availability
Project Manager	Project Manager (1) Project Management Asst. (2)	(1) 2° team PM (2) Team PMA
Organic Department Manager	Analyst (1) Analyst (2)	Selected based on department, experience and availability
Inorganic Department Manager	Analyst (1) Analyst (2)	Selected based on department, experience and availability
Data Validation / Data Packaging Manager	Data Validation Specialist Data Packaging Specialist	Selected based on department and availability
EHS Coordinator	Safety Officer (1) Sample Mng't Manager (2)	
Sample Management Manager	Sample Custodian (1) EHS Coordinator (2)	
Bottle Preparation / Shipping Manager	Bottle Prep Technician (1) Sample Mng't Manager (2)	

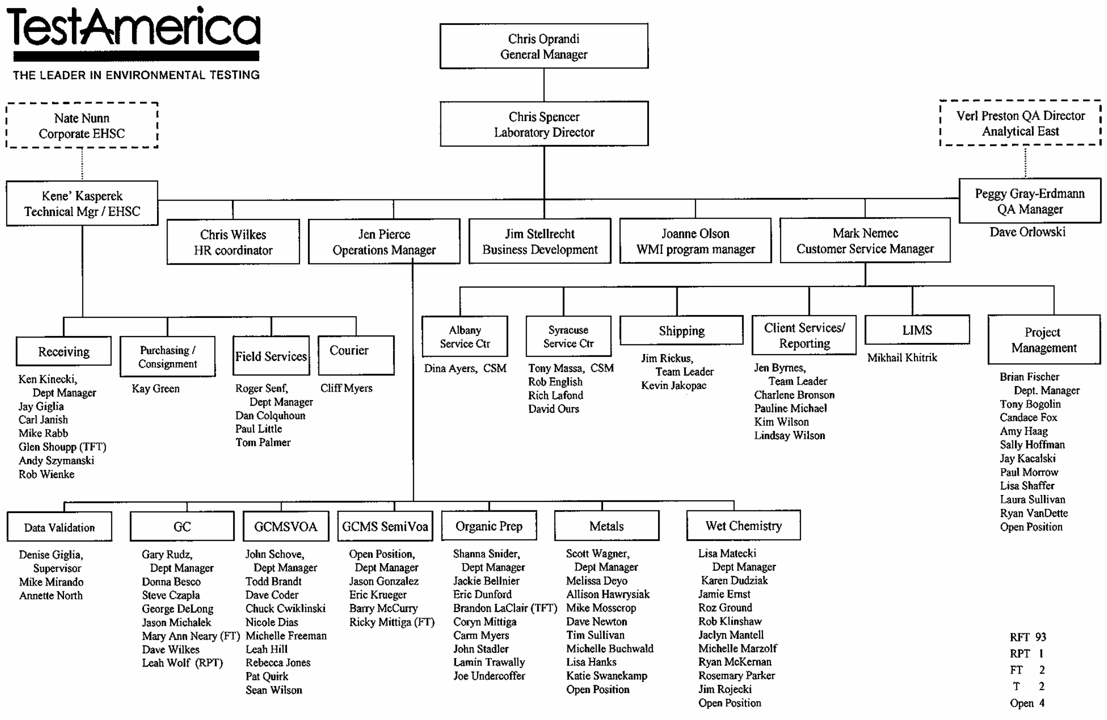
Figure 4-1.
Corporate and Laboratory Organization Charts



Note: QA Managers and Safety Coordinators in all laboratories and facilities have a dotted line reporting relationship to Corporate QA and EHS.

November 2008

TestAmerica Organizational Chart
Buffalo Laboratory



Updated: 02/02/09

SECTION 5

QUALITY SYSTEM
(NELAC 5.4.2)

5.1 QUALITY POLICY STATEMENT

It is TestAmerica's Policy to:

- Provide data of known quality to its clients by adhering to approved methodologies, regulatory requirements and the QA/QC protocols.
- Effectively manage all aspects of the laboratory and business operations by the highest ethical standards.
- Continually improve systems and provide support to quality improvement efforts in laboratory, administrative and managerial activities. TestAmerica recognizes that the implementation of a quality assurance program requires management's commitment and support as well as the involvement of the entire staff.
- Provide clients with the highest level of professionalism and the best service practices in the industry.

Every staff member at the laboratory plays an integral part in quality assurance and is held responsible and accountable for the quality of their work. It is, therefore, required that all laboratory personnel are trained and agree to comply with applicable procedures and requirements established by this document.

5.2 ETHICS AND DATA INTEGRITY

TestAmerica is committed to ensuring the integrity of its data and meeting the quality needs of its clients. The 7 elements of TestAmerica's Ethics and Data Integrity Program include:

- An Ethics Policy (Corporate Policy No. CA-L-P-001) and Employee Ethics Statements.
- Ethics and Compliance Officers (ECOs).
- A training program.
- Self-governance through disciplinary action for violations.
- A confidential mechanism for anonymously reporting alleged misconduct and a means for conducting internal investigations of all alleged misconduct. (Corporate SOP No. CA-L-S-001)
- Procedures and guidance for recalling data if necessary (Corporate SOP No. CA-L-S-001).
- Effective external and internal monitoring system that includes procedures for internal audits (Section 16).

- Produce results, which are accurate and include QA/QC information that meets client pre-defined Data Quality Objectives (DQOs).
- Present services in a confidential, honest and forthright manner.
- Provide employees with guidelines and an understanding of the Ethical and Quality Standards of our industry.
- Operate our facilities in a manner that protects the environment and the health and safety of employees and the public.
- Obey all pertinent federal, state and local laws and regulations and encourage other members of our industry to do the same.
- Educate clients as to the extent and kinds of services available.
- Assert competency only for work for which adequate personnel and equipment are available and for which adequate preparation has been made.
- Promote the status of environmental laboratories, their employees, and the value of services rendered by them.

5.3 QUALITY SYSTEM DOCUMENTATION

The laboratory's Quality System is communicated through a variety of documents:

- Quality Assurance Manual – Each laboratory has a lab specific quality assurance manual.
- Corporate SOPs and Policies - Corporate SOPs and Policies are developed for use by all relevant laboratories. They are incorporated into the laboratories normal SOP distribution, training and tracking system. Corporate SOPs may be general or technical.
- Work Instructions - A subset of procedural steps, tasks or forms associated with an operation of a management system (e.g., checklists, preformatted bench sheets, forms).
- Laboratory SOPs – General and Technical
- Corporate Quality Policy Memorandums
- Laboratory QA/QC Policy Memorandums

5.3.1 Order of Precedence

In the event of a conflict or discrepancy between policies, the order of precedence is as follows:

- Corporate Quality Policy Memorandum
- Corporate Quality Management Plan (CQMP)
- Corporate SOPs and Policies
- Laboratory QA/QC Policy Memorandum
- Laboratory Quality Assurance Manual (QAM)
- Laboratory SOPs and Policies
- Other (Work Instructions (WI), memos, flow charts, etc.)

Note: The laboratory's has the responsibility and authority to operate in compliance with regulatory requirements of the jurisdiction in which the work is performed. Where the CQMP conflicts with those regulatory requirements, the regulatory requirements of the jurisdiction shall hold primacy. The laboratory's (QAM) shall take precedence over the CQMP in those cases.

5.4 QA/QC OBJECTIVES FOR THE MEASUREMENT OF DATA

Quality Assurance (QA) and Quality Control (QC) are activities undertaken to achieve the goal of producing data that accurately characterize the sites or materials that have been sampled. Quality Assurance is generally understood to be more comprehensive than Quality Control. Quality Assurance can be defined as the integrated system of activities that ensures that a product or service meets defined standards.

Quality Control is generally understood to be limited to the analyses of samples and to be synonymous with the term *"analytical quality control"*. QC refers to the routine application of statistically based procedures to evaluate and control the accuracy of results from analytical measurements. The QC program includes procedures for estimating and controlling precision and bias and for determining reporting limits.

Request for Proposals (RFPs) and Quality Assurance Project Plans (QAPP) provide a mechanism for the client and the laboratory to discuss the data quality objectives in order to ensure that analytical services closely correspond to client needs. The client is responsible for developing the QAPP. In order to ensure the ability of the laboratory to meet the Data Quality Objectives (DQOs) specified in the QAPP, clients are advised to allow time for the laboratory to review the QAPP before being finalized. Additionally, the laboratory will provide support to the client for developing the sections of the QAPP that concern laboratory activities.

Historically, laboratories have described their QC objectives in terms of precision, accuracy, representativeness, comparability, completeness, selectivity and sensitivity (PARCCSS).

5.4.1 Precision

The laboratory objective for precision is to meet the performance for precision demonstrated for the methods on similar samples and to meet data quality objectives of the EPA and/or other regulatory programs. Precision is defined as the degree of reproducibility of measurements under a given set of analytical conditions (exclusive of field sampling variability). Precision is documented on the basis of replicate analysis, usually duplicate or matrix spike (MS) duplicate samples.

5.4.2 Accuracy

The laboratory objective for accuracy is to meet the performance for accuracy demonstrated for the methods on similar samples and to meet data quality objectives of the EPA and/or other regulatory programs. Accuracy is defined as the degree of bias in a measurement system. Accuracy may be documented through the use of laboratory control samples (LCS) and/or MS. A statement of accuracy is expressed as an interval of acceptance recovery about the mean recovery.

5.4.3 Representativeness

The laboratory objective for representativeness is to provide data which is representative of the sampled medium. Representativeness is defined as the degree to which data represent a characteristic of a population or set of samples and is a measurement of both analytical and field sampling precision. The representativeness of the analytical data is a function of the procedures used in procuring and processing the samples. The representativeness can be documented by the relative percent difference between separately procured, but otherwise identical samples or sample aliquots.

The representativeness of the data from the sampling sites depends on both the sampling procedures and the analytical procedures. The laboratory may provide guidance to the client regarding proper sampling and handling methods in order to assure the integrity of the samples.

5.4.4 Comparability

The comparability objective is to provide analytical data for which the accuracy, precision, representativeness and reporting limit statistics are similar to these quality indicators generated by other laboratories for similar samples, and data generated by the laboratory over time.

The comparability objective is documented by inter-laboratory studies carried out by regulatory agencies or carried out for specific projects or contracts, by comparison of periodically generated statements of accuracy, precision and reporting limits with those of other laboratories.

5.4.5 Completeness

The completeness objective for data is 90% (or as specified by a particular project), expressed as the ratio of the valid data to the total data over the course of the project. Data will be considered valid if they are adequate for their intended use. Data usability will be defined in a QAPP, project scope or regulatory requirement. Data validation is the process for reviewing data to determine its usability and completeness. If the completeness objective is not met, actions will be taken internally and with the data user to improve performance. This may take the form of an audit to evaluate the methodology and procedures as possible sources for the difficulty or may result in a recommendation to use a different method.

5.4.6 Selectivity

Selectivity is defined as: The capability of a test method or instrument to respond to a target substance or constituent in the presence of non-target substances. Target analytes are separated from non-target constituents and subsequently identified/detected through one or more of the following, depending on the analytical method: extractions (separation), digestions (separation), interelement corrections (separation), use of matrix modifiers (separation), specific retention times (separation and identification), confirmations with different columns or detectors (separation and identification), specific wavelengths (identification), specific mass spectra (identification), specific electrodes (separation and identification), etc..

5.4.7 Sensitivity

Sensitivity refers to the amount of analyte necessary to produce a detector response that can be reliably detected (Method Detection Limit) or quantified (Reporting Limit).

5.5 CRITERIA FOR QUALITY INDICATORS

The laboratory maintains a *Quality Control Limit Summary that contains tables* that summarize the precision and accuracy acceptability limits for performed analyses. This summary includes an effective date, is updated each time new limits are generated and are managed by the laboratory's QA department. Unless otherwise noted, limits within these tables are laboratory generated. Some acceptability limits are derived from US EPA methods when they are required. Where US EPA method limits are not required, the laboratory has developed limits from evaluation of data from similar matrices. Criteria for development of control limits is contained in Section 24.

5.6 STATISTICAL QUALITY CONTROL

Statistically-derived precision and accuracy limits are required by selected methods (such as SW-846) and programs [such as the Ohio Voluntary Action Plan (VAP)]. The laboratory routinely utilizes statistically-derived limits to evaluate method performance and determine when corrective action is appropriate. The procedure for determining the statistical limits may be found in SOP BF-QA-002, Quality Control Limits. The analysts are instructed to use the current limits in the laboratory (dated and approved the QA Manager) and entered into the Laboratory Information Management System (LIMS). The Quality Assurance department maintains an archive of all limits used within the laboratory through date sensitive tables within the LIMs System. If a method defines the QC limits, the method limits are used.

If a method requires the generation of historical limits, the lab develops such limits from recent data in the QC database of the LIMS following the guidelines described in Section 25. All calculations and limits are documented and dated when approved and effective. On occasion, a client requests contract-specified limits for a specific project.

Surrogate recoveries are determined for a specific time period as defined above. The resulting ranges are entered in LIMS.

Current QC limits are entered and maintained in the LIMS analyte database. As sample results and the related QC are entered into LIMS, the sample QC values are compared with the limits in LIMS to determine if they are within the acceptable range. The analyst then evaluates if the sample needs to be rerun or re-extracted/rerun or if a comment should be added to the report explaining the reason for the QC outlier.

5.6.1 QC Charts

The QA Manager periodically evaluates these to determine if adjustments need to be made or for corrective actions to methods. All findings are documented and kept on file.

5.7 QUALITY SYSTEM METRICS

In addition to the QC parameters discussed above, the entire Quality System is evaluated on a monthly basis through the use of specific metrics (refer to Section 16). These metrics are used to drive continuous improvement in the laboratory's Quality System.

SECTION 6

DOCUMENT CONTROL
(NELAC 5.4.3)

6.1 OVERVIEW

The QA Department is responsible for the control of documents used in the laboratory to ensure that approved, up-to-date documents are in circulation and out-of-date (obsolete) documents are archived or destroyed. The following documents, at a minimum, must be controlled:

- Laboratory Quality Assurance Manual
- Laboratory Standard Operating Procedures (SOP)
- Laboratory Policies
- Work Instructions and Forms
- Corporate Policies and Procedures distributed outside the intranet

Corporate Quality posts Corporate Manuals, SOPs, Policies, Work Instructions, White Papers and Training Materials on the company intranet site. These Corporate documents are only considered controlled when they are read on the intranet site. Printed copies are considered uncontrolled unless the laboratory physically distributes them as controlled documents. A detailed description of the procedure for issuing, authorizing, controlling, distributing, and archiving corporate documents is found in Corporate SOP No. CW-Q-S-001, Corporate Document Control and Archiving. The laboratory's internal document control procedure is defined in SOP No. BF-QA-003.

The laboratory QA Department also maintains access to various references and document sources integral to the operation of the laboratory. This includes reference methods and regulations. Instrument manuals (hard or electronic copies) are also maintained by the laboratory.

The laboratory maintains control of records for raw analytical data and supporting records such as audit reports and responses, logbooks, standard logs, training files, MDL studies, Proficiency Testing (PT) studies, certifications and related correspondence, and corrective action notices. Raw analytical data consists of bound logbooks, instrument printouts, any other notes, magnetic media, electronic data and final reports.

6.2 DOCUMENT APPROVAL AND ISSUE

The pertinent elements of a document control system for each document include a unique document title and number, the number of pages of the item, the effective date, revision number and the laboratory's name. The Quality personnel are responsible for the maintenance of the system.

Controlled documents are authorized by the QA Department and other management. In order to develop a new document, a Department Manager submits an electronic draft to the QA

Department for suggestions and approval before use. Upon approval, QA personnel add the identifying version information to the document and retain the official document on file. The official document is provided to all applicable operational units. Controlled documents are identified as such and records of their distribution are kept by the QA Department. Document control may be achieved by either electronic or hardcopy distribution.

The QA Department maintains a list of the official versions of controlled documents.

Quality System Policies and Procedures will be reviewed at a minimum of every two years for the majority of procedures, every 1 year for Drinking Water programs and revised as appropriate. Changes to documents occur when a procedural change warrants.

6.3 PROCEDURES FOR DOCUMENT CONTROL POLICY

For changes to the QA Manual, refer to SOP No. BF-QA-003, “Writing, Reviewing and Revising Controlled Documents”. Uncontrolled copies must not be used within the laboratory. Previous revisions and back-up data are stored by the QA department. A controlled electronic copy of the current version is maintained on the laboratory IntraNet site and is available to all personnel.

For changes to SOPs, refer to SOP No. BF-QA-003, “Writing, Reviewing and Revising Controlled Documents”.

Forms, worksheets, work instructions and information are organized by department in the QA office. Electronic versions are kept in a controlled access electronic folder in the QA department. As revisions are required, a new version number and revision date is assigned and the document placed on the laboratory IntraNet (BufNet) for use.

6.4 OBSOLETE DOCUMENTS

All invalid or obsolete documents are removed, or otherwise prevented from unintended use. The laboratory has specific procedures as described above to accomplish this. In general, obsolete documents are collected from employees according to distribution lists and are marked obsolete on the cover or destroyed. At least one copy of the obsolete document is archived according to SOP No. BF-GP-015.

SECTION 7

SERVICE TO THE CLIENT

7.1 OVERVIEW

The laboratory has established procedures for the review of work requests and contracts, oral or written. The procedures include evaluation of the laboratory's capability and resources to meet the contract's requirements within the requested time period. All requirements, including the methods to be used, must be adequately defined, documented and understood. For many environmental sampling and analysis programs, testing design is site or program specific and does not necessarily "fit" into a standard laboratory service or product. It is the laboratory's intent to provide both standard and customized environmental laboratory services to our clients.

A thorough review of technical and QC requirements contained in contracts is performed to ensure project success. The appropriateness of requested methods, and the lab's capability to perform them must be established. Projects, proposals and contracts are reviewed for adequately defined requirements and the laboratory's capability to meet those requirements. Alternate test methods that are capable of meeting the clients' requirements may be proposed by the lab. A review of the lab's capability to analyze non-routine analytes is also part of this review process.

All projects, proposals and contracts are reviewed for the client's requirements in terms of compound lists, test methodology requested, sensitivity (detection and reporting levels), accuracy, and precision requirements (% Recovery and RPD). The reviewer ensures that the laboratory's test methods are suitable to achieve these regulatory and client requirements and that the laboratory holds the appropriate certifications and approvals to perform the work. The laboratory and any potential subcontract laboratories must be certified, as required, for all proposed tests.

The laboratory must determine if it has the necessary physical, personnel and information resources to meet the contract, and if the personnel have the expertise needed to perform the testing requested. Each proposal is checked for its impact on the capacity of the laboratory's equipment and personnel. As part of the review, the proposed turnaround time will be checked for feasibility.

Electronic or hard copy deliverable requirements are evaluated against the lab's capacity for production of the documentation.

If the laboratory cannot provide all services but intends to subcontract such services, whether to another TestAmerica facility or to an outside firm, this will be documented and discussed with the client prior to contract approval. (Refer to Section 8 for Subcontracting Procedures.)

The laboratory informs the client of the results of the review if it indicates any potential conflict, deficiency, lack of accreditation, or inability of the lab to complete the work satisfactorily. Any discrepancy between the client's requirements and the laboratory's capability to meet those requirements is resolved in writing before acceptance of the contract. It is necessary that the

contract be acceptable to both the laboratory and the client. Amendments initiated by the client and/or TestAmerica, are documented in writing.
All contracts, QAPPs, Sampling and Analysis Plans (SAPs), contract amendments, and documented communications become part of the project record.

The same contract review process used for the initial review is repeated when there are amendments to the original contract by the client and the participating personnel are informed of the changes.

7.2 REVIEW SEQUENCE AND KEY PERSONNEL

Appropriate personnel will review the work request at each stage of evaluation.

For routine projects and other simple tasks, a review by the Project Manager (PM) is considered adequate. The PM confirms that the laboratory has any required certifications, that it can meet the clients' data quality and reporting requirements and that the lab has the capacity to meet the clients turn around needs. It is recommended that, where there is a sales person assigned to the account, an attempt should be made to contact that sales person to inform them of the incoming samples.

For new, complex or large projects, the proposed contract is given to the National Account Director, who will decide which lab will receive the work based on the scope of work and other requirements, including certification, testing methodology, and available capacity to perform the work. The contract review process is outlined in TestAmerica's Corporate SOP No. CA-L-P-002**,** Contract Compliance Policy.

This review encompasses all facets of the operation. The scope of work is distributed to the appropriate personnel, as needed based on scope of contract, to evaluate all of the requirements shown above (not necessarily in the order below):

- Legal & Contracts Director
- General Manager
- Customer Service Manager
- Operations Manager
- Laboratory and/or Corporate Technical Directors
- Corporate Information Technology Managers/Directors
- Regional and/or National Account representatives
- Laboratory and/or Corporate Quality
- Laboratory and/or Corporate Environmental Health and Safety Managers/Directors
- The Laboratory Director reviews the formal laboratory quote and makes final acceptance for their facility.

The National Account Director, Legal Contracts Director, or local account representative then submits the final proposal to the client.

In the event that one of the above personnel is not available to review the contract, his or her back-up will fulfill the review requirements.

The Legal & Contracts Director maintains copies of all signed contracts. The Customer Service Manager at the TestAmerica Buffalo facility also maintains copies of these documents.

7.3 DOCUMENTATION

Appropriate records are maintained for every contract or work request. All stages of the contract review process are documented and include records of any significant changes.

The contract will be distributed to and maintained by the appropriate sales/marketing personnel and the Regional Account Manager. A copy of the contract and formal quote will be filed with the laboratory PM and the Customer Service Manager.

Records are maintained of pertinent discussions with a client relating to the client's requirements or the results of the work during the period of execution of the contract. The PM keeps a phone log of conversations with the client.

7.3.1 Project-Specific Quality Planning

Communication of contract specific technical and QC criteria is an essential activity in ensuring the success of site specific testing programs. To achieve this goal, the laboratory assigns a PM to each client. The PM is the first point of contact for the client. It is the PM's responsibility to ensure that project specific technical and QC requirements are effectively evaluated and communicated to the laboratory personnel before and during the project. QA department involvement may be needed to assist in the evaluation of custom QC requirements. Specific information related to project planning may be found in SOP BF-PM-001, Project Information Requirements.

PM's are the primary client contact and they ensure resources are available to meet project requirements. Although PM's do not have direct reports or staff in production, they coordinate opportunities and work with laboratory management staff to ensure available resources are sufficient to perform work for the client's project. Project management is positioned between the client and laboratory resources.

Prior to work on a new project, the dissemination of project information and/or project opening meetings may occur to discuss schedules and unique aspects of the project. Items to be discussed may include the project technical profile, turnaround times, holding times, methods, analyte lists, reporting limits, deliverables, sample hazards, or other special requirements. The PM introduces new projects to the laboratory staff through project kick-off meetings or to the management staff during production meetings. These meetings provide direction to the laboratory staff in order to maximize production and client satisfaction, while maintaining quality. In addition, project notes may be associated with each sample batch as a reminder upon sample receipt and analytical processing.

During the project, any change that may occur within an active project is agreed upon between the client/regulatory agency and the PM/laboratory. These changes (e.g., use of a non-standard method or modification of a method) and approvals must be documented prior to implementation. Documentation pertains to any document, e.g., letter, e-mail, variance, contract addendum, which has been signed by both parties.

Such changes are also communicated to the laboratory during production meetings. Such changes are updated to the project notes and are introduced to the managers at these meetings. The laboratory staff is then introduced to the modified requirements via the PM or the individual laboratory Department Manager.

The laboratory strongly encourages client visits to the laboratory and for formal/informal information sharing session with employees in order to effectively communicate ongoing client needs as well as project specific details for customized testing programs.

7.4 SPECIAL SERVICES

The laboratory cooperates with clients and their representatives to monitor the laboratory's performance in relation to work performed for the client. It is the laboratory's goal to meet all client requirements in addition to statutory and regulatory requirements. The laboratory has procedures to ensure confidentiality to clients (Section 15 and 25).

Note: ISO 17025/NELAC 2003 states that a laboratory "shall afford clients or their representative's cooperation to clarify the client's request". This topic is discussed in Section 7.

The laboratory's standard procedures for reporting data are described in Section 25. Special services are also available and provided upon request. These services include:

- Reasonable access for our clients or their representatives to the relevant areas of the laboratory for the witnessing of tests performed for the client.
- Assist client-specified third party data validators as specified in the client's contract.
- Supplemental information pertaining to the analysis of their samples. Note: An additional charge may apply for additional data/information that was not requested prior to the time of sample analysis or previously agreed upon.

7.5 CLIENT COMMUNICATION

Project managers are the primary communication link to the clients. They shall inform their clients of any delays in project completion as well as any non-conformances in either sample receipt or sample analysis. Project management will maintain ongoing client communication throughout the entire client project.

Technical Directors are available to discuss any technical questions or concerns that the client may have.

7.6 REPORTING

The laboratory works with our clients to produce any special communication reports required by the contract.

7.7 CLIENT SURVEYS

The laboratory assesses both positive and negative client feedback. The results are used to improve overall laboratory quality and client service.

TestAmerica’s Sales and Marketing teams periodically develops lab and client specific surveys to assess client satisfaction.

SECTION 8

SUBCONTRACTING OF TESTS
(NELAC 5.4.5)

8.1 OVERVIEW

For the purpose of this quality manual, the phrase subcontract laboratory refers to a laboratory external to the TestAmerica laboratories. The phrase “work sharing” refers to internal transfers of samples between the TestAmerica laboratories. The term outsourcing refers to the act of subcontracting tests.

When contracting with our clients, the laboratory makes commitments regarding the services to be performed and the data quality for the results to be generated. When the need arises to outsource testing for our clients because project scope, changes in laboratory capabilities, capacity or unforeseen circumstances, we must be assured that the subcontractors or work sharing laboratories understand the requirements and will meet the same commitments we have made to the client. Refer to TestAmerica’s Corporate SOP’s on Subcontracting Procedures (CA-L-S-002) and the Work Sharing Process SOP (CA-C-S-001).

When outsourcing analytical services, the laboratory will assure, to the extent necessary, that the subcontract or work sharing laboratory maintains a program consistent with the requirements of this document, the requirements specified in NELAC/ISO 17025 and/or the client’s Quality Assurance Project Plan (QAPP). All QC guidelines specific to the client’s analytical program are transmitted to the subcontractor and agreed upon before sending the samples to the subcontract facility. Additionally, work requiring accreditation will be placed with an appropriately accredited laboratory. The laboratory performing the subcontracted work will be identified in the final report, as will non-NELAC accredited work where required.

Project Managers (PMs), Customer Service Managers (CSM), or Regional Account Executives (RAE) for the Export Lab are responsible for obtaining client approval prior to outsourcing any samples. The laboratory will advise the client of a subcontract or work sharing arrangement in writing and when possible approval from the client shall be retained in the project folder.

Note: In addition to the client, some regulating agencies, such as the Department of Energy and the USDA, require notification prior to placing such work**.**

Approval may be documented through reference in a quote / contract or e-mail correspondence.

8.2 QUALIFYING AND MONITORING SUBCONTRACTORS

Whenever a PM, Regional Account Executive (RAE) or Customer Service Manager (CSM] becomes aware of a client requirement or laboratory need where samples must be outsourced to another laboratory, the other laboratory(s) shall be selected based on the following:

- The first priority is to attempt to place the work in a qualified TestAmerica laboratory;
- Firms specified by the client for the task (Documentation that a subcontractor was

designated by the client must be maintained with the project file. This documentation can be

- as simple as placing a copy of an e-mail from the client in the project folder);
- Firms listed as pre-qualified and currently under a subcontract with TestAmerica. A listing of all approved subcontracting laboratories and supporting documentation is available on the TestAmerica intranet site. Verify necessary accreditation, where applicable (e.g. on the subcontractors NELAC, A2LA accreditation or State certification.
- Firms identified in accordance with the company's Small Business Subcontracting program as small, women-owned, veteran-owned and/or minority-owned businesses;
- NELAC or A2LA accredited laboratories.
- In addition, the firm must hold the appropriate certification to perform the work required.

All TestAmerica laboratories are pre-qualified for work-sharing provided they hold the appropriate accreditations, can adhere to the project/program requirements, and the client approved sending samples to that laboratory. The client must provide acknowledgement that the samples can be sent to that facility (an e-mail is sufficient documentation or if acknowledgement is verbal, the date, time, and name of person providing acknowledgement must be documented). The originating laboratory is responsible for communicating all technical, quality, and deliverable requirements as well as other contract needs. (Corporate SOP No. CA-C-S-001, Work Sharing Process.

When the potential sub-contract laboratory has not been previously approved, then to begin the process, Account Executives or PMs may nominate a laboratory as a subcontractor based on need. The decision to nominate a laboratory must be approved by the Laboratory Director. The Laboratory Director requests that the QA Manager begin the process of approving the subcontract laboratory as outlined in Corporate SOP No. CA-L-S-002, Subcontracting Procedures. The client must provide acknowledgement that the samples can be sent to that facility (an e-mail is sufficient documentation or if acknowledgement is verbal, the date, time, and name of person providing acknowledgement must be documented).

8.2.1 Once the appropriate accreditation and legal information is received by the laboratory, it is evaluated for acceptability (where applicable) and forwarded to Corporate Contracts for formal contracting with the laboratory. They will add the lab to the approved list on the intranet site along with the associate documentation and notify the finance group for JD Edwards.

8.2.2 The client will assume responsibility for the quality of the data generated from the use of a subcontractor they have requested the lab to use. The qualified subcontractors on the intranet site are known to meet minimal standards. TestAmerica does not certify laboratories. The subcontractor is on our approved list and can only be recommended to the extent that we would use them.

8.2.3 The status and performance of qualified subcontractors will be monitored periodically by the Corporate Contracts and/or Quality Departments. Any problems identified will be brought to the attention of TestAmerica's Corporate Finance or Corporate Quality personnel.

- Complaints shall be investigated. Documentation of the complaint, investigation and
- corrective action will be maintained in the subcontractor's file on the intranet site. Complaints are posted using the Vendor Performance Report (Form No. CW-F-WI-009)**.**
- Information shall be updated on the intranet when new information is received from the subcontracted laboratories.
- Subcontractors in good standing will be retained on the intranet listing. The QA Manager will notify all TestAmerica laboratories and Corporate Quality and Corporate Contracts if any laboratory requires removal from the intranet site. This notification will be posted on the intranet site and e-mailed to all Lab Directors/Managers, QA Managers and Sales Personnel.

8.3 OVERSIGHT AND REPORTING

The PM must request that the selected subcontractor be presented with a subcontract, if one is not already executed between the laboratory and the subcontractor. The subcontract must include terms which flow down the requirements of our clients, either in the subcontract itself or through the mechanism of work orders relating to individual projects. A standard subcontract and the Lab Subcontractor Vendor Package (posted on the intranet) can be used to accomplish this, and the Legal & Contracts Director can tailor the document or assist with negotiations, if needed. The PM (or RAE or CSM, etc.) responsible for the project must advise and obtain client consent to the subcontract as appropriate, and provide the scope of work to ensure that the proper requirements are made a part of the subcontract and are made known to the subcontractor.

Prior to sending samples to the subcontracted laboratory, the PM confirms their certification status to determine if it's current and scope-inclusive. The information is documented on a Subcontract Laboratory Certification Verification Form (Figure 8-1) and the form is retained in the project folder. For TestAmerica laboratories, certifications can be viewed on the company TotalAccess Database.

The Sample Control department is responsible for ensuring compliance with QA requirements and applicable shipping regulations when shipping samples to a subcontracted laboratory.

All subcontracted samples must be accompanied by a Chain of Custody (COC). A copy of the original COC sent by the client must be included with all samples subbed within TestAmerica.

Through communication with the subcontracted laboratory, the PM monitors the status of the subcontracted analyses, facilities successful execution of the work, and ensures the timeliness and completeness of the analytical report.

Non-NELAC accredited work must be identified in the subcontractor's report as appropriate. If NELAC accreditation is not required, the report does not need to include this information.

Reports submitted from subcontractor laboratories are not altered and are included in their original form in the final project report. This clearly identifies the data as being produced by a subcontractor facility. If subcontract laboratory data are incorporated into the laboratories EDD (i.e., imported), the report must explicitly indicate which lab produced the data for which methods and samples.

Note: The results submitted by TestAmerica work sharing laboratory may be transferred electronically and the results reported by the TestAmerica work sharing lab are identified on the final report. The report must explicitly indicate which lab produced the data for which methods and samples. The final report must include a copy of the completed COC for all work sharing reports.

8.4 CONTINGENCY PLANNING

The Laboratory Director may waive the full qualification of a subcontractor process temporarily to meet emergency needs. In the event this provision is utilized, The QA Manager will be required to verify certifications. The comprehensive approval process must then be initiated within 30 calendar days of subcontracting.

Figure 8-1 Subcontracting Laboratory Approval Form (Initial / Renewal)

SUBCONTRACTING LABORATORY APPROVAL

Reference: Section 8 – Quality Assurance Manual

Date: _____________________
Laboratory: ____
Address:____
____
Contact and e-mail address: ____
Phone: Direct ________________________________ Fax ______________________________

Requested Item[3]	**Date Received**	**Reviewed/ Accepted**	**Date**
1. Copy of State Certification[1]			
2. Insurance Certificate			
3. USDA Soil Permit			
4. Description of Ethics Program[3]			
5. QA Manual[3]			
6. Most Recent (and relevant) 2 Sets of WP/WS Reports with Corrective Action Response[1,3]			
7. State Audit with Corrective Action Response (or NELAC or A2LA Audit)[3]			
8. Sample Report[3]			
9. SOQ or Summary list of Technical Staff and Qualifications [3]			
10. SOPs for Methods to Be Loadshifted[2,3]			
11. For DoD Work: Statement that Lab quality system complies with QSM.			
12. For DoD Work: Approved by specific DoD Component laboratory approval process.			

1 - Required when emergency procedures are implemented.
2 - Some labs may not submit copies due to internal policies. In these cases, a copy of the first page and signature page of the SOP is acceptable. This requirement may also be fulfilled by supplying a table of SOPs with effective dates.
3 – If the laboratory has NELAC accreditation, Item #s 4 through 10 are not required.

On Site Audit Planned: YES NO If yes, Date Completed: __________ By Whom: ______________

Comments:

Lab Acceptable for Subcontracting Work: YES NO Limitations: _________________________

QA Manager (Signature): _____________________________________ Date: ________________
(Printed Name)

☐ Forwarded to Contract Coordinator, by: _______________________________ Date:________________

SECTION 9

PURCHASING SERVICES AND SUPPLIES
(NELAC 5.4.6)

9.1 OVERVIEW

Evaluation and selection of suppliers and vendors is performed, in part, on the basis of the quality of their products, their ability to meet the demand for their products on a continuous and short term basis, the overall quality of their services, their past history, and competitive pricing. This is achieved through evaluation of objective evidence of quality furnished by the supplier, which can include certificates of analysis, recommendations, and proof of historical compliance with similar programs for other clients. To ensure that quality critical consumables and equipment conform to specified requirements, all purchases from specific vendors are approved by a member of the supervisory or management staff.

Capital expenditures are made in accordance with TestAmerica's Corporate Controlled Purchases Procedure, SOP No. CW-F-S-007

Contracts will be signed in accordance with TestAmerica's Corporate Authorization Matrix Policy, Policy No. CW-F-P-002. Request for Proposals (RFP's) will be issued where more information is required from the potential vendors than just price. Process details are available in TestAmerica's Corporate Procurement and Contracts Policy(Policy No. CW-F-P-004). RFP's allow TestAmerica to determine if a vendor is capable of meeting requirements such as supplying all of the TestAmerica facilities, meeting required quality standards and adhering to necessary ethical and environmental standards. The RFP process also allows potential vendors to outline any additional capabilities they may offer.

9.2 GLASSWARE

Glassware used for volumetric measurements must be Class A or verified for accuracy according to laboratory procedure. Pyrex (or equivalent) glass should be used where possible. For safety purposes, thick-wall glassware should be used where available.

9.3 REAGENTS, STANDARDS & SUPPLIES

Purchasing guidelines for equipment and reagents must meet the requirements of the specific method and testing procedures for which they are being purchased. Solvents and acids are pre-tested in accordance with TestAmerica's Corporate SOP on Solvent & Acid Lot Testing & Approval, SOP No. CA-Q-S-001 and TestAmerica Buffalo SOP on Solvent Purity, SOP BF-OP-013.

9.3.1 Purchasing

Chemical reagents, solvents, glassware and general supplies are ordered as needed to maintain sufficient quantities on hand. Materials used in the analytical process must be of a known quality. The wide variety of materials and reagents available makes it advisable to specify recommendations for the name, brand, and grade of materials to be used in any determination. This information is contained in the method SOP. Purchase requisitions are placed into the J.D. Edwards system by designated departmental personnel. The listing of items available in the J.D. Edwards system has been approved for use by the corporate purchasing staff. Each purchase requisition receives final approval by the laboratory Operations Manager or purchasing coordinator before the order is submitted.

The analyst may also check the item out of the on-site consignment system that contains items approved for laboratory use.

9.3.2 Receiving

It is the responsibility of the purchasing coordinator to receive the shipment. It is the responsibility of the department that ordered the materials to date the material when received. Once the ordered reagents or materials are received, the department that submitted the order compares the information on the label or packaging to the original order to ensure that the purchase meets quality level specified. Material Safety Data Sheets (MSDSs) are available online through the Company's intranet website. Anyone may review these for relevant information on the safe handling and emergency precautions of on-site chemicals.

9.3.3 Specifications

All methods in use in the laboratory specify the grade of reagent that must be used in the procedure. If the quality of the reagent is not specified, it may be assumed that it is not significant in that procedure and, therefore, any grade reagent may be used. It is the responsibility of the analyst to check the procedure carefully for the suitability of grade of reagent.

Chemicals must not be used past the manufacturer's expiration date and must not be used past the expiration time noted in a method SOP. If expiration dates are not provided, the laboratory may contact the manufacturer to determine an expiration date.

The laboratory assumes a five year expiration date on inorganic dry chemicals unless noted otherwise by the manufacturer or by the reference source method. Chemicals should not be used past the manufacturer's or SOP expiration date unless 'verified' (refer to item 3 listed below).

- An expiration date can not be extended if the dry chemical is discolored or appears otherwise physically degraded, the dry chemical must be discarded.

- Expiration dates can be extended if the dry chemical is found to be satisfactory based on acceptable performance of quality control samples (Continuing Calibration Verification (CCV), Blanks, Laboratory Control Sample (LCS), etc.).

- If the dry chemical is used for the preparation of standards, the expiration dates can be extended 6 months if the dry chemical is compared to an unexpired independent source in performing the method and the performance of the dry chemical is found to be satisfactory. The comparison must show that the dry chemical meets CCV limits. The comparison studies are maintained along with the calibration raw data for which the reagent was used.

Wherever possible, standards must be traceable to national or international standards of measurement or to national or international reference materials. Records to that effect are available to the user.

Compressed gases in use are checked for pressure and secure positioning daily. The minimum total pressure must be 200 psig or the tank must be replaced. The quality of the gases must meet method or manufacturer specification or be of a grade that does not cause any analytical interference.

Water used in the preparation of standards or reagents must have a specific conductivity of less than 1- mmho/cm (or specific resistivity of greater than 1.0 megaohm-cm) at 25^{o}C. The specific conductivity is checked and recorded daily. If the water's specific conductivity is greater than the specified limit, the Facility Manager and appropriate Department Managers/Supervisors must be notified immediately in order to notify all departments, decide on cessation (based on intended use) of activities, and make arrangements for correction.

The laboratory may purchase reagent grade (or other similar quality) water for use in the laboratory. This water must be certified "clean" by the supplier for all target analytes or otherwise verified by the laboratory prior to use. This verification is documented.

Standard lots are verified before first time use if the laboratory switches manufacturers or has historically had a problem with the type of standard.

Purchased VOA vials must be certified clean and the certificates must be maintained. If uncertified VOA vials are purchased, all lots must be verified clean prior to use. This verification must be maintained.

Records of manufacturer's certification and traceability statements are maintained in files or binders in each laboratory section. These records include date of receipt, lot number (when applicable), and expiration date (when applicable). Incorporation of the item into the record indicates that the analyst has compared the new certificate with the previous one for the same purpose and that no difference is noted, unless approved and so documented by the Technical Director or QA Manager.

9.3.4 Storage

Reagent and chemical storage is important from the aspects of both integrity and safety. Light-sensitive reagents may be stored in brown-glass containers. Storage conditions are per the Corporate Environmental Health & Safety Manual (Corp. DOC No. CW-E-M-001) and method SOPs or manufacturer instructions.

9.4 PURCHASE OF EQUIPMENT/INSTRUMENTS/SOFTWARE

When a new piece of equipment is needed, either for additional capacity or for replacing inoperable equipment, the analyst or supervisor makes a supply request to the Technical Director and/or the Laboratory Director. If they agree with the request the procedures outlined in TestAmerica's Corporate Policy No. CA-T-P-001, Qualified Products List, are followed. A decision is made as to which piece of equipment can best satisfy the requirements. The appropriate written requests are completed and purchasing places the order.

Upon receipt of a new or used piece of equipment, an identification name is assigned and added to the equipment list. IT must also be notified so that they can synchronize the instrument for back-ups. Its capability is assessed to determine if it is adequate or not for the specific application. For instruments, a calibration curve is generated, followed by MDLs, Demonstration of Capabilities (DOCs), and other relevant criteria (refer to Section 19). For software, its operation must be deemed reliable and evidence of instrument verification must be retained by the IT Department or QA Department. Software certificates supplied by the vendors are filed with the LIMS Administrator. The manufacturer's operation manual is retained at the bench.

9.5 SERVICES

Service to analytical instruments (except analytical balances) is performed on an as needed basis. Routine preventative maintenance is discussed in Section 20. The need for service is determined by analysts and/or Department Managers. The service providers that perform the services are approved by the Department Managers, Operations Manager and/or Technical Director.

9.6 SUPPLIERS

TestAmerica selects vendors through a competitive proposal / bid process, strategic business alliances or negotiated vendor partnerships (contracts). This process is defined in the Corporate Finance documents on Vendor Selection (SOP No. CW-F-S-018) and Procurements & Contracts Policy (Policy No. CW-F-P-004). The level of control used in the selection process is dependent on the anticipated spending amount and the potential impact on TestAmerica business. Vendors that provide test and measuring equipment, solvents, standards, certified containers, instrument related service contracts or subcontract laboratory services shall be subject to more rigorous controls than vendors that provide off-the-shelf items of defined quality that meet the end use requirements. The JD Edwards purchasing system includes all suppliers /vendors that have been approved for use.

Evaluation of suppliers is accomplished by ensuring the supplier ships the product or material ordered and that the material is of the appropriate quality. This is documented by signing off on packing slips or other supply receipt documents. The purchasing documents contain the data that adequately describe the services and supplies ordered.

Any issues of vendor performance are to be reported immediately by the laboratory staff to the Corporate Purchasing Group by completing a Vendor Performance Report.

The Corporate Purchasing Group will work through the appropriate channels to gather the information required to clearly identify the problem and will contact the vendor to report the problem and to make any necessary arrangements for exchange, return authorization, credit, etc.

As deemed appropriate, the Vendor Performance Reports will be summarized and reviewed to determine corrective action necessary, or service improvements required by vendors

The laboratory has access to a listing of all approved suppliers of critical consumables, supplies and services. This information is provided through the JD Edwards purchasing system.

9.6.1 New Vendor Procedure

TestAmerica employees who wish to request the addition of a new vendor must complete a J.D. Edwards Vendor Add Request Form (available on the intranet site).

New vendors are evaluated based upon criteria appropriate to the products or services provided as well as their ability to provide those products and services at a competitive cost. Vendors are also evaluated to determine if there are ethical reasons or potential conflicts of interest with TestAmerica employees that would make it prohibitive to do business with them as well as their financial stability. The QA Department and/or the Technical Director are consulted with vendor and product selection that have an impact on quality.

SECTION 10

COMPLAINTS *(NELAC 5.4.8)*

10.1 OVERVIEW

The laboratory considers an effective client complaint handling processes to be of significant business and strategic value. Listening to and documenting client concerns captures 'client knowledge' that enables our operations to continually improve processes and client satisfaction. An effective client complaint handling process also provides assurance to the data user that the laboratory will stand behind its data, service obligations and products.

A client complaint is any expression of dissatisfaction with any aspect of our business services, e.g, communications, responsiveness, data, reports, invoicing and other functions expressed by any party, whether received verbally or in written form. Client inquiries, complaints or noted discrepancies are documented, communicated to management, and addressed promptly and thoroughly.

The laboratory has procedures for addressing with both external and internal complaints with the goal of providing satisfactory resolution to complaints in a timely and professional manner..

The nature of the complaint is identified, documented and investigated, and an appropriate action is determined and taken. In cases where a client complaint indicates that an established policy or procedure was not followed, the QA Department must evaluate whether a special audit must be conducted to assist in resolving the issue. A written confirmation or letter to the client, outlining the issue and response taken is recommended as part of the overall action taken.

The process of complaint resolution and documentation utilizes the procedures outlined in Section 12 (Corrective Actions) and is documented following the laboratory SOPs related to Data Quality Review (BF-QA-006) and Corrective Action (BF-QA-005).

10.2 EXTERNAL COMPLAINTS

An employee that receives a complaint initiates the complaint resolution process by first documenting the complaint according to SOPs BF-QA-006 and BF-QA-005.

Complaints fall into two categories: correctable and non-correctable. An example of a correctable complaint would be one where a report re-issue would resolve the complaint. An example of a non-correctable complaint would be one where a client complains that their data was repeatedly late. Non-correctable complaints should be reviewed for preventive action measures to reduce the likely hood of future occurrence and mitigation of client impact.

The general steps in the complaint handling process are:

- Receiving and Documenting Complaints
- Complaint Investigation and Service Recovery
- Process Improvement

The laboratory shall inform the initiator of the complaint of the results of the investigation and the corrective action taken, if any.

10.3 INTERNAL COMPLAINTS

Internal complaints include, but are not limited to: errors and non-conformances, training issues, internal audit findings, and deviations from methods. Corrective actions may be initiated by any staff member who observes a nonconformance and shall follow the procedures outlined in Section 13. In addition, Corporate Management, Sales and Marketing and Information Technology (IT) may initiate a complaint by contacting the laboratory or through the corrective action system described in Section 12.

10.4 MANAGEMENT REVIEW

The number and nature of client complaints is reported by the QA Manager to the laboratory and QA Director in the QA Monthly report. Monitoring and addressing the overall level and nature of client complaints and the effectiveness of the solutions is part of the Annual Management Review (Section 16)

SECTION 11

CONTROL OF NON-CONFORMING WORK
(NELAC 5.4.9)

11.1 OVERVIEW

When data discrepancies are discovered or deviations and departures from laboratory standard procedures, policies and/or client requests have occurred, corrective action is taken immediately. First, the laboratory evaluates the significance of the nonconforming work. Then, a corrective action plan is initiated based on the outcome of the evaluation. If it is determined that the nonconforming work is an isolated incident, the plan could be as simple as adding a qualifier to the final results and/or making a notation in the case narrative. If it is determined that the nonconforming work is a systematic or improper practices issue, the corrective action plan could include a more in depth investigation and a possible suspension of an analytical method. In all cases, the actions taken are documented using the laboratory's corrective action system (refer to Section 12).

Due to the frequently unique nature of environmental samples, sometimes departures from documented policies and procedures are needed. When an analyst encounters such a situation, the problem is presented to the department manager for resolution. The department manager may elect to discuss it with the Technical Director, QA Manager or have a representative contact the client to decide on a logical course of action. Once an approach is agreed upon, the analyst documents it using the laboratory's job exception and corrective action system described in Section 12. This information can then be supplied to the client in the form of a footnote or a case narrative with the report.

Project Management may encounter situations where a client may request that a special procedure be applied to a sample that is not standard lab practice. Based on a technical evaluation, the lab may accept or opt to reject the request based on technical or ethical merit. An example might be the need to report a compound that the lab does not normally report. The lab would not have validated the method for this compound following the procedures in Section 19. The client may request that the compound be reported based only on the calibration. Such a request would need to be approved by the Laboratory Director, Technical Director, Operations Manager or QA Manager, documented and included in the project folder. Deviations must also be noted on the final report with a statement that the compound is not reported in compliance with the analytical method requirements and the reason.

11.2 RESPONSIBILITIES AND AUTHORITIES

TestAmerica's Corporate SOP entitled Internal Investigation of Potential Data Discrepancies and Determination for Data Recall (SOP No. CA-L-S-001), outlines the general procedures for the reporting and investigation of data discrepancies and alleged incidents of misconduct or violations of TestAmerica's data integrity policies as well as the policies and procedures related to the determination of the potential need to recall data.

Under certain circumstances the Laboratory Director, the Technical Director, the Operations Manager or the QA Manager may exceptionally authorize departures from documented procedures or policies. The departures may be a result of procedural changes due to the nature of the sample; a one-time procedure for a client; QC failures with insufficient sample to reanalyze, etc. In most cases, the client will be informed of the departure prior to the reporting of the data. Any departures must be well documented using the laboratory's job exception and corrective action procedures described in Section 12. This information may also need to be documented in logbooks and/or data review checklists as appropriate***.*** Any impacted data must be referenced in a case narrative and/or flagged with an appropriate data qualifier.

Any misrepresentation or possible misrepresentation of analytical data discovered by any laboratory staff member must be reported to facility senior laboratory management within 24-hours. The Senior Management staff is comprised of the Laboratory Director, Technical Director, Operations Manager, QA Manager, Customer Service Manager, Human Resources Manager and Business Development Manager. Suspected misrepresentation issues may also be reported to any member of the corporate staff as identified in Ethics Policy, CA-L-P-001. The data integrity hotline (1-800-736-9407) may also be used. The reporting of issues involving alleged violations of the company's Data Integrity or Manual Integration procedures must be conveyed to an Ethics and Compliance Officer (ECO), Director of Quality & Client Advocacy and the laboratory's Quality Director within 24 hours of discovery.

Whether an inaccurate result was reported due to calculation or quantitation errors, data entry errors, improper practices, or failure to follow SOPs, the data must be evaluated to determine the possible effect.

The Laboratory Director, QA Manager, ECOs, Corporate Quality, the COO, General Managers and the Quality Directors have the authority and responsibility to halt work, withhold final reports, or suspend an analysis for due cause as well as authorize the resumption of work.

11.3 EVALUATION OF SIGNIFICANCE AND ACTIONS TAKEN

For each nonconforming issue reported, an evaluation of its significance and the level of management involvement needed is made. This includes reviewing its impact on the final data, whether or not it is an isolated or systematic issue, and how it relates to any special client requirements.

TestAmerica's Corporate Data Investigation & Recall Procedure (SOP No. CA-L-S-001 distinguishes between situations when it would be appropriate for laboratory management to make the decision on the need for client notification (written or verbal) and data recall (report revision) and when the decision must be made with the assistance of the ECO's and Corporate Management. Laboratory level decisions are documented and approved using the laboratory's standard nonconformance/corrective action reporting in lieu of the data recall determination form contained in TestAmerica's Corporate SOP No. CA-L-S-001.

11.4 PREVENTION OF NONCONFORMING WORK

If it is determined that the nonconforming work could recur, further corrective actions must be made following the laboratory's corrective action system.

On a monthly basis, the QA Department evaluates non-conformances to determine if any nonconforming work has been repeated multiple times. If so, the laboratory's corrective action process may be followed.

11.5 METHOD SUSPENSION/RESTRICTION (STOP WORK PROCEDURES)

In some cases it may be necessary to suspend/restrict the use of a method or target compound which constitutes significant risk and/or liability to the laboratory. Suspension/restriction procedures can be initiated by any of the persons noted in Section 11.2, Paragraph 5.

Prior to suspension/restriction, confidentiality will be respected, and the problem with the required corrective and preventive action will be stated in writing and presented to the Laboratory Director.

The Laboratory Director shall arrange for the appropriate personnel to meet with the QA Manager as needed. This meeting shall be held to confirm that there is a problem, that suspension/restriction of the method is required and will be concluded with a discussion of the steps necessary to bring the method/target or test fully back on line. In some cases that may not be necessary if all appropriate personnel have already agreed there is a problem and there is agreement on the steps needed to bring the method, target or test fully back on line.

The QA Manager will also initiate a corrective action report as described in Section 12 if one has not already been started. A copy of any meeting notes and agreed upon steps should be faxed or e-mailed by the laboratory to the appropriate General Manager and member of Corporate QA. This fax/e-mail acts as notification of the incident.

After suspension/restriction, the lab will hold all reports to clients pending review. No faxing, mailing or distributing through electronic means may occur. The report must not be posted for viewing on the internet. It is the responsibility of the Laboratory Director to hold all reporting and to notify all relevant laboratory personnel regarding the suspension/restriction (i.e., Project Management, Log-in, etc…). Clients will NOT generally be notified at this time. Analysis may proceed in some instances depending on the non-conformance issue.

Within 72 hours, the QA Manager will determine if compliance is now met and reports can be released, OR determine the plan of action to bring work into compliance, and release work. A team, with all principals involved (Laboratory Director, Technical Director, Operations Manager, QA Manager, Department Manager) can devise a start-up plan to cover all steps from client notification through compliance and release of reports. Project Management and the Customer Service Manager and Sales and Marketing must be notified if clients must be notified or if the suspension/restriction affects the laboratory's ability to accept work. The QA Manager must approve start-up or elimination of any restrictions after all corrective action is complete. This approval is given by final signature on the completed corrective action report.

SECTION 12

CORRECTIVE ACTION
(NELAC 5.4.10)

12.1 OVERVIEW

A major component of TestAmerica's Quality Assurance (QA) Program is the problem investigation and feedback mechanism designed to keep the laboratory staff informed on quality related issues and to provide insight to problem resolution. When nonconforming work or departures from policies and procedures in the quality system or technical operations are identified, the corrective action procedure provides a systematic approach to assess the issues, restore the laboratory's system integrity, and prevent reoccurrence. Corrective actions are documented using Non-Conformance Report (NCR) also know as Job Exception Reports (JER) and Corrective Action Reports (CAR) (refer to Figure 12-1).

12.2 GENERAL

Problems within the quality system or within analytical operations may be discovered in a variety of ways, such as QC sample failures, internal or external audits, proficiency testing (PT) performance, client complaints, staff observation, etc..

The purpose of a corrective action system is to:

- Identify non-conformance events and assign responsibility for investigating.
- Resolve non-conformance events and assign responsibility for any required corrective action.
- Identify Systematic Problems before they become serious.
- Identify and track client complaints and provide resolution

12.2.1 Non-Conformance Report (NCR) - (previously known as Job Exception Report and Data Quality Review (DQR) - is used to document the following types of corrective actions:

- Deviations from an established procedure or SOP
- QC outside of limits (non matrix related)
- Isolated reporting / calculation errors
- Client complaints
- Project Management concerns regarding specific analytical results

12.2.2 Corrective Action Report (CAR) - is used to document the following types of corrective actions:

- Questionable trends that are found in the monthly review of JERs.
- Issues found while reviewing JERs that warrant further investigation.
- Questionable trends that are found in the monthly review of DQRs or client complaints

- Internal and External Audit Findings
- Failed or Unacceptable PT results.
- Corrective actions that cross multiple departments in the laboratory.
- Systematic Reporting / Calculation Errors

12.3 CLOSED LOOP CORRECTIVE ACTION PROCESS

Any employee in the company can initiate a corrective action. There are four main components to a closed-loop corrective action process once an issue has been identified: Cause Analysis, Selection and Implementation of Corrective Actions (both short and long term), Monitoring of the Corrective Actions, and Follow-up.

12.3.1 Cause Analysis

- Upon discovery of a non-conformance event, the event must be defined and documented. A NCR or CAR must be initiated, someone is assigned to investigate the issue and the event is investigated for cause. Table 12-1 provides some general guidelines on determining responsibility for assessment.
- The cause analysis step is the key to the process as a long term corrective action cannot be determined until the cause is determined.
- If the cause is not readily obvious, the Department Manager, Operations Manager, Technical Director, or QA Manager (or QA designee) is consulted.

12.3.2 Selection and Implementation of Corrective Actions

- Where corrective action is needed, the laboratory shall identify potential corrective actions. The action(s) most likely to eliminate the problem and prevent recurrence are selected and implemented. Responsibility for implementation is assigned.
- Corrective actions shall be to a degree appropriate to the magnitude of the problem identified through the cause analysis.
- Whatever corrective action is determined to be appropriate, the laboratory shall document and implement the changes. The NCR or CAR is used for this documentation.

12.3.3 Monitoring of the Corrective Actions

- The Department Manager, Operations Manager and QA Manager are responsible to ensure that the corrective action taken was effective.
- Ineffective actions are documented and re-evaluated until acceptable resolution is achieved. Department Managers and the Operations Manager are accountable to the Laboratory Director to ensure final acceptable resolution is achieved and documented appropriately.
- Each NCR and DQR are entered into a database and each CAR is entered into a spreadsheet for tracking purposes and a monthly summary of all corrective actions is printed out for review to aid in ensuring that the corrective actions have taken effect.
- The QA Manager reviews monthly NCR and CARs for trends. Highlights are included in the QA monthly report (refer to Section 16). If a significant trend develops that adversely affects quality, an audit of the area is performed and corrective action implemented.

- Any out-of-control situations that are not addressed acceptably at the laboratory level may be reported to the Corporate Quality Director by the QA Manager, indicating the nature of the out-of-control situation and problems encountered in solving the situation.

12.3.4 Follow-up Audits

- Follow-up audits may be initiated by the QA Manager and shall be performed as soon as possible when the identification of a nonconformance casts doubt on the laboratory's compliance with its own policies and procedures, or on its compliance with state or federal requirements.
- These audits often follow the implementation of the corrective actions to verify effectiveness. An additional audit would only be necessary when a critical issue or risk to business is discovered.
- Also refer to Section 15.1.4, Special Audits)

12.4 TECHNICAL CORRECTIVE ACTIONS

In addition to providing acceptance criteria and specific protocols for technical corrective actions in the method SOPs the laboratory has general procedures to be followed to determine when departures from the documented policies and procedures and quality control have occurred (refer to Section 11). The documentation of these procedures is through the use of a NCR or CAR.

Table 12-1 includes examples of general technical corrective actions. For specific criteria and corrective actions refer to the analytical methods or specific method SOPs. The laboratory may also maintain work instructions on these items that are available upon request.

Table 12-1 provides some general guidelines for identifying the individual(s) responsible for assessing each QC type and initiating corrective action. The table also provides general guidance on how a data set should be treated if associated QC measurements are unacceptable. Specific procedures are included in Method SOPs, work instructions, QAM Sections 19 and 20. All corrective actions are reviewed monthly at a minimum by the QA Manager and highlights are included in the QA monthly report.

To the extent possible, samples shall be reported only if all quality control measures are acceptable. If the deficiency does not impair the usability of the results, data will be reported with an appropriate data qualifier and/or the deficiency will be noted in the case narrative. Where sample results may be impaired, the Project Manager is notified by an NCR and appropriate corrective action (e.g., reanalysis) is taken and documented.

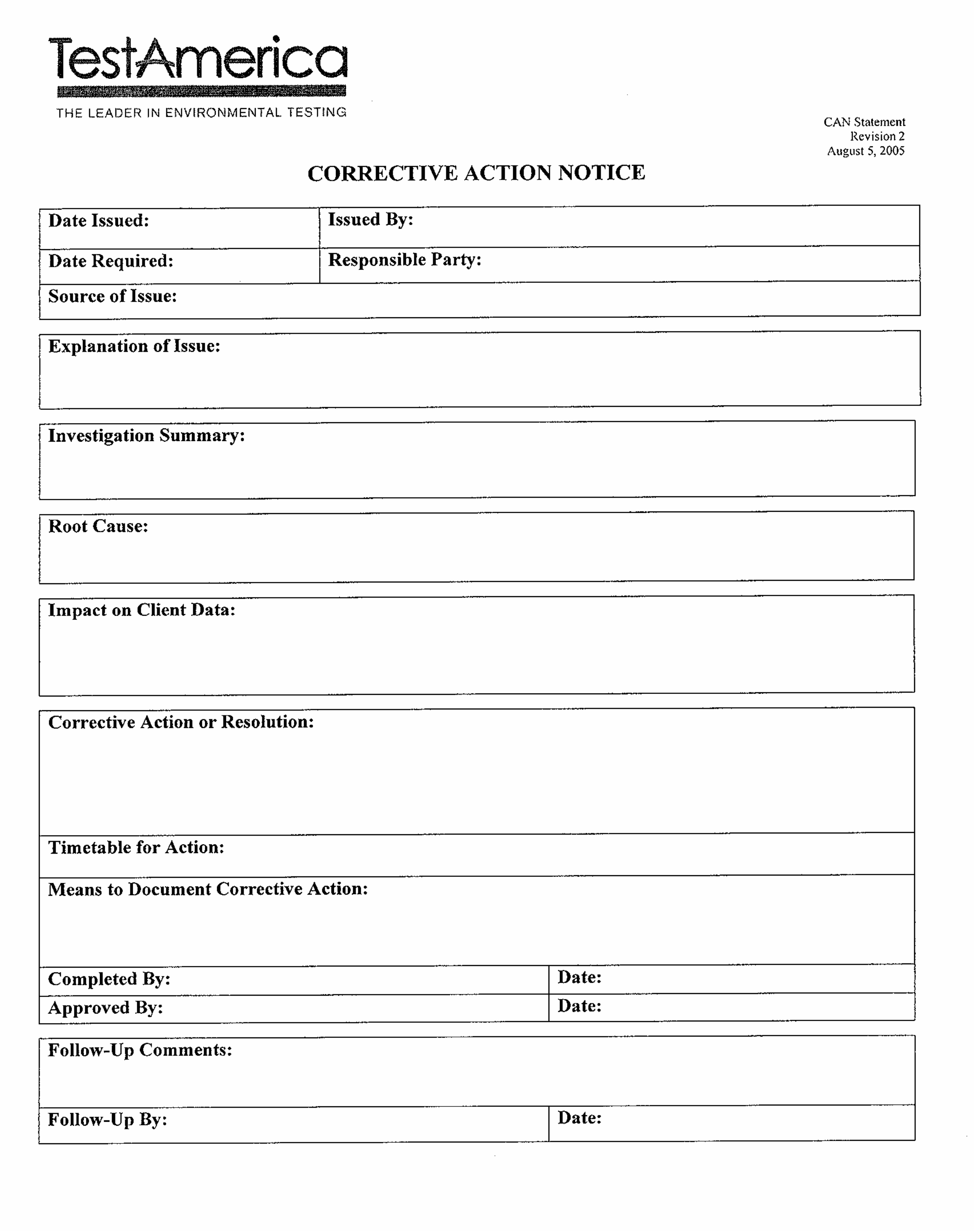
12.5 BASIC CORRECTIONS

When mistakes occur in records, each mistake shall be crossed-out, not obliterated (e.g. no white-out), and the correct value entered alongside. All such corrections shall be initialed (or signed) and dated by the person making the correction. In the case of records stored electronically, the original "uncorrected" file must be maintained intact and a second "corrected" file is created.

This same process applies to adding additional information to a record. All additions made later than the initial must also be initialed (or signed) and dated.

When corrections are due to reasons other than obvious transcription errors, the reason for the corrections (or additions) shall also be documented.

Figure 12-1.
Example – Corrective Action Notice



TestAmerica
THE LEADER IN ENVIRONMENTAL TESTING

CAN Statement
Revision 2
August 5, 2005

CORRECTIVE ACTION NOTICE

Date Issued:	**Issued By:**
Date Required:	**Responsible Party:**
Source of Issue:	

Explanation of Issue:

Investigation Summary:

Root Cause:

Impact on Client Data:

Corrective Action or Resolution:

Timetable for Action:

Means to Document Corrective Action:

Completed By:	**Date:**
Approved By:	**Date:**

Follow-Up Comments:

Follow-Up By:	**Date:**

Table 12-1.

Example – General Corrective Action Procedures

QC Activity ***(Individual Responsible for Initiation/Assessment)***	**Acceptance Criteria**	**Recommended Corrective Action**
Initial Instrument Blank *(Analyst)*	- Instrument response < MDL.	- Prepare another blank. - If same response, determine cause of contamination: reagents, environment, instrument equipment failure, etc..
Initial Calibration Standards *(Analyst, Department Manager)*	- Correlation coefficient > 0.99 or standard concentration value. - % Recovery within acceptance range. - See details in Method SOP.	- Reanalyze standards. - If still unacceptable, remake standards and recalibrate instrument.
Independent Calibration Verification (Second Source) *(Analyst, Department Manager)*	- % Recovery within control limits.	- Remake and reanalyze standard. - If still unacceptable, then remake calibration standards or use new primary standards and recalibrate instrument.
Continuing Calibration Standards *(Analyst, Data Reviewer)*	% Recovery within control limits.	- Reanalyze standard. - If still unacceptable, then recalibrate and rerun affected samples.
Matrix Spike / Matrix Spike Duplicate (MS/MSD) *(Analyst, Data Reviewer)*	- % Recovery within limits documented in LIMs.	- If the acceptance criteria for duplicates or matrix spikes are not met because of matrix interferences, the acceptance of the analytical batch is determined by the validity of the LCS. - If the LCS is within acceptable limits the batch is acceptable. - The results of the duplicates, matrix spikes and the LCS are reported with the data set.
Laboratory Control Sample (LCS) *(Analyst, Data Reviewer)*	- % Recovery within limits specified in LIMs.	- Batch must be re-prepared and re-analyzed. **Note:** If there is insufficient sample or the holding time cannot be met, contact client and report with flags.

QC Activity ***(Individual Responsible for Initiation/Assessment)***	**Acceptance Criteria**	**Recommended Corrective Action**
Surrogates *(Analyst, Data Reviewer)*	- % Recovery within limits of method or within three standard deviations of the historical mean.	- Individual sample must be repeated. Place comment in LIMS.
Method Blank (MB) *(Analyst, Data Reviewer)*	< Reporting Limit [1]	- Reanalyze blank. - If still positive, determine source of contamination. If necessary, reprocess (i.e. digest or extract) entire sample batch. Report blank results.
Proficiency Testing (PT) Samples *(QA Manager, Department Manager)*	- Criteria supplied by PT Supplier.	- Any failures or warnings must be investigated for cause. Failures may result in the need to repeat a PT sample to show the problem is corrected.
Internal / External Audits *(QA Manager, Department Manager, Operations Manager, Technical Director, Laboratory Director)*	- Defined in Quality System documentation such as SOPs, QAM, etc..	- Non-conformances must be investigated through CAR system and necessary corrections must be made.
Reporting / Calculation Errors *(Depends on issue – possible individuals include: Analysts, Data Reviewers, Project Managers, Department Manager, QA Manager, Corporate QA, Corporate Management)*	- SOP CA-L-S-001, Internal Investigation of Potential Data Discrepancies and Determination for Data Recall.	- Corrective action is determined by type of error. Follow the procedures in SOP CA-L-S-001.
Client Complaints *(Project Managers, Lab Director, Sales and Marketing, QA Manager)*	-	- Corrective action is determined by the type of complaint. For example, a complaint regarding an incorrect address on a report will result in the report being corrected and then follow-up must be performed on the reasons the address was incorrect (e.g., database needs to be updated).

QC Activity ***(Individual Responsible for Initiation/Assessment)***	**Acceptance Criteria**	**Recommended Corrective Action**
QA Monthly Report (Refer to Section 17 for an example) *(QA Manager, Lab Director, Operations Manager Department Managers)*	- QAM, SOPs.	- Corrective action is determined by the type of issue. For example, CARs for the month are reviewed and possible trends are investigated.
Health and Safety Violation *(EH&S Coordinator, Lab Director, Operations Manager, Department Manager)*	- Environmental Health and Safety (EHS) Manual.	- Non-conformance is investigated and corrected through EH&S office.

Note:

1. Except as noted below for certain compounds, the method blank should be below the reporting limit. Concentrations up to five times the reporting limit will be allowed for the ubiquitous laboratory and reagent contaminants: methylene chloride, acetone, 2-butanone and phthalates provided they appear in similar levels in the reagent blank and samples. This allowance presumes that the reporting limit is significantly below any regulatory limit to which the data are to be compared and that blank subtraction will not occur. For benzene and ethylene dibromide (EDB) and the other analytes for which regulatory limits are extremely close to the detection limit, the method blank must be below the method detection limit.

.

SECTION 13.0

PREVENTIVE ACTION
(NELAC 5.4.11)

13.1 OVERVIEW

The laboratory's preventive action programs improve, or eliminate potential causes of nonconforming product and/or nonconformance to the quality system. This preventive action process is a proactive continuous process improvement activity that can be initiated through feedback from clients, employees, business providers, and affiliates. The QA Department has the overall responsibility to ensure that the preventive action process is in place, and that relevant information on actions is submitted for management review.

Dedicating resources to an effective preventive action system emphasizes the laboratory's commitment to its Quality Program. It is beneficial to identify and address negative trends before they develop into complaints, problems and corrective actions. Additionally, customer service and satisfaction can be improved through continuous improvements to laboratory systems.

Opportunities for improvement may be discovered during management reviews, the QA Metrics Report, internal or external audits, proficiency testing performance, client complaints, staff observation, etc..

The monthly QA Metrics Report shows performance indicators in all areas of the quality system. These areas include revised reports, corrective actions, audit findings, internal auditing and data authenticity audits, client complaints, PT samples, holding time violations, SOPs, ethics training, etc. These metrics are used to help evaluate quality system performance on an ongoing basis and provide a tool for identifying areas for improvement.

The laboratory's Corrective Action process is integral to implementation of preventive actions. A critical piece of the corrective action process is the implementation of actions to prevent further occurrence of a non-compliance event. Historical review of corrective action provides a valuable mechanism for identifying preventive action opportunities.

13.1.1 The following elements are part of a preventive action system:

- Identification of an opportunity for preventive action.
- Process for the preventive action.
- Define the measurements of the effectiveness of the process once undertaken.
- Execution of the preventive action.
- Evaluation of the plan using the defined measurements.
- Verification of the effectiveness of the preventive action.
- Close-Out by documenting any permanent changes to the Quality System as a result of the Preventive Action. Documentation of Preventive Action is incorporated into the monthly QA reports, corrective action process and management review

13.1.2 Any Preventive Actions undertaken or attempted shall be taken into account during the Annual Management Review (Section 17). A highly detailed recap is not required; a simple recount of success and failure within the preventive action program will provide management a measure for evaluation.

13.2 MANAGEMENT OF CHANGE

The Management of Change process is designed to manage significant events and changes that occur within the laboratory. Through these procedures, the potential risks inherent with a new event or change are identified and evaluated. The risks are minimized or eliminated through pre-planning and the development of preventive measures. The types of changes covered under this system include: Facility Changes, Major Accreditation Changes, Addition or Deletion to Division's Capabilities or Instrumentation, Key Personnel Changes, Laboratory Information Management System (LIMS) changes.

SECTION 14.0

CONTROL OF RECORDS
(NELAC 5.4.12)

The laboratory maintains a record system appropriate to its needs and that complies with applicable standards or regulations as required. The system produces unequivocal, accurate records that document all laboratory activities. The laboratory retains all original observations, calculations and derived data, calibration records and a copy of the analytical report for a minimum of five years after it has been issued. TestAmerica Buffalo SOP BF-GP-015, Record Storage and Retention specify additional storage, archiving and retention procedures.

14.1 OVERVIEW

The laboratory has established procedures for identification, collection, indexing, access, filing, storage, maintenance and disposal of quality and technical records. A record index is listed in Table 14-1. Quality records are maintained by the QA department in a database which is backed up as past of the regular laboratory backup. Records are of two types; either electronic or hard copy paper formats depending on whether the record is computer or hand generated (some records may be in both formats). Hardcopy technical records are maintained by the Data Deliverables Manager while electronic technical records are maintained by the IT Administrator.

Table 14-1. Record Index[1]

	Record Types [1]**:**	**Retention Time:**
Technical Records	- Raw Data - Logbooks[2] - Standards - Certificates - Analytical Records - Lab Reports	5 Years from analytical report issue*
Official Documents	- Quality Assurance Manual (QAM) - Work Instructions - Policies - SOPs - Manuals	5 Years from document retirement date*
QA Records	- Internal & External Audits/Respones - Certifications - Corrective/Preventive Actions - Management Reviews - Method & Software Validation / Verification Data - Data Investigation	5 Years from archival* **Data Investigation:** 5 years or the life of the affected raw data storage whichever is greater (beyond 5 years if ongoing project or pending investigation)

	Record Types [1]:	**Retention Time:**
Project Records	- Sample Receipt & COC Documentation - Contracts and Amendments - Correspondence - QAPP -SAP - Telephone Logbooks - Lab Reports	5 Years from analytical report issue*
Administrative Records	Finance and Accounting	10 years
	EH&S Manual, Permits, Disposal Records	7 years
	Employee Handbook	Indefinitely
	Personnel files, Employee Signature & Initials, Administrative Training Records (e.g., Ethics)	7 Years (HR Personnel Files must be maintained indefinitely
	Administrative Policies Technical Training Records	7 years

[1] Record Types encompass hardcopy and electronic records.
[2] Examples of Logbook types: Maintenance, Instrument Run, Preparation (standard and samples), Standard and Reagent Receipt, Archiving, Balance Calibration, Temperature (hardcopy or electronic records).
* Exceptions listed in Table 14-2.

14.1.1 All records are stored and retained in such a way that they are secure and readily retrievable at the laboratory facility or an offsite location that provides a suitable environment to prevent damage or deterioration and to prevent loss. Retention of records is maintained on-site at the laboratory for at least 3 months after their generation and moved offsite for the remainder of the required storage time. Records are maintained for a minimum of five years unless other wise specified by a client or regulatory requirement. All records hall be protected against fire, theft, loss, environmental deterioration and vermin. In the case of electronic records, electronic or magnetic sources, storage media are protected from deterioration caused by magnetic fields and/or electronic deterioration. Access to the data is limited to laboratory and company employees.

For raw data and project records, record retention shall be calculated from the date the project report is issued. For other records, such as Controlled Documents, QA, or Administrative Records, the retention time is calculated from the date the record is formally retired. Records related to the programs listed in Table 14-2 have lengthier retention requirements and are subject to the requirements in Section 14.1.3.

14.1.2 Programs with Longer Retention Requirements

Some regulatory programs have longer record retention requirements than the standard record retention time. These are detailed in Table 14-2 with their retention requirements. In these cases, the longer retention requirement is enacted. If special instructions exist such that client data cannot be destroyed prior to notification of the client, the container or box containing that data is marked as to who to contact for authorization prior to destroying the data. Specific Information related to archival of data for greater than 5 years may be found in TestAmerica Buffalo SOP BF-GP-015.

Table 14-2. Special Record Retention Requirements

Program	**[1]Retention Requirement**
Drinking Water – All States	10 years (project records)
Drinking Water Lead and Copper Rule	12 years (project records)
Commonwealth of MA – All environmental data 310 CMR 42.14	10 years
FIFRA – 40 CFR Part 160	Retain for life of research or marketing permit for pesticides regulated by EPA
Housing and Urban Development (HUD) Environmental Lead Testing	10 years
Alaska	10 years
Louisiana – All	10 years
Michigan Department of Environmental Quality – all environmental data	10 years
Navy Facilities Engineering Service Center (NFESC)	5 years
NY Potable Water NYCRR Part 55-2	10 years
TSCA - 40 CFR Part 792	10 years after publication of final test rule or negotiated test agreement

[1]Note: Extended retention requirements are noted with the archive documents or addressed in TestAmerica Buffalo facility-specific records retention procedure BF-GP-015.

14.1.3 All records are held secure and in confidence. Records maintained at the laboratory are located in the locked on-site storage room. Records archived off-site are stored in a secure location. Access to the off-site storage facility is controlled and logs are maintained for the documented removal/return of records

14.1.4 The laboratory has procedures to protect and back-up records stored electronically and to prevent unauthorized access to or amendment of these records. All analytical data is maintained as hard copy or in a secure readable electronic format. TestAmerica Buffalo SOP BF-GP-015 also contains specific information for archival of scanned data.

14.1.5 The record keeping system allows for historical reconstruction of all laboratory activities that produced the analytical data, as well as rapid recovery of historical data (records stored off site should be accessible within 2 business days of a request for such records). The history of the sample from when the laboratory took possession of the samples must be readily understood through the documentation. This shall include inter-laboratory transfers of samples and/or extracts.

- The records include the identity of personnel involved in sampling, sample receipt, preparation, or testing. All analytical work contains the initials (at least) of the personnel involved. The laboratory's copy of the chain of custody is stored with the project file and the Job Number analytical service request form (ASRF) generated by the LIMS. The chain of custody would indicate the name of the sampler. If any sampling notes are provided with a work order, they are kept with this package.

- All information relating to the laboratory facilities equipment, analytical test methods, and related laboratory activities, such as sample receipt, sample preparation, or data verification are documented.

- The record keeping system facilitates the retrieval of all working files and archived records for inspection and verification purposes (e.g., set format for naming electronic files, set format for what is included with a given analytical data set. Instrument data is stored sequentially by instrument. Calibration data for a given sequence are maintained in the order of the analysis. Sample data are stored on a job number basis in the project file or as part of the daily batch or sequence. Run logs are maintained for each instrument or method; a copy of each day's run log or instrument sequence is stored with the data to aid in re-constructing an analytical sequence. Where an analysis is performed without an instrument, bound logbooks, bench sheets or excel spreadsheets are used to record and file data. Standard and reagent information is recorded in logbooks or on the raw data for each method as required.

- Changes to hardcopy records shall follow the procedures outlined in Section 13 and 20. Changes to electronic records in LIMS or instrument data are recorded in audit trails.

- The reason for a signature or initials on a document is clearly indicated in the records such as "sampled by," "prepared by," "reviewed by", or "analyzed by".

- All generated data except those that are generated by automated data collection systems, are recorded directly, promptly and legibly in permanent dark ink.

- Hard copy data may be scanned into PDF format for record storage as long as the scanning process can be verified in order to ensure that no data is lost and the data files and storage media must be tested to verify the laboratory's ability to retrieve the information prior to the destruction of the hard copy that was scanned. The procedure for this verification can be found in TestAmerica SOP BF-GP-015.

- Also refer to Section 19.14.1 'Computer and Electronic Data Related Requirements'**.**

14.2 TECHNICAL AND ANALYTICAL RECORDS

14.2.1 The laboratory retains records of original observations, derived data and sufficient information to establish an audit trail, calibration records, staff records and a copy of each analytical report issued, for a minimum of five years unless otherwise specified by a client or regulatory requirement. The records for each analysis shall contain sufficient information to enable the analysis to be repeated under conditions as close as possible to the original. The records shall include the identity of laboratory personnel responsible for the sampling, performance of each analysis and reviewing of results.

14.2.2 Observations, data and calculations are recorded real-time.

14.2.3 Changes to hardcopy records shall follow the procedures outlined in Section 13 and 20. Changes to electronic records in LIMS or instrument data are recorded in audit trails.
The essential information to be associated with analysis, such as strip charts, tabular printouts, computer data files, analytical notebooks, and run logs, include:

- laboratory sample ID code**;**
- Date of analysis; time of analysis is also required if the holding time is seventy-two (72) hours or less, or when time critical steps are included in the analysis (e.g., drying times, incubations, etc.); instrumental analyses have the date and time of analysis recorded as part of their general operations. Where a time critical step exists in an analysis, location for such a time is included as part of the documentation in a specific logbook or on a bench sheet.
- Instrumentation identification and instrument operating conditions/parameters. Operating conditions/parameters are typically recorded in the method specific SOPs, in the instrument method detail records or the instrument maintenance logs where available.
- analysis type**;**
- all manual calculations and manual integrations**;**
- analyst's or operator's initials/signature**;**
- sample preparation including cleanup, separation protocols, incubation periods, ID codes, volumes, weights, instrument printouts, meter readings, temperatures, calculations, reagents**;**
- test results**;**
- standard and reagent origin, receipt, preparation, and use**;**
- calibration criteria, frequency and acceptance criteria**;**
- data and statistical calculations, review, confirmation, interpretation, assessment and reporting conventions**;**
- quality control protocols and assessment**;**
- electronic data security, software documentation and verification, software and hardware audits, backups, and records of any changes to automated data entries; and
- Method performance criteria including expected quality control requirements. These are indicated both in the LIMS and on specific analytical report formats**.**

14.3 <u>LABORATORY SUPPORT ACTIVITIES</u>

In addition to documenting all the above-mentioned activities, the following are retained QA records and project records (previous discussions in this section relate where and how these data are stored):

- all original raw data, whether hard copy or electronic, for calibrations, samples and quality control measures, including analysts' work sheets and data output records (chromatograms, strip charts, and other instrument response readout records)**;**
- a written description or reference to the specific test method used which includes a description of the specific computational steps used to translate parametric observations into a reportable analytical value**;**
- copies of final reports**;**
- archived SOPs**;**
- correspondence relating to laboratory activities for a specific project**;**
- all corrective action reports, audits and audit responses**;**
- proficiency test results and raw data; and
- results of data review, verification, and crosschecking procedures

14.3.2 Sample Handling Records

Records of all procedures to which a sample is subjected while in the possession of the laboratory are maintained. These include but are not limited to records pertaining to:

- sample preservation including appropriateness of sample container and compliance with holding time requirement;
- sample identification, receipt, acceptance or rejection and login;
- sample storage and tracking including shipping receipts, sample transmittal / COC forms; and
- Procedures for the receipt and retention of samples, including all provisions necessary to protect the integrity of samples.

14.4 ADMINISTRATIVE RECORDS

The laboratory also maintains the administrative records in either electronic or hard copy form. Refer to Table 14-1.

14.5 RECORDS MANAGEMENT, STORAGE AND DISPOSAL

14.5.1 All records (including those pertaining to test equipment), certificates and reports are safely stored, held secure and in confidence to the client. Certification related records are available upon request.

14.5.2 All information necessary for the historical reconstruction of data is maintained by the laboratory. Records that are stored only on electronic media must be supported by the hardware and software necessary for their retrieval.

14.5.3 Records that are stored or generated by computers or personal computers have hard copy, write-protected backup copies, or an electronic audit trail controlling access**.**

14.5.4 The laboratory has a record management system (also known as document control) for control of laboratory notebooks, instrument logbooks, standards logbooks, and records for data reduction, validation, storage and reporting. Laboratory notebooks are issued on a per instrument or analysis basis, and are numbered sequentially as they are issued. No instrument or analysis has more than one active notebook at a time, so all data are recorded sequentially within a series of sequential notebooks. Bench sheets and raw data sequence files are filed sequentially by date. Standard and reagent information is maintained in logbooks which are maintained on a departmental basis and are numbered sequentially as they are issued or as they are archived by QA.

14.5.5 Records are considered archived when noted as such in the records management system. Access to archived hard-copy information is documented with an access log and in/out records is used to note data that is removed and returned.

14.5.6 Transfer of Ownership

In the event that the laboratory transfers ownership or goes out of business, the laboratory shall ensure that the records are maintained or transferred according to client's instructions. Upon ownership transfer, record retention requirements shall be addressed in the ownership transfer agreement and the responsibility for maintaining archives is clearly established. In addition, in cases of bankruptcy, appropriate regulatory and state legal requirements concerning laboratory records must be followed. In the event of the closure of the laboratory, all records will revert to the control of the corporate headquarters. Should the entire company cease to exist, as much notice as possible will be given to clients and the accrediting bodies who have worked with the laboratory during the previous 5 years of such action.

14.5.7 Records Disposal

14.5.7.1 Records are removed from the archive and destroyed after 5 years unless otherwise specified by a client or regulatory requirement. On a project specific or program basis, clients may need to be notified prior to record destruction. Records are destroyed in a manner that ensures their confidentiality such as shredding, mutilation or incineration. (Refer to Tables 14-1 and 14-2).

14.5.7.2 Electronic copies of records must be destroyed by erasure or physically damaging off-line storage media so no records can be read.

14.5.7.3 If a third party records Management Company is hired to dispose of records, a "Certificate of Destruction" is required**.**

SECTION 15

AUDITS
(NELAC 5.4.13)

15.1 INTERNAL AUDITS

Internal audits are performed to verify that laboratory operations comply with the requirements of the lab's quality system and with the external quality programs under which the laboratory operates. Audits are planned and organized by the QA staff. Personnel conducting the audits should be independent of the area being evaluated. Auditors will have sufficient authority, access to work areas, and organizational freedom necessary to observe all activities affecting quality and to report the assessments to laboratory management and when requested to corporate management.

Audits are conducted and documented as described in the TestAmerica Corporate SOP on performing Internal Audits, SOP No. CA-Q-S-004. The types and frequency of routine internal audits are shown in Table 15-1. Special or ad hoc assessments may be conducted as needed under the direction of the QA staff.

Table 15-1. Types of Internal Audits and Frequency

Description	Performed by	Frequency
Quality Systems	QA Department or Designee	All areas of the laboratory annually
QA Technical Audits - Evaluate raw data versus final reports - Analyst integrity - Data authenticity	QA Department or Designee	All methods within a 2-year period, with at least 15% of methods every quarter
SOP Method Compliance	Technical Director	- All SOPs within a 2-year period - All new analysts or new analyst/methods within 3 months of IDOC
Special	QA Department or Designee	Surveillance or spot checks performed as needed
Performance Testing	Analysts with QA oversight	Two successful per year for each NELAC field of testing or as dictated by regulatory requirements

15.1.1 Annual Quality Systems Audit

An annual quality systems audit is required to ensure compliance to analytical methods and SOPs, the laboratory's Data Integrity and Ethics Policies, NELAC quality systems client and state requirements, and the effectiveness of the internal controls of the analytical process, including but not limited to data review, quality controls, preventive action and corrective action.

The completeness of earlier corrective actions is assessed. The audit is divided into modules for each operating or support area of the lab, and each module is comprehensive for a given area. The area audits may be done on a rotating schedule throughout the year to ensure adequate coverage of all areas. This schedule may change as situations in the laboratory warrant.

15.1.2 QA Technical Audits

QA technical audits are based on client projects, associated sample delivery groups, and the methods performed. Reported results are compared to raw data to verify the authenticity of results. The validity of calibrations and QC results are compared to data qualifiers, footnotes, and case narratives. Documentation is assessed by examining run logs and records of manual integrations. Manual calculations are checked. Where possible, MintMiner is used to identify unusual manipulations of the data deserving closer scrutiny. QA technical audits will include all methods within a two-year period.

15.1.3 SOP Method Compliance

Compliance of all SOPs with the source methods and compliance of the operational groups with the SOPs will be assessed by the Technical Director at least every two years. The work of each newly hired analyst is assessed within 3 months of working independently, (e.g., completion of method IDOC). In addition, as analysts add methods to their capabilities, (new IDOC) reviews of the analyst work products will be performed within 3 months of completing the documented training.

15.1.4 Special Audits

Special audits are conducted on an as needed basis, generally as a follow up to specific issues such as client complaints, corrective actions, PT results, data audits, system audits, validation comments, regulatory audits or suspected ethical improprieties. Special audits are focused on a specific issue, and report format, distribution, and timeframes are designed to address the nature of the issue.

15.1.5 Performance Testing

The laboratory participates semi-annually in performance audits conducted through the analysis of PT samples provided by a third party. The laboratory generally participates in the following types of PT studies: Drinking Water, Nonpotable Water, Soil, Air.

It is TestAmerica’s policy that PT samples be treated as typical samples in the production process. Furthermore, where PT samples present special or unique problems, in the regular production process they may need to be treated differently, as would any special or unique request submitted by any client. The QA Manager must be consulted and in agreement with any decisions made to treat a PT sample differently due to some special circumstance.

Written responses to unacceptable PT results are required. In some cases it may be necessary for blind QC samples to be submitted to the laboratory to show a return to control.

15.2 EXTERNAL AUDITS

External audits are performed when certifying agencies or clients conduct on-site inspections or submit performance testing samples for analysis. It is TestAmerica's policy to cooperate fully with regulatory authorities and clients. The laboratory makes every effort to provide the auditors with access to personnel, documentation, and assistance. Laboratory supervisors are responsible for providing corrective actions to the QA Manager who coordinates the response for any deficiencies discovered during an external audit. Audit responses are due in the time allotted by the client or agency performing the audit. A copy of the audit report and the labs corrective action plan will be forwarded to Corporate Quality.

The laboratory cooperates with clients and their representatives to monitor the laboratory's performance in relation to work performed for the client. The client may only view data and systems related directly to the client's work. All efforts are made to keep other client information confidential.

15.2.1 Confidential Business Information (CBI) Considerations

During on-site audits, auditors may come into possession of information claimed as business confidential. A business confidentiality claim is defined as "a claim or allegation that business information is entitled to confidential treatment for reasons of business confidentiality or a request for a determination that such information is entitled to such treatment." When information is claimed as business confidential, the laboratory must place on (or attach to) the information at the time it is submitted to the auditor, a cover sheet, stamped or typed legend or other suitable form of notice, employing language such as "trade secret", "proprietary" or "company confidential". Confidential portions of documents otherwise non-confidential must be clearly identified. CBI may be purged of references to client identity by the responsible laboratory official at the time of removal from the laboratory. However, sample identifiers may not be obscured from the information. Additional information regarding CBI can be found in within the 2003 NELAC standards.

15.3 AUDIT FINDINGS

Audit findings are documented using the corrective action process and database. The laboratory's corrective action responses for both types of audits may include action plans that could not be completed within a predefined timeframe. In these instances, a completion date must set and agreed to by operations management and the QA Manager.

Developing and implementing corrective actions to findings is the responsibility of the Department Manager where the finding originated. Findings that are not corrected by specified due dates are reported monthly to management in the QA monthly report. . A copy of the audit report and the labs corrective action plan will be forwarded to Corporate Quality.

If any audit finding casts doubt on the effectiveness of the operations or on the correctness or validity of the laboratory's test results, the laboratory shall take timely corrective action, and shall notify clients in writing if the investigations show that the laboratory results have been affected. Once corrective action is implemented, a follow-up audit is scheduled to ensure that the problem has been corrected.

Clients must be notified promptly in writing, of any event such as the identification of defective measuring or test equipment that casts doubt on the validity of results given in any test report or amendment to a test report. The investigation must begin within 24-hours of discovery of the problem and all efforts are made to notify the client within two weeks after the completion of the investigation.

SECTION 16

MANAGEMENT REVIEWS
(NELAC 5.4.14)

16.1 QUALITY ASSURANCE REPORT

A comprehensive QA Report shall be prepared each month by the laboratory's QA Department and forwarded to the Laboratory Director for review and comments. The final report shall be submitted to the Operation Manager as well as the appropriate Quality Director and General Manager. All aspects of the QA system are reviewed to evaluate the suitability of policies and procedures. During the course of the year, the Laboratory Director, General Manager or Corporate QA may request that additional information be added to the report.

On a monthly basis, Corporate QA compiles information from all the monthly laboratory reports. The Corporate Quality Director prepares a report that includes a compilation of all metrics and notable information and concerns regarding the QA programs within the laboratories. The report also includes a listing of new regulations that may potentially impact the laboratories. This report is presented to the Senior Management Team and General Managers.

16.2 ANNUAL MANAGEMENT REVIEW

The senior lab management team (Laboratory Director, Technical Director, Operations Manager, Customer Service Manager, QA Manager) conducts a review annually of its quality systems and LIMS to ensure its continuing suitability and effectiveness in meeting client and regulatory requirements and to introduce any necessary changes or improvements. It will also provide a platform for defining quality goals and objectives. Corporate Operations and Corporate QA personnel may be included in this meeting at the discretion of the Laboratory Director. The LIMS review consists of examining any audits, complaints or concerns that have been raised through the year that are related to the LIMS. The laboratory will summarize any critical findings that can not be solved by the lab and report them to Corporate IT.

This management review (Corporate Work Instruction No. CA-Q-WI-020) uses information generated during the preceding year to assess the "big picture" by ensuring that routine actions taken and reviewed on a monthly basis are not components of larger systematic concerns. The monthly review should keep the quality systems current and effective; therefore, the annual review is a formal senior management process to review specific existing documentation. Significant issues from the following documentation are compiled or summarized by the QA Manager prior to the review meeting:

- Matters arising from the previous annual review.
- Prior Monthly QA Reports issues.
- Laboratory QA Metrics.
- Review of report reissue requests.
- Review of client feedback and complaints.

- Issues arising from any prior management or staff meetings.
- Minutes from prior senior lab management meetings. Issues that may be raised from these meetings include:
  - Adequacy of staff, equipment and facility resources.
  - Adequacy of policies and procedures.
  - Future plans for resources and testing capability and capacity.
- The annual internal double blind PT program sample performance (if performed),
- Compliance to the Ethics Policy and Data Integrity Plan. Including any evidence/incidents of inappropriate actions or vulnerabilities related to data Integrity.

A report is generated by the QA Manager and management. The report is distributed to the appropriate General Manager and the Quality Director. The report includes, but is not limited to:

- The date of the review and the names and titles of participants.
- A reference to the existing data quality related documents and topics that were reviewed.
- Quality system or operational changes or improvements that will be made as a result of the review [e.g., an implementation schedule including assigned responsibilities for the changes.

Changes to the quality systems requiring update to the laboratory QA Manual shall be included in the next revision of the QA Manual.

16.3 POTENTIAL INTEGRITY RELATED MANAGERIAL REVIEWS

Potential integrity issues (data or business related) must be handled and reviewed in a confidential manner until such time as a follow-up evaluation, full investigation, or other appropriate actions have been completed and issues clarified. The TestAmerica Corporate Data Investigation/ Recall SOP shall be followed (SOP No. CA-L-S-001). All investigations that result in finding of inappropriate activity are documented and include any disciplinary actions involved, corrective actions taken, and all appropriate notifications of clients.

TestAmerica’s COO, VP of Client & Technical Services, General Managers and Quality Directors receive a monthly report from the Director of Quality & Client Advocacy summarizing any current data integrity or data recall investigations. The General Manager’s are also made aware of progress on these issues for their specific labs.

SECTION 17

PERSONNEL
(NELAC 5.5.2)

17.1 OVERVIEW

The laboratory's management believes that its highly qualified and professional staff is the single most important aspect in assuring a high level of data quality and service. The staff consists of professionals and support personnel as outlined in the organization chart in Figure 4-1.

All personnel must demonstrate competence in the areas where they have responsibility. Any staff that is undergoing training shall have appropriate supervision until they have demonstrated their ability to perform their job function on their own. Staff shall be qualified for their tasks based on appropriate education, training, experience and/or demonstrated skills as required.

The laboratory employs sufficient personnel with the necessary education, training, technical knowledge and experience for their assigned responsibilities.

All personnel are responsible for complying with all QA/QC requirements that pertain to the laboratory and their area of responsibility. Each staff member must have a combination of experience and education to adequately demonstrate a specific knowledge of their particular area of responsibility. Technical staff must also have a general knowledge of lab operations, test methods, QA/QC procedures and records management.

Laboratory management is responsible for formulating goals for lab staff with respect to education, training and skills and ensuring that the laboratory has a policy and procedures for identifying training needs and providing training of personnel. The training shall be relevant to the present and anticipated responsibilities of the lab staff.

The laboratory only uses personnel that are employed by or under contract to, the laboratory. Contracted personnel, when used, must meet competency standards of the laboratory and work in accordance to the laboratory's quality system.

17.2 EDUCATION AND EXPERIENCE REQUIREMENTS FOR TECHNICAL PERSONNEL

The laboratory makes every effort to hire analytical staff that possesses a college degree (AA, BA, BS) in an applied science with some chemistry in the curriculum. Exceptions can be made based upon the individual's experience and ability to learn. Selection of qualified candidates for laboratory employment begins with documentation of minimum education, training, and experience prerequisites needed to perform the prescribed task. Minimum education and training requirements for TestAmerica employees are outlined in job descriptions and are generally summarized for analytical staff in the table below.

The laboratory maintains job descriptions for all personnel who manage, perform or verify work affecting the quality of the environmental testing the laboratory performs. Job Descriptions are located in the TestAmerica Buffalo Human Resource office (Also see Section 4 for position descriptions/responsibilities).

Experience and specialized training are occasionally accepted in lieu of a college degree (basic lab skills such as using a balance, pipette, quantitation techniques, etc. are also considered).

As a general rule for analytical staff:

Specialty	Education	Experience
Extractions, Digestions, some electrode methods (pH, DO, Redox, etc.), or Titrimetric and Gravimetric Analyses	H.S. Diploma	On the job training (OJT)
CVAA, Single component or short list Chromatography (e.g., Fuels, BTEX-GC, IC)	A college degree in an applied science or 2 years of college and at least 1 year of college chemistry	Or 2 years prior analytical experience is required
ICP, ICPMS, Long List or complex chromatography (e.g., Pesticides, PCB, Herbicides, HPLC, etc.), GCMS	A college degree in an applied science or 2 years of college chemistry	or 5 years of prior analytical experience
Spectra Interpretation	A college degree in an applied science or 2 years of college chemistry	And 2 years relevant experience Or 5 years of prior analytical experience
Technical Directors/Department Managers – **General**	Bachelors Degree in an applied science or engineering with 24 semester hours in chemistry An advanced (MS, PhD.) degree may substitute for one year of experience	And 2 years experience in environmental analysis of representative analytes for which they will oversee

When an analyst does not meet these requirements, they can perform a task under the direct supervision of a qualified analyst, peer reviewer or Department Manager, and are considered an analyst in training. The person supervising an analyst in training is accountable for the quality of the analytical data and must review and approve data and associated corrective actions.

17.3 TRAINING

The laboratory is committed to furthering the professional and technical development of employees at all levels.

Orientation to the laboratory's policies and procedures, in-house method training, and employee attendance at outside training courses and conferences all contribute toward employee proficiency. Below are examples of various areas of required employee training:

Required Training	**Time Frame**	**Employee Type**
Environmental Health & Safety	Prior to lab work	All
Ethics – New Hires	1 week of hire	All
Ethics - Comprehensive	90 days of hire	All
Data Integrity	30 days of hire	Technical and PMs
Quality Assurance	90 days of hire	All
Ethics – Refresher	Annually	All
Initial Demonstration of Capability (DOC)	Prior to unsupervised method performance	Technical

The laboratory maintains records of relevant authorization/competence, education, professional qualifications, training, skills and experience of technical personnel (including contracted personnel) as well as the date that approval/authorization was given. These records are kept on file at the laboratory. Also refer to "Demonstration of Capability" in Section 19.

The training of technical staff is kept up to date by:

- Each employee must have documentation in their training file that they have read, understood and agreed to follow the most recent version of the laboratory QA Manual and SOPs in their area of responsibility. This documentation is updated as SOPs are updated.
- Documentation from any training courses or workshops on specific equipment, analytical techniques or other relevant topics are maintained in their training file.
- Documentation of proficiency (refer to Section 20).
- An Ethics Agreement signed by each staff member (renewed each year) and evidence of annual ethics training.
- A Confidentiality Agreement signed by each staff member signed at the time of employment.
- The Human Resource office maintains documentation and attestation forms on employment status & records; benefit programs; timekeeping/payroll; and employee conduct (e.g., ethics). This information is maintained in the employee's secured personnel file.

Further details of the laboratory's training program are described in TestAmerica Buffalo SOP BF-QA-004, Laboratory Personnel Training.

17.4 DATA INTEGRITY AND ETHICS TRAINING PROGRAM

Establishing and maintaining a high ethical standard is an important element of a Quality System. Ethics and data integrity training is integral to the success of TestAmerica and is provided for each employee at TestAmerica. It is a formal part of the initial employee orientation within 1 week of hire, comprehensive training within 90 days, and an annual refresher for all employees. Senior management at each facility performs the ethics training for their staff.

In order to ensure that all personnel understand the importance TestAmerica places on maintaining high ethical standards at all times; TestAmerica has established a Corporate Ethics Policy No. CA-L-P-001 and an Ethics Statement. All initial and annual training is documented by signature on the signed Ethics demonstrating that the employee has participated in the training and understands their obligations related to ethical behavior and data integrity.

Violations of this Ethics Policy will not be tolerated. Employees who violate this policy will be subject to disciplinary actions up to and including termination. Criminal violations may also be referred to the Government for prosecution. In addition, such actions could jeopardize TestAmerica's ability to do work on Government contracts, and for that reason, TestAmerica has a Zero Tolerance approach to such violations.

Employees are trained as to the legal and environmental repercussions that result from data misrepresentation. Key topics covered in the presentation include:

- Organizational mission and its relationship to the critical need for honesty and full disclosure in all analytical reporting.
- Ethics Policy
- How and when to report ethical/data integrity issues. Confidential reporting.
- Record keeping.
- Discussion regarding data integrity procedures.
- Specific examples of breaches of ethical behavior (e.g. peak shaving, altering data or computer clocks, improper macros, etc., accepting/offering kickbacks, illegal accounting practices, unfair competition/collusion)
- Internal monitoring. Investigations and data recalls.
- Consequences for infractions including potential for immediate termination, debarment, or criminal prosecution.
- Importance of proper written narration / data qualification by the analyst and project manager with respect to those cases where the data may still be usable but are in one sense or another partially deficient.

Additionally, a data integrity hotline (1-800-736-9407) is maintained by TestAmerica and administered by the Corporate Quality Department.

SECTION 18

ACCOMMODATIONS AND ENVIRONMENTAL CONDITIONS
(NELAC 5.5.3)

18.1 OVERVIEW

TestAmerica Buffalo is a 32,000 ft^2 secure laboratory facility with controlled access and designed to accommodate an efficient workflow and to provide a safe and comfortable work environment for employees. All visitors sign in and are escorted by laboratory personnel. Access is controlled by various measures.

The laboratory is equipped with structural safety features. Each employee is familiar with the location, use, and capabilities of general and specialized safety features associated with their workplace. The laboratory provides and requires the use of protective equipment including safety glasses, protective clothing, gloves, etc. OSHA and other regulatory agency guidelines regarding required amounts of bench and fume hood space, lighting, ventilation (temperature and humidity controlled), access, and safety equipment are met or exceeded.

Traffic flow through sample preparation and analysis areas is minimized to reduce the likelihood of contamination. Adequate floor space and bench top area is provided to allow unencumbered sample preparation and analysis space. Sufficient space is also provided for storage of reagents and media, glassware, and portable equipment. Ample space is also provided for refrigerated sample storage before analysis and archival storage of samples after analysis. Laboratory HVAC and deionized water systems are designed to minimize potential trace contaminants.

The laboratory is separated into specific areas for field operations, bottle kit preparation, sample receiving, sample preparation, volatile organic sample analysis, non-volatile organic sample analysis, inorganic sample analysis and administrative functions.

18.2 ENVIRONMENT

Laboratory accommodation, test areas, energy sources, lighting are adequate to facilitate proper performance of tests. The facility is equipped with heating, ventilation, and air conditioning (HVAC) systems appropriate to the needs of environmental testing performed at this laboratory.

The environment in which these activities are undertaken does not invalidate the results or adversely affect the required accuracy of any measurements.

The laboratory provides for the effective monitoring, control and recording of environmental conditions that may affect the results of environmental tests as required by the relevant specifications, methods, and procedures. Such environmental conditions include humidity, voltage, temperature, and vibration levels in the laboratory. Key equipment has been provided with back-up power supply in the event of a power outage.

When any of the method or regulatory required environmental conditions change to a point where they may adversely affect test results, analytical testing will be discontinued until the environmental conditions are returned to the required levels.

Environmental conditions of the facility housing the computer network and LIMS are regulated to protect against raw data loss.

18.3 WORK AREAS

There is effective separation between neighboring areas when the activities therein are incompatible with each other. Examples include:

- Volatile organic chemical handling areas, including sample preparation and waste disposal, and volatile organic chemical analysis areas.

Access to and use of all areas affecting the quality of analytical testing is defined and controlled by secure access to the laboratory building as described below in the Building Security section.

Adequate measures are taken to ensure good housekeeping in the laboratory and to ensure that any contamination does not adversely affect data quality. These measures include regular cleaning to control dirt and dust within the laboratory.

Work areas are available to ensure an unencumbered work area. Work areas include:

- Access and entryways to the laboratory.
- Sample receipt areas.
- Sample storage areas.
- Chemical and waste storage areas.
- Data handling and storage areas.
- Sample processing areas.
- Sample analysis areas.

18.4 FLOOR PLAN

A floor plan can be found in Appendix 1.

18.5 BUILDING SECURITY

Building pass cards and alarm codes are distributed to all facility employees.

Visitors to the laboratory sign in and out in a visitor's logbook. A visitor is defined as any person who visits the laboratory who is not an employee of the laboratory. [The reason for this is that it is important to know who is in the building in case of a safety emergency. The visitors logbook is used to ensure that everyone got out of the building safely.] In addition to signing into the laboratory, the Environmental, Health and Safety Manual contains requirements for visitors and vendors. There are specific safety forms that must be reviewed and signed.

Visitors (with the exception of company employees) are escorted by laboratory personnel at all times, or the location of the visitor is noted in the visitor's logbook.

SECTION 19.0

TEST METHODS AND METHOD VALIDATION
(NELAC 5.5.4)

19.1 OVERVIEW

The laboratory uses methods that are appropriate to meet our clients' requirements and that are within the scope of the laboratory's capabilities. These include sampling, handling, transport, storage and preparation of samples, and, where appropriate, an estimation of the measurement of uncertainty as well as statistical techniques for analysis of environmental data.

Instructions are available in the laboratory for the operation of equipment as well as for the handling and preparation of samples. All instructions, Standard Operating Procedures (SOPs), reference methods and manuals relevant to the working of the laboratory are readily available to all staff. Deviations from published methods are documented (with justification) in the laboratory's approved SOPs. SOPs are submitted to clients for review at their request. Significant deviations from published methods require client approval and regulatory approval where applicable.

19.2 STANDARD OPERATING PROCEDURES (SOPs)

The laboratory maintains SOPs that accurately reflect all phases of the laboratory such as assessing data integrity, corrective actions, handling customer complaints as well as all analytical methods and sampling procedures. The method SOPs are derived from the most recently promulgated/approved, published methods and are specifically adapted to the laboratory facility. Modifications or clarifications to published methods are clearly noted in the SOPs. All SOPs are controlled in the laboratory:

- All SOPs contain a revision number, effective date, and appropriate approval signatures. Controlled copies are available to all staff.
- Procedures for writing an SOP are incorporated by reference to TestAmerica's Corporate SOP CW-Q-S-002, Writing a Standard Operating Procedure (SOP) and Laboratory SOP BF-QA-003, Procedure for Writing, Reviewing and Revising Controlled Quality Documents (QAM, SOP, etc)

- SOPs are reviewed at a minimum of every 2 years (annually for Drinking Water), and where necessary, revised to ensure continuing suitability and compliance with applicable requirements.

19.3 LABORATORY METHODS MANUAL

For each test method, the laboratory shall have available the published referenced method as well as the laboratory developed SOP.

Note: If more stringent standards or requirements are included in a mandated test method or regulation than those specified in this manual, the laboratory shall demonstrate that such requirements are met. If it is not clear which requirements are more stringent, the standard from

the method or regulation is to be followed. Any exceptions or deviations from the referenced methods or regulations are noted in the specific analytical SOP.
The laboratory maintains an SOP Index for both technical and non-technical SOPs. Technical SOPs are maintained to describe a specific test method. Non-technical SOPs are maintained to describe functions and processes not related to a specific test method.

19.4 SELECTION OF METHODS

Since numerous methods and analytical techniques are available, continued communication between the client and laboratory is imperative to assure the correct methods are utilized. Once client methodology requirements are established, this and other pertinent information is summarized by the Project Manager. These mechanisms ensure that the proper analytical methods are applied when the samples arrive for log-in. For non-routine analytical services (e.g., special matrices, non-routine compound lists, etc.), the method of choice is selected based on client needs and available technology. The methods selected should be capable of measuring the specific parameter of interest, in the concentration range of interest, and with the required precision and accuracy.

19.4.1 Sources of Methods

Routine analytical services are performed using standard EPA-approved methodology. In some cases, modification of standard approved methods may be necessary to provide accurate analyses of particularly complex matrices. When the use of specific methods for sample analysis is mandated through project or regulatory requirements, only those methods shall be used.

When clients do not specify the method to be used or methods are not required, the methods used will be clearly validated and documented in an SOP and available to clients and/or the end user of the data.

19.4.1.1 The analytical methods used by the laboratory are those currently accepted and approved by the U. S. EPA and the state or territory from which the samples were collected. Reference methods include:

- *Method 1664, Revision A: N-Hexane Extractable Material (HEM; Oil and Grease) and Silica Gel Treated N-Hexane Extractable Material (SGT-HEM); Non-polar Material) by Extraction and Gravimetry, EPA-821-R-98-002, February 1999*
- *Compendium of Methods for the Determination of Toxic Organic Compounds in Ambient Air, US EPA, January 1996.*
- *Guidelines Establishing Test Procedures for the Analysis of Pollutants Under the Clean Water Act, and Appendix A-C; 40 CFR Part 136, USEPA Office of Water. Revised as of July 1, 1995, Appendix A to Part 136 - Methods for Organic Chemical Analysis of Municipal and Industrial Wastewater (EPA 600 Series)*
- *Methods for Chemical Analysis of Water and Wastes, EPA 600 (4-79-020), 1983.*
- *Methods for the Determination of Inorganic Substances in Environmental Samples, EPA-600/R-93/100, August 1993.*

- *Methods for the Determination of Metals in Environmental Samples, EPA/600/4-91/010, June 1991. Supplement I: EPA-600/R-94/111, May 1994.*
- *Methods for the Determination of Organic Compounds in Drinking Water, EPA-600/4-88-039, December 1988, Revised, July 1991, Supplement I, EPA-600-4-90-020, July 1990, Supplement II, EPA-600/R-92-129, August 1992. Supplement III EPA/600/R-95/131 - August 1995 (EPA 500 Series) (EPA 500 Series methods)*
- *Technical Notes on Drinking Water Methods, EPA-600/R94-173, October 1994*
- *NIOSH Manual of Analytical Methods, 4th ed., August 1994.*
- *Statement of Work for Inorganics Analysis, ILM04.1, USEPA Contract Laboratory Program Multi-media, Multi-concentration.*
- *Statement of Work for Inorganics Analysis, ILM05.2/5.3, USEPA Contract Laboratory Program Multi-media, Multi-concentration*
- *Statement of Work for Organics Analysis, OLM04.2/4.3, USEPA Contract Laboratory Program, Multi-media, Multi-concentration.*
- *Standard Methods for the Examination of Water and Wastewater, 18th/19th /20th edition; Eaton, A.D. Clesceri, L.S. Greenberg, A.E. Eds; American Water Works Association, Water Pollution Control Federation, American Public Health Association: Washington, D.C.*
- *Test Methods for Evaluating Solid Waste Physical/Chemical Methods (SW846), Third Edition, September 1986, Final Update I, July 1992, Final Update IIA, August 1993, Final Update II, September 1994; Final Update IIB, January 1995; Final Update III, December 1996.*
- *Annual Book of ASTM Standards, American Society for Testing & Materials (ASTM), Philadelphia, PA.*
- *National Status and Trends Program, National Oceanographic and Atmospheric Administration, Volume I-IV, 1985-1994.*
- *Manual for the Certification of Laboratories Analyzing Drinking Water (EPA 815-R-05-004, January 2005)* ***(DW labs only)***
- *Code of Federal Regulations (CFR) 40, Parts 136, 141, 172, 173, 178, 179 and 261*
- *New York State DEC Analytical Services Protocol, 2005*
- *New York State DOH Methods Manual*

The laboratory reviews updated versions to all the aforementioned references for adaptation based upon capabilities, instrumentation, etc., and implements them as appropriate. As such, the laboratory strives to perform only the latest versions of each approved method as regulations allow or require.

Other reference procedures for non-routine analyses may include methods established by specific states (e.g., Underground Storage Tank methods), ASTM or equipment manufacturers. Sample type, source, and the governing regulatory agency requiring the analysis will determine the method utilized.

The laboratory shall inform the client when a method proposed by the client may be inappropriate or out of date. After the client has been informed, and they wish to proceed contrary to the laboratory's recommendation, it will be documented.

19.4.2 Demonstration of Capability

Before the laboratory may institute a new method and begin reporting results, the laboratory shall confirm that it can properly operate the method. In general, this demonstration does not test the performance of the method in real world samples, but in an applicable and available clean matrix sample. If the method is for the testing of analytes that are not conducive to spiking, demonstration of capability may be performed on quality control samples.

19.4.2.1 A demonstration of capability (BF-QA-004) is performed whenever there is a significant change in instrument type (e.g., new instrumentation), method or personnel.

19.4.2.2 The initial demonstration of capability must be thoroughly documented and approved by the Operations Manager and QA Manager prior to independently analyzing client samples. All associated documentation must be retained in accordance with the laboratories archiving procedures.

19.4.2.3 The laboratory must have an approved SOP, demonstrate satisfactory performance, and conduct a method detection limit study (when applicable). There may be other requirements as stated within the published method or regulations (i.e., retention time window study).

Note: In some instances, a situation may arise where a client requests that an unusual analyte be reported using a method where this analyte is not normally reported. If the analyte is being reported for regulatory purposes, the method must meet all procedures outlined within this QA Manual (SOP, MDL, and Demonstration of Capability). If the client states that the information is not for regulatory purposes, the result may be reported as long as the following criteria are met:

- The instrument is calibrated for the analyte to be reported using the criteria for the method and ICV/CCV criteria are met (unless an ICV/CCV is not required by the method or criteria are per project DQOs).
- The laboratory's nominal or default reporting limit (RL) is equal to the quantitation limit (QL), must be at or above the lowest non-zero standard in the calibration curve and must be reliably determined. Project RLs are client specified reporting levels which may be higher than the QL. Results reported below the QL must be qualified as estimated values. Also see Section 19.6.1.3, Relationship of Limit of Detection (LOD) to Quantitation Limit (QL).
- The client request is documented and the lab informs the client of its procedure for working with unusual compounds. The final report must be footnoted: *Reporting Limit based on the low standard of the calibration curve.*

19.4.3 Initial Demonstration of Capability (IDOC) Procedures

Procedures for generation of IDOCs are detailed below and in laboratory SOP BF-QA-004, Laboratory Personnel Training.

19.4.3.1 The spiking standard used must be prepared independently from those used in instrument calibration.

19.4.3.2 The analyte(s) shall be diluted in a volume of clean matrix sufficient to prepare four aliquots at the concentration specified by a method or the laboratory SOP.

19.4.3.3 At least four aliquots shall be prepared (including any applicable clean-up procedures) and analyzed according to the test method (either concurrently or over a period of days).

19.4.3.4 Using all of the results, calculate the mean recovery in the appropriate reporting units and the standard deviations for each parameter of interest.

19.4.3.5 When it is not possible to determine the mean and standard deviations, such as for presence, absence and logarithmic values, the laboratory will assess performance against criteria described in the Method SOP.

19.4.3.6 Compare the information obtained above to the corresponding acceptance criteria for precision and accuracy in the test method (if applicable) or in laboratory generated acceptance criteria (LCS or interim criteria) if there is no mandatory criteria established. If any one of the parameters do not meet the acceptance criteria, the performance is unacceptable for that parameter.

19.4.3.7 When one or more of the tested parameters fail at least one of the acceptance criteria, the analyst must proceed according to either option listed below:

- Locate and correct the source of the problem and repeat the test for all parameters of interest beginning with 19.4.3.3 above.
- Beginning with 19.4.3.3 above, repeat the test for all parameters that failed to meet criteria. Repeated failure, however, will confirm a general problem with the measurement system. If this occurs, locate and correct the source of the problem and repeat the test for all compounds of interest beginning with 20.4.3.1 above.

Note: Results of successive LCS analyses can be used to fulfill the DOC requirement.

A certification statement (see Figure 19-1) shall be used to document the completion of each initial demonstration of capability. A copy of the certification is archived in the analyst's training folder.

19.5 LABORATORY DEVELOPED METHODS AND NON-STANDARD METHODS

Any new method developed by the laboratory must be fully defined in an SOP and validated by qualified personnel with adequate resources to perform the method. Method specifications and the relation to client requirements must be clearly conveyed to the client if the method is a non-standard method (not a published or routinely accepted method). The client must also be in agreement to the use of the non-standard method.

19.6 VALIDATION OF METHODS

Validation is the confirmation by examination and the provision of objective evidence that the particular requirements for a specific intended use are fulfilled.

All non-standard methods, laboratory designed/developed methods, standard methods used outside of their scope, and major modifications to published methods must be validated to confirm they are fit for their intended use. The validation will be as extensive as necessary to meet the needs of the given application. The results are documented with the validation procedure used and contain a statement as to the fitness for use.

19.6.1 Method Validation and Verification Activities for All New Methods

While method validation can take various courses, the following activities can be required as part of method validation. Method validation records are designated QC records and are archived accordingly.

19.6.1.1 Determination of Method Selectivity

Method selectivity is the demonstrated ability to discriminate the analyte(s) of interest from other compounds in the specific matrix or matrices from other analytes or interference. In some cases to achieve the required selectivity for an analyte, a confirmation analysis is required as part of the method.

19.6.1.2 Determination of Method Sensitivity

Sensitivity can be both estimated and demonstrated. Whether a study is required to estimate sensitivity depends on the level of method development required when applying a particular measurement system to a specific set of samples. Where estimations and/or demonstrations of sensitivity are required by regulation or client agreement, such as the procedure in 40 CFR Part 136 Appendix B, under the Clean Water Act, these shall be followed.

19.6.1.3 Relationship of Limit of Detection (LOD) to the Quantitation Limit (QL)

An important characteristic of expression of sensitivity is the difference in the LOD and the QL. The LOD is the minimum level at which the presence of an analyte can be reliably concluded. The QL is the minimum concentration of analyte that can be quantitatively determined with acceptable precision and bias. For most instrumental measurement systems, there is a region where semi-quantitative data is generated around the LOD (both above and below the estimated MDL or LOD) and below the QL. In this region, detection of an analyte may be confirmed but quantification of the analyte is unreliable within the accuracy and precision guidelines of the measurement system. When an analyte is detected below the QL, and the presence of the analyte is confirmed by meeting the qualitative identification criteria for the analyte, the analyte can be reliably reported, but the amount of the analyte can only be estimated. If data is to be reported in this region, it must be done so with a qualification that denotes the semi-quantitative nature of the result.

19.6.1.4 Determination of Interferences

A determination that the method is free from interferences in a blank matrix is performed.

19.6.1.5 Determination of Range

Where appropriate to the method, the quantitation range is determined by comparison of the response of an analyte in a curve to established or targeted criteria. Generally the upper quantitation limit is defined by highest acceptable calibration concentration. The lower quantitation limit or QL cannot be lower than the lowest non-zero calibration level, and can be constrained by required levels of bias and precision.

19.6.1.6 Determination of Accuracy and Precision

Accuracy and precision studies are generally performed using replicate analyses, with a resulting percent recovery and measure of reproducibility (standard deviation, relative standard deviation) calculated and measured against a set of target criteria.

19.6.1.7 Documentation of Method

The method is formally documented in an SOP. If the method is a minor modification of a standard laboratory method that is already documented in an SOP, an SOP Attachment describing the specific differences in the new method is acceptable in place of a separate SOP.

19.6.1.8 Continued Demonstration of Method Performance

Continued demonstration of Method Performance is addressed in the SOP. Continued demonstration of method performance is generally accomplished by batch specific QC samples such as LCS, method blanks or PT samples.

19.7 METHOD DETECTION LIMITS (MDL)/ LIMITS OF DETECTION (LOD)

Method detection limits (MDL) are initially determined in accordance with 40 CFR Part 136, Appendix B or alternatively by other technically acceptable practices that have been accepted by regulators. MDL is also sometimes referred to as Limit of Detection (LOD). The MDL theoretically represents the concentration level for each analyte within a method at which the Analyst is 99% confident that the true value is not zero. The MDL is determined for each analyte initially during the method validation process and updated as required in the analytical methods, whenever there is a significant change in the procedure or equipment, or based on project specific requirements (refer to 19.7.10). Generally the analyst prepares at least seven replicates of solution spiked at one to five times the estimated method detection limit (most often at the lowest standard in the calibration curve) into the applicable matrix with all the analytes of interest. Each of these aliquots is extracted (including any applicable clean-up procedures) and analyzed in the same manner as the samples. Where possible, the seven replicates should be analyzed over 2-4 days to provide a more realistic MDL. To allow for some flexibility, this low level standard may be analyzed every batch or every week or some other frequency rather than doing the study all at once. In addition, a larger number of data points may be used if the appropriate t-value multiplier is used.

Refer to the Corporate SOP No. CA-Q-S-006 or the laboratory's SOP No. BF-QA-001 for details on the laboratory's MDL process.

19.8 INSTRUMENT DETECTION LIMITS (IDL)

19.8.1 The IDL is sometimes used to assess the reasonableness of the MDLs or in some cases required by the analytical method or program requirements. IDLs are most used in metals analyses but may be useful in demonstration of instrument performance in other areas.

19.8.2 IDLs are calculated to determine an instrument's sensitivity independent of any preparation method. IDLs are calculated either using 7 replicate spike analyses, like MDL but without sample preparation, or by the analysis of 10 instrument blanks and calculating 3 x the absolute value of the standard deviation. (For CLP procedures, the IDL is determined using the standard deviation of 7 replicate spike analyses on each of 3 non-consecutive days.)

19.8.3 If IDL is > than the MDL, it may be used as the reported MDL.

19.9 VERIFICATION OF DETECTION AND REPORTING LIMITS

19.9.1 Once an MDL is established, it must be verified, on each instrument, by analyzing a quality control sample (prepared as a sample) at approximately 2-3 times the calculated MDL for single analyte analyses (e.g. most wet chemistry methods, CVAA, etc.) and 1-4 times the calculated MDL for multiple analyte methods (e.g. GC, GCMS, ICP, etc.). The analytes must be qualitatively identified or see section 20.7.9 for other options. This verification does not apply to methods that are not readily spiked (e.g. pH, turbidity, etc.) or where the lab does not report to the MDL. If the MDL does not verify, then the lab will not report to the MDL, or redevelop their MDL or use the level where qualitative identification is established. MDLs must be verified at least annually

19.9.2 When the laboratory establishes a quantitation limit, it must be initially verified by the analysis of a low level standard or QC sample at 1-2 the reporting limit and annually thereafter. The annual requirement is waved for methods that have an annually verified MDL. The laboratory will comply with any regulatory

19.10 RETENTION TIME WINDOWS

Most organic analyses and some inorganic analyses use chromatography techniques for qualitative and quantitative determinations. For every chromatography analysis each analyte will have a specific time of elution from the column to the detector. This is known as the analyte's retention time. The variance in the expected time of elution is defined as the retention time window. As the key to analyte identification in chromatography, retention time windows must be established on every column for every analyte used for that method. These records are kept with the files associated with an instrument for later quantitation of the analytes. Complete details are available in the laboratory's Sops.

19.11 EVALUATION OF SELECTIVITY

The laboratory evaluates selectivity by following the checks within the applicable analytical methods, which include mass spectral tuning, second column confirmation, ICP interelement interference checks, chromatography retention time windows, sample blanks, spectrochemical or atomic absorption profiles and specific electrode response factors.

19.12 ESTIMATION OF UNCERTAINTY OF MEASUREMENT

19.12.1 Uncertainty is “a parameter associated with the result of a measurement, that characterizes the dispersion of the values that could reasonably be attributed to the measurand” (as defined by the International Vocabulary of Basic and General Terms in Metrology, ISO Geneva, 1993, ISBN 92-67-10175-1). Knowledge of the uncertainty of a measurement provides additional confidence in a result’s validity. Its value accounts for all the factors which could possibly affect the result, such as adequacy of analyte definition, sampling, matrix effects and interferences, climatic conditions, variances in weights, volumes, and standards, analytical procedure, and random variation. Some national accreditation organizations require the use of an “expanded uncertainty”: the range within which the value of the measurand is believed to lie within at least a 95% confidence level with the coverage factor k=2.

19.12.2 Uncertainty is not error. Error is a single value, the difference between the true result and the measured result. On environmental samples, the true result is never known. The measurement is the sum of the unknown true value and the unknown error. Unknown error is a combination of systematic error, or bias, and random error. Bias varies predictably, constantly, and independently from the number of measurements. Random error is unpredictable, assumed to be Gaussian in distribution, and reducible by increasing the number of measurements.

19.12.3 The minimum uncertainty associated with results generated by the laboratory can be determined by using the Laboratory Control Sample (LCS) accuracy range for a given analyte. The LCS limits are used to assess the performance of the measurement system since they take into consideration all of the laboratory variables associated with a given test over time (except for variability associated with the sampling and the variability due to matrix effects). The percent recovery of the LCS is compared either to the method-required LCS accuracy limits or to the statistical, historical, in-house LCS accuracy limits.

19.12.4 To calculate the uncertainty for the specific result reported, multiply the result by the decimal of the lower end of the LCS range percent value for the lower end of the uncertainty range, and multiply the result by the decimal of the upper end of the LCS range percent value for the upper end of the uncertainty range. These calculated values represent a 99%-certain range for the reported result. As an example, suppose that the result reported is 1.0 mg/l, and the LCS percent recovery range is 50 to 150%. The uncertainty range would be 0.5 to 1.5 mg/l, which could also be written as 1.0 +/- 0.5 mg/l.

19.12.5 In the case where a well recognized test method specifies limits to the values of major sources of uncertainty of measurement (e.g. 524.2, 525, etc) and specifies the form of presentation of calculated results, no further discussion of uncertainty is required.

19.13 SAMPLE REANALYSIS GUIDELINES

Because there is a certain level of uncertainty with any analytical measurement, a sample reanalysis may result in either a higher or lower value from an initial sample analysis. There are also variables that may be present (e.g., sample homogeneity, analyte precipitation over time, etc.) that may affect the results of a reanalysis. Based on the above comments, the laboratory will reanalyze samples at a client's request with the following caveats. Client specific Contractual Terms & Conditions for reanalysis protocols may supersede the following items.

- Homogenous samples: If a reanalysis agrees with the original result to within the RPD limits for MS/MSD or Duplicate analyses, or within ± 1 reporting limit for samples ≤ 5x the reporting limit, the original analysis will be reported. At the client's request, both results may be reported on the same report but not on two separate reports.

- If the reanalysis does not agree (as defined above) with the original result, then the laboratory will investigate the discrepancy and reanalyze the sample a third time for confirmation if sufficient sample is available.

- Any potential charges related to reanalysis are discussed in the contract terms and conditions or discussed at the time of the request. The client will typically be charged for reanalysis unless it is determined that the lab was in error.

- Due to the potential for increased variability, reanalysis may not be applicable to Non-homogenous, Encore, and Sodium Bisulfate preserved samples. See the Department Supervisor or Laboratory Director/Manager if unsure.

19.14 CONTROL OF DATA

The laboratory has policies and procedures in place to ensure the authenticity, integrity, and accuracy of the analytical data generated by the laboratory.

19.14.1 Computer and Electronic Data Related Requirements

The three basic objectives of our computer security procedures and policies are shown below. The laboratory is currently running the 'Element Data System' which is a 3rd party LIMs system that has been highly customized to meet the needs of the laboratory. It is referred to as LIMS for the remainder of this section. The LIMS utilizes a SQL server which is an industry standard relational database platform. It is referred to as Database for the remainder of this section.

19.14.1.1 Maintain the Database Integrity

Assurance that data is reliable and accurate through data verification (review) procedures, password-protecting access, anti-virus protection, data change requirements, as well as an internal LIMS permissions procedure.

- LIMS Database Integrity is achieved through data input validation, internal user controls, and data change requirements.
- Spreadsheets and other software developed in-house must be verified with documentation through hand calculations prior to use.

19.14.1.2 Ensure Information Availability

Protection against loss of information or service is ensured through scheduled back-ups, stable file server network architecture, secure storage of media, line filter, Uninterruptible Power Supply (UPS), and maintaining older versions of software as revisions are implemented.

19.14.1.3 Maintain Confidentiality

Ensure data confidentiality through physical access controls, and encryption of when electronically transmitting data.

19.14.2 Data Reduction

The complexity of the data reduction depends on the analytical method and the number of discrete operations involved (e.g., extractions, dilutions, instrument readings and concentrations). The analyst calculates the final results from the raw data or uses appropriate computer programs to assist in the calculation of final reportable values.

For manual data entry, e.g., Wet Chemistry, the data is reduced by the analyst and then verified by the Department Manager or alternate analyst prior to updating the data in LIMS. The data review sheets, or any other type of applicable documents, are signed by both the analyst and alternate reviewer to confirm the accuracy of the manual entry(s).

Manual integration of peaks will be documented and reviewed and the raw data will be flagged in accordance with the TestAmerica Corporate SOP CA-Q-S-002, *Acceptable Manual Integration Practices*.

Analytical results are reduced to appropriate concentration units specified by the analytical method, taking into account factors such as dilution, sample weight or volume, etc. Blank correction will be applied only when required by the method or per manufacturer's indication; otherwise, it should not be performed. Calculations are independently verified by appropriate laboratory staff. Calculations and data reduction steps for various methods are summarized in the respective analytical SOPs or program requirements.

19.14.2.1 All raw data must be retained in the project job folder, computer file, and/or run log. All criteria pertinent to the method must be recorded. The documentation is recorded at the time observations or calculations are made and must be signed or initialed/dated (month/day/year). It must be easily identifiable who performed which tasks if multiple people were involved.

19.14.2.2 In general, concentration results are reported in milligrams per liter (mg/l) or micrograms per liter (µg/l) for liquids and milligrams per kilogram (mg/kg) or micrograms per kilogram (µg/kg) for solids. For values greater than 10,000 mg/l, results can be reported in percent, i.e., 10,000 mg/l = 1%. Units are defined in each lab SOP.

19.14.2.3 In reporting, the analyst or the instrument output records the raw data result using values of known certainty plus one uncertain digit. If final calculations are performed external to LIMS, the results should be entered in LIMS with at least three significant figures. In general, final inorganic results are reported to 2 significant figures for values less than 10 and 3 significant figures for values greater than 10 on the final report. Organic results are generally reported to 1 significant figure for values less than 10 and 2 significant figures for values greater than 10 on the final report. The number of significant figures may be adjusted based on client or project requirements.

19.14.2.4 For those methods that do not have an instrument printout, an instrumental output or a calculation spreadsheet upload compatible with the LIMS System, the final results and dilution factors are entered directly into LIMS by the analyst, and the software formats the final result for the analytical report. LIMS has a defined significant figure criterion for each analyte.

19.14.2.5 The laboratory strives to import data directly from instruments or calculation spreadsheets to ensure that the reported data are free from transcription and calculation errors. For those analyses with an instrumental output compatible with the LIMS, the raw results and dilution factors are transferred into LIMS electronically after reviewing the quantitation report, and removing unrequested or poor spectrally-matched compounds. The analyst prints a copy of what has been entered to check for errors. This printout and the instrument's printout of calibrations, concentrations, retention times, chromatograms, and mass spectra, if applicable, are retained with the data file. The data file is automatically transferred to the network server and, eventually, to a back-up tape file.

19.14.3 Logbook / Worksheet Use Guidelines

Logbooks and worksheets are filled out 'real time' and have enough information on them to trace the events of the applicable analysis/task. (e.g. calibrations, standards, analyst, sample ID, date, time on short holding time tests, temperatures when applicable, calculations are traceable, etc.)

- Corrections are made following the procedures outlined in Section 12.
- Logbooks are controlled by the QA department. A record is maintained of all logbooks in the lab.
- Unused portions of pages must be "Z"'d out, signed and dated.
- Worksheets are created with the approval of the Technical Director/QA Manager at the facility. The QA Manager controls all worksheets following the procedures in Section 6.

19.14.4 Review / Verification Procedures

Review procedures are out lined in several laboratory SOPs (e.g. BF-SR-002, "Receipt of Analytical Samples", BF-GP-012, "Technical Data Review", and BF-PM-001, "Project

Information Requirements") to ensure that reported data are free from calculation and transcription errors, that QC parameters have been reviewed and evaluated before data is reported. The laboratory also has an SOP discussing Manual Integrations to ensure the authenticity of the data (BF-GP-013, Manual Integration). The general review concepts are discussed below, more specific information can be found in the SOPs.

19.14.4.1 The data review process at the laboratory starts at the Sample Control level. Sample Control personnel review chain-of-custody forms and input the sample information and required analyses into a computer LIMS. The Project Managers perform review of the chain-of-custody forms and inputted information and approve the input in LIMs to make the samples available to the laboratory departments for batching and processing.

19.14.4.2 The next level of data review occurs with the Analysts. As results are generated, analysts review their work to ensure that the results generated meet QC requirements and relevant EPA methodologies. The Analysts transfer the data into the LIMS and add any manual data qualifiers or dilution codes if applicable. To ensure data compliance, a different analyst performs a second level of review. Second level review is accomplished by checking reported results against raw data and evaluating the results for accuracy. During the second level review, blank runs, QA/QC check results, continuing calibration results, laboratory control samples, sample data, qualifiers and spike information are evaluated. Approximately 10% of all sample data from manual methods and from automated methods, all GC/MS spectra and all manual integrations are reviewed. Issues that deem further review include the following:

- QC data are outside the specified control limits for accuracy and precision
- Reviewed sample data does not match with reported results
- Unusual detection limit changes are observed
- Samples having unusually high results
- Samples exceeding a known regulatory limit
- Raw data indicating some type of contamination or poor technique
- Inconsistent peak integration
- Transcription errors
- Results outside of calibration range
- Results deviate from historical trends (if history available)

19.14.4.3 Unacceptable analytical results may require reanalysis of the samples. Any unusual or uncharacteristic circumstances are brought to the attention of the Department Manager. The Department Manager may involve the Project Manager, the Technical Director and/or the QA Manager for further investigation depending on the issue. Corrective action is initiated whenever necessary.

19.14.4.4 The results are then entered or directly transferred into the computer database and a hard copy (or .pdf) is printed for the client.

19.14.4.5 As a final review prior to the release of the report, the Project Manager reviews the results for appropriateness and completeness. This review and approval ensures that client requirements have been met and that the final report has been properly completed. The process includes, but is not limited to, verifying that chemical relationships are evaluated, COC is followed, cover letters/ narratives are present, flags are appropriate, and project specific requirements are met.

19.14.4.6 Any project that requires a data package is subject to a tertiary data review for transcription errors and acceptable quality control requirements. The Project Manager then signs the final report and creates the invoice. When complete, the report is issued to the client.

19.14.5 Manual Integrations

Computerized data systems provide the analyst with the ability to re-integrate raw instrument data in order to optimize the interpretation of the data. Though manual integration of data is an invaluable tool for resolving variations in instrument performance and some sample matrix problems, when used improperly, this technique would make unacceptable data appear to meet quality control acceptance limits. Improper re-integrations lead to legally indefensible data, a poor reputation, or possible laboratory decertification. Because guidelines for re-integration of data are not provided in the methods and most methods were written prior to widespread implementation of computerized data systems, the laboratory trains all analytical staff on proper manual integration techniques using SOP CA-Q-S-002 as the guidelines**.**

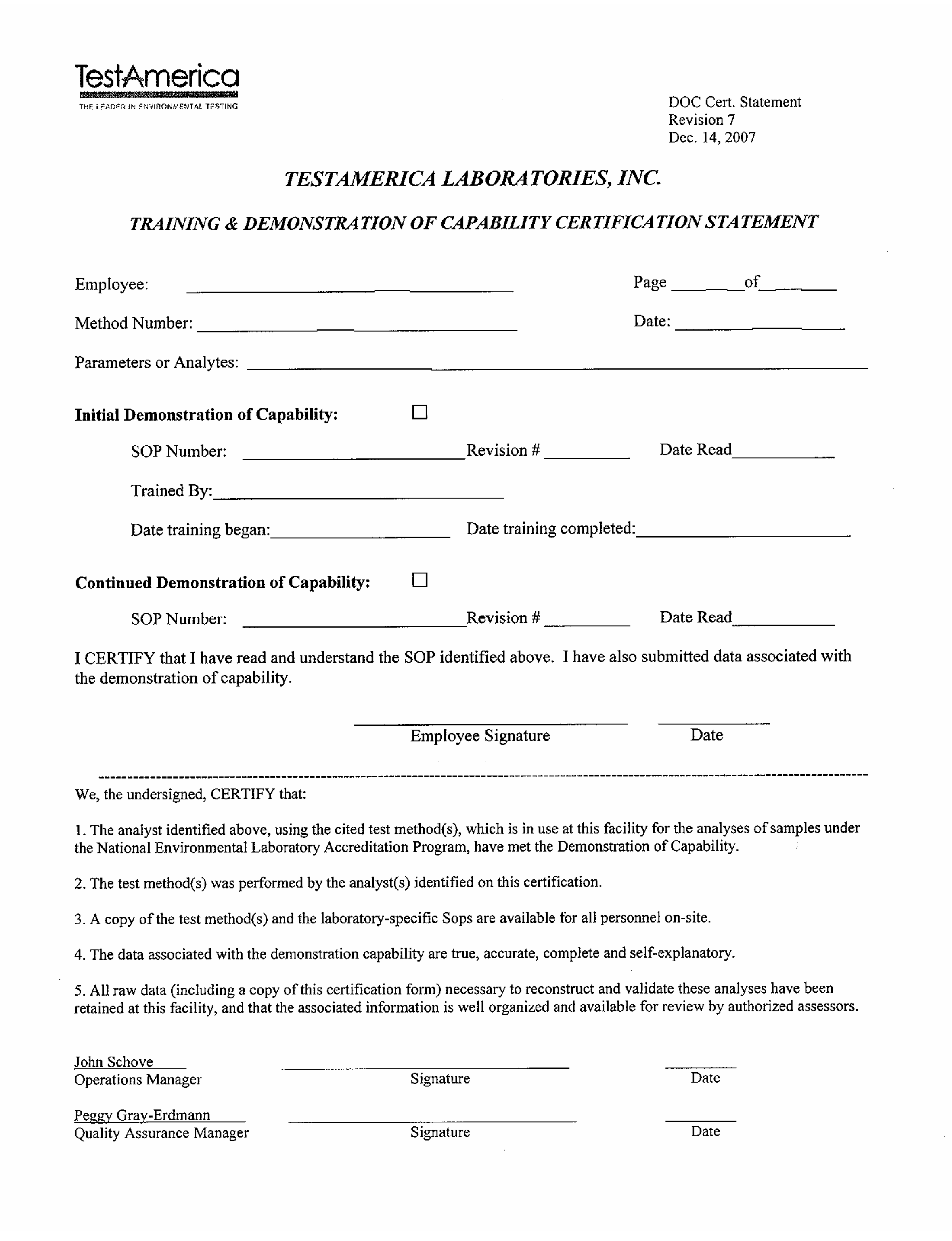
19.14.5.1 The analyst must adjust baseline or the area of a peak in some situations, for example when two compounds are not adequately resolved or when a peak shoulder needs to be separated from the peak of interest. The analyst must use professional judgment and common sense to determine when manual integrating is required. Analysts are encouraged to ask for assistance from a senior analyst or manager when in doubt.

19.14.5.2 Analysts shall not increase or decrease peak areas for the sole purpose of achieving acceptable QC recoveries that would have otherwise been unacceptable. The intentional recording or reporting of incorrect information (or the intentional omission of correct information) is against company principals and policy and is grounds for immediate termination.

19.14.5.3 Client samples, performance evaluation samples, and quality control samples are all treated equally when determining whether or not a peak area or baseline should be manually adjusted.

19.14.5.4 All manual integrations receive a second level review. Manual integrations must be indicated on an expanded scale “after” chromatograms such that the integration performed can be easily evaluated during data review. Expanded scale “before” chromatograms are also required for all manual integrations on QC parameters (calibrations, calibration verifications, laboratory control samples, internal standards, surrogates, etc.) unless the laboratory has another documented corporate approved procedure in place that can demonstrate an active process for detection and deterrence of improper integration practices.

Figure 19-1.
Example - Demonstration of Capability Documentation



TestAmerica
THE LEADER IN ENVIRONMENTAL TESTING

DOC Cert. Statement
Revision 7
Dec. 14, 2007

TESTAMERICA LABORATORIES, INC.

TRAINING & DEMONSTRATION OF CAPABILITY CERTIFICATION STATEMENT

Employee: ______________________ Page ______of________

Method Number: ______________________ Date: ______________

Parameters or Analytes: ____

Initial Demonstration of Capability: ☐

SOP Number: ____________________Revision # __________ Date Read____________

Trained By:______________________________

Date training began:__________________ Date training completed:______________________

Continued Demonstration of Capability: ☐

SOP Number: ____________________Revision # __________ Date Read____________

I CERTIFY that I have read and understand the SOP identified above. I have also submitted data associated with the demonstration of capability.

______________________________ ____________
Employee Signature Date

We, the undersigned, CERTIFY that:

1. The analyst identified above, using the cited test method(s), which is in use at this facility for the analyses of samples under the National Environmental Laboratory Accreditation Program, have met the Demonstration of Capability.

2. The test method(s) was performed by the analyst(s) identified on this certification.

3. A copy of the test method(s) and the laboratory-specific Sops are available for all personnel on-site.

4. The data associated with the demonstration capability are true, accurate, complete and self-explanatory.

5. All raw data (including a copy of this certification form) necessary to reconstruct and validate these analyses have been retained at this facility, and that the associated information is well organized and available for review by authorized assessors.

John Schove
Operations Manager
______________________________ Signature ____________ Date

Peggy Gray-Erdmann
Quality Assurance Manager
______________________________ Signature ____________ Date

SECTION 20

EQUIPMENT (AND CALIBRATIONS)
(NELAC 5.5.5)

20.1 OVERVIEW

The laboratory purchases the most technically advanced analytical instrumentation for sample analyses. Instrumentation is purchased on the basis of accuracy, dependability, efficiency and sensitivity. Each laboratory is furnished with all items of sampling, preparation, analytical testing and measurement equipment necessary to correctly perform the tests for which the laboratory has capabilities. Each piece of equipment is capable of achieving the required accuracy and complies with specifications relevant to the method being performed. Before being placed into use, the equipment (including sampling equipment) is calibrated and checked to establish that it meets its intended specification. The calibration routines for analytical instruments establish the range of quantitation. Calibration procedures are specified in laboratory SOPs. A list of laboratory equipment and instrumentation is presented in Table 20-1***.***

Equipment is only operated by authorized and trained personnel. Manufacturer's instructions for equipment use are readily accessible to all appropriate laboratory personnel.

20.2 PREVENTIVE MAINTENANCE

20.2.1 The laboratory follows a well-defined maintenance program to ensure proper equipment operation and to prevent the failure of laboratory equipment or instrumentation during use. This program of preventive maintenance helps to avoid delays due to instrument failure.

20.2.2 Routine preventive maintenance procedures and frequency, such as lubrication, cleaning, and replacements, should be performed according to the procedures outlined in the manufacturer's manual. Qualified personnel must also perform maintenance when there is evidence of degradation of peak resolution, a shift in the calibration curve, loss of sensitivity, or failure to continually meet one of the quality control criteria.

20.2.3 Table 20-2 lists examples of scheduled routine maintenance. It is the responsibility of each Department Manager to ensure that instrument maintenance logs are kept for all equipment in his/her department. Preventative maintenance procedures may also be outlined in analytical SOPs or instrument manuals. (Note: for some equipment, the log used to monitor performance is also the maintenance log. Multiple pieces of equipment may share the same log as long as it is clear as to which instrument is associated with an entry.)

20.2.4 Instrument maintenance logs are controlled and are used to document instrument problems, instrument repair and maintenance activities. Maintenance logs shall be kept for all major pieces of equipment. Instrument maintenance logs may also be used to specify instrument parameters.

20.2.4.1 Documentation must include all major maintenance activities such as contracted preventive maintenance and service and in-house activities such as the replacement of electrical components, lamps, tubing, valves, columns, detectors, cleaning and adjustments.

20.2.4.2 Each entry in the instrument log includes the Analyst's initials, the date, a detailed description of the problem (or maintenance needed/scheduled), a detailed explanation of the solution or maintenance performed, and a verification that the equipment is functioning properly (state what was used to determine a return to control. e.g. CCV run on *‘date’* was acceptable, or instrument recalibrated on ‘date’ with acceptable verification, etc.) must also be documented in the instrumentation records.

20.2.4.3 When maintenance or repair is performed by an outside agency, service receipts detailing the service performed can be affixed into the logbooks adjacent to pages describing the maintenance performed. This stapled in page must be signed across the page entered and the logbook so that it is clear that a page is missing if only half a signature is found in the logbook.

20.2.5 If an instrument requires repair (subjected to overloading or mishandling, gives suspect results, or otherwise has shown to be defective or outside of specified limits) it shall be taken out of operation and tagged as out of service or otherwise isolated until such a time as the repairs have been made and the instrument can be demonstrated as operational by calibration and/or verification or other test to demonstrate acceptable performance. The laboratory shall examine the effect of this defect on previous analyses

20.2.6 In the event of equipment malfunction that cannot be resolved, service shall be obtained from the instrument vendor manufacturer, or qualified service technician, if such a service can be tendered. If on-site service is unavailable, arrangements shall be made to have the instrument shipped back to the manufacturer for repair. Back up instruments, which have been approved, for the analysis shall perform the analysis normally carried out by the malfunctioning instrument. If the back up is not available and the analysis cannot be carried out within the needed timeframe, the samples shall be subcontracted.

If an instrument is sent out for service or transferred to another facility, it must be recalibrated and verified (including new initial MDL study) prior to return to lab operations.

20.3 SUPPORT EQUIPMENT

This section applies to all devices that may not be the actual test instrument, but are necessary to support laboratory operations. These include but are not limited to: balances, ovens, refrigerators, freezers, incubators, water baths, field sampling devices, temperature measuring devices and volumetric dispensing devices if quantitative results are dependent on their accuracy, as in standard preparation and dispensing or dilution into a specified volume. All raw data records associated with the support equipment are retained to document instrument performance. Laboratory SOPs BF-GP-001,”Calibration of Autopipettes and Repipetters” and BF-GP-002, “Support Equipment: Maintenance, Record Keeping and Corrective Actions of Analytical Balances, Temperature Control Devises and Reagent Water” provide additional detail on the monitoring and record keeping for support equipment.

20.3.1 Weights and Balances

The accuracy of the balances used in the laboratory is checked every working day, before use. All balances are placed on stable counter tops.

Each balance is checked prior to initial serviceable use with at least two certified ASTM type 1 weights spanning its range of use (weights that have been calibrated to ASTM type 1 weights may also be used for daily verification). ASTM type 1 weights used only for calibration of other weights (and no other purpose) are inspected for corrosion, damage or nicks at least annually and if no damage is observed, they are calibrated at least every 5 years by an outside calibration laboratory. Any weights (including ASTM Type 1) used for daily balance checks or other purposes are recalibrated/recertified annually to NIST standards (this may be done internally if laboratory maintains “calibration only” ASTM type 1 weights).

All balances are serviced annually by a qualified service representative, who supplies the laboratory with a certificate that identifies traceability of the calibration to the NIST standards.

All of this information is recorded in logs, and the recalibration/recertification certificates are kept on file.

20.3.2 pH, Conductivity, and Turbidity Meters

The pH meters used in the laboratory are accurate to ± 0.1 pH units, and have a scale readability of at least 0.05 pH units. The meters automatically compensate for the temperature, and are calibrated with at least two working range buffer solutions before each use.

Conductivity meters are also calibrated before each use with a known standard to demonstrate the meters do not exceed an error of 1% or one umhos/cm.

Turbidity meters are also calibrated before each use. All of this information is documented in logs.

Consult pH and Conductivity, and Turbidity SOPs for further information.

20.3.3 Thermometers

All reusable thermometers are calibrated on an annual basis with a NIST-traceable thermometer. Disposable glycol thermometers are discarded upon expiration and replaced with newly purchased thermometers. IR thermometers are verified daily and calibrated annually. Digital probes and thermocouples are calibrated quarterly.

The NIST thermometer is recalibrated every five years (unless thermometer has been exposed to temperature extremes or apparent separation of internal liquid) by an approved outside service and the provided certificate of traceability is kept on file. The NIST thermometer(s) have increments of 1 degree (0.5 degree or less increments are required for drinking water microbiological laboratories) and have ranges applicable to method and certification requirements. The NIST traceable thermometer is used for no other purpose than to calibrate other thermometers.

All of this information is documented in logbooks. Monitoring method-specific temperatures, including incubators, heating blocks, water baths, and ovens, is documented in method-specific logbooks. More information on this subject can be found in the laboratory SOP BF-GP-020, “Thermometer Calibration”.

20.3.4 Refrigerators/Freezer Units, Waterbaths, Ovens and Incubators

The temperatures of all refrigerator units and freezers used for sample and standard storage are monitored each working day.

Ovens, waterbaths and incubators are monitored on days of use.

All of this equipment has a unique identification number, and is assigned a unique thermometer for monitoring.

Sample storage refrigerator temperatures are kept between > 0ºC and ≤ 6 ºC.

Specific temperature settings/ranges for other refrigerators, ovens waterbaths, and incubators can be found in method specific SOPs.

All of this information is documented in Daily Temperature Logbooks and method-specific logbooks.

20.3.5 Autopipettors, Dilutors, and Syringes

Mechanical volumetric dispensing devices including burettes (except Class A Glassware) are given unique identification numbers and the delivery volumes are verified gravimetrically at a minimum on a quarterly basis. Glass micro-syringes are considered the same as Class A glassware.

For those dispensers that are not used for analytical measurements, a label is applied to the device stating that it is not calibrated. Any device not regularly verified can not be used for any quantitative measurements.

Micro-syringes are purchased from Hamilton Company. Each syringe is traceable to NIST. The laboratory keeps on file an “Accuracy and Precision Statement of Conformance” from Hamilton attesting established accuracy.

20.3.6 Field Sampling Devices (Isco Auto Samplers)

Each Auto Sampler (ISCO) is assigned a unique identification number in order to keep track of the calibration. This number is also recorded on the sampling documentation.

The Auto Sampler is calibrated monthly (or if not utilized monthly, immediately prior to its usage) by setting the sample volume to 100ml and recording the volume received. The results are filed in a logbook/binder. The Auto Sampler is programmed to run three (3) cycles and each of the three cycles is measured into a graduated cylinder to verify 100ml are received.

If the RSD (Relative Standard Deviation) between the 3 cycles is greater than 10%, the procedure is repeated and if the result is still greater than 10%, then the Auto Sampler is taken out of service until it is repaired and calibration verification criteria can be met. The results of this check are kept in a logbook/binder.

Additional calibration and use information is detailed in laboratory SOP BF-FS-006, “Calibration of Field Meter”.

20.4 INSTRUMENT CALIBRATIONS

Calibration of analytical instrumentation is essential to the production of quality data. Strict calibration procedures are followed for each method. These procedures are designed to determine and document the method detection limits, the working range of the analytical instrumentation and any fluctuations that may occur from day to day.

Sufficient raw data records are retained to allow an outside party to reconstruct all facets of the initial calibration. Records contain, but are not limited to, the following: calibration date, method, instrument, analyst(s) initials or signatures, analysis date, analytes, concentration, response, type of calibration (Avg RF, curve, or other calculations that may be used to reduce instrument responses to concentration.)

Sample results must be quantitated from the initial calibration and may not be quantitated from any continuing instrument calibration verification unless otherwise required by regulation, method or program.

If the initial calibration results are outside of the acceptance criteria, corrective action is performed and any affected samples are reanalyzed if possible. If the reanalysis is not possible, any data associated with an unacceptable initial calibration will be reported with appropriate data qualifiers (refer to Section 12).

Note: Instruments are calibrated initially and as needed after that and at least annually.

20.4.1 Calibration Standards

Calibration standards are prepared using the procedures indicated in the Reagents and Standards section of the determinative method SOP.

20.4.1.1 Standards for instrument calibration are obtained from a variety of sources. All standards are traceable to national or international standards of measurement, or to national or international standard reference material.

20.4.1.2 The lowest concentration calibration standard that is analyzed during an initial calibration must be at or below the stated reporting limit for the method based on the final volume of extract (or sample).

20.4.1.3 All initial calibrations are verified with a standard obtained from a second source and traceable to a national standard, when available (or vendor certified different lot if a second source is not available). For unique situations, such as air analysis where no other source or lot is available, a standard made by a different analyst would be considered a second source. This verification occurs immediately after the calibration curve has been analyzed, and before the analysis of any samples.

20.4.2 Calibration Verification

The calibration relationship established during the initial calibration must be verified at least daily as specified in the laboratory method SOPs in accordance with the referenced analytical methods and NELAC (2003) standard, Section 5.5.5.10. The process of calibration verification applies to both external standard and internal standard calibration techniques, as well as to linear and non-linear calibration models.

Note: The process of calibration verification referred to is fundamentally different from the approach called "calibration" in some methods. As described in those methods, the calibration factors or response factors calculated during calibration are used to update the calibration factors or response factors used for sample quantitation. This approach, while employed in other EPA programs, amounts to a daily single-point calibration.

All target analytes and surrogates, including those reported as non-detects, must be included in periodic calibration verifications for purposes of retention time confirmation and to demonstrate that calibration verification criteria are being met.

All samples must be bracketed by periodic analyses of standards that meet the QC acceptance criteria (e.g., calibration and retention time). The frequency is found in the determinative methods or SOPs.

Note: If an internal standard calibration is being used (basically GCMS) then bracketing standards are not required, only daily verifications are needed. The results from these verification standards must meet the calibration verification criteria and the retention time criteria (if applicable).

20.4.2.1 Verification of Linear Calibrations

Calibration verification for calibrations involves the calculation of the percent drift or the percent difference of the instrument response between the initial calibration and each subsequent analysis of the verification standard. (These calculations are available in the laboratory method SOPs.) Verification standards are evaluated based on the % Difference from the average CF or RF of the initial calibration or based on % Drift or % Recovery if a linear or quadratic curve is used.

20.4.2.2 Verification of a Non-Linear Calibration

Calibration verification of a non-linear calibration is performed using the percent drift or percent recovery calculations.

Regardless of whether a linear or non-linear calibration model is used, if initial verification criterion is not met, then no sample analyses may take place until the calibration has been verified or a new initial calibration is performed that meets the specifications listed in the method SOPs. If the calibration cannot be verified after the analysis of a single verification standard, then adjust the instrument operating conditions and/or perform instrument maintenance, and analyze another aliquot of the verification standard. If the calibration cannot be verified with the second standard, then a new initial calibration is performed.

20.5 TENTATIVELY IDENTIFIED COMPOUNDS (TICS) – GC/MS ANALYSIS

For samples containing components not associated with the calibration standards, a library search may be made for the purpose of tentative identification. The necessity to perform this type of identification will be determined by the purpose of the analyses being conducted. Data system library search routines should not use normalization routines that would misrepresent the library or unknown spectra when compared to each other.

Note: If the TIC compound is not part of the client target analyte list but is calibrated by the laboratory and is both qualitatively and/or quantitatively identifiable, it should not be reported as a TIC. If the compound is reported on the same form as true TICs, it should be qualified and/or narrated that the reported compound is qualitatively and quantitatively (if verification in control) reported compared to a known standard that is in control (where applicable).

For example, the RCRA permit or waste delisting requirements may require the reporting of non-target analytes. Only after visual comparison of sample spectra with the nearest library searches may the analyst assign a tentative identification. See laboratory SOP’s BF-MB-005 and BF-MV-007 for guidelines for making tentative identifications

Note:
For general reporting if TICs are requested, the ten (10), largest non-target analyte peaks whose area count exceeds 10% of the nearest internal standard will be termed “Tentatively Identified Compounds” (TICs). More or fewer TICs may be identified based on client requirements.

20.6 GC/MS TUNING

Prior to any GCMS analytical sequence, including calibration, the instrument parameters for the tune and subsequent sample analyses within that sequence must be set.

Prior to tuning/auto-tuning the mass spec, the parameters may be adjusted within the specifications set by the manufacturer or the analytical method. These generally don't need any adjustment but it may be required based on the current instrument performance. If the tune verification does not pass it may be necessary to clean the source or perform additional maintenance. Any maintenance is documented in the maintenance log.

Table 20-1. Laboratory Equipment and Instrumentation – TestAmerica Buffalo

Instrument Type	Manufacturer	Model Number	Serial Number	Year Put into Service	Condition When Received
GC/MS	Hewlett Packard	5973	US44621446	2005	new
	Hewlett Packard	5973	US52420646	2005	new
	Hewlett Packard	5973	US41720721	2004	new
	Hewlett Packard	5973	US35120354	2004	new
	Hewlett Packard	5973	US41720707	2004	new
	Hewlett Packard	5973	US10241053	2003	new
	Hewlett Packard	5973	US30965634	2003	new
	Hewlett Packard	5973	US03965692	2003	new
	Hewlett Packard	5973	US05060076	2001	new
	Hewlett Packard	5973	US05060084	2001	new
	Hewlett Packard	5973	US03950346	2001	new
	Hewlett Packard	5973	US82321636	2001	new
GC	Hewlett Packard	6890 dual uECD	CN10520009	2005	new
	Hewlett Packard	6890 dual uECD	CN10520010	2005	new
	Hewlett Packard	6890 dual uECD	CN10448015	2005	new
	Hewlett Packard	5890II dual ECD	3336A53126	1994	new
	Hewlett Packard	5890II dual ECD	3336A63465	1994	new
	Hewlett Packard	5890II dual ECD	3336A53464	1994	new
	Hewlett Packard	5890II dual ECD	3336A53463	1994	new
	Hewlett Packard	5890II dual ECD	3336A54409	1994	new
	Hewlett Packard	5890II dual ECD	3336A54408	1994	new
	Hewlett Packard	5890II FID/FID	3115A34892	1994	new
	Hewlett Packard	5890II PID/FID	3336A60622	1994	new
	Hewlett Packard	5890II Hall/PID	3235A54089	1994	new
	Hewlett Packard	5890II PID/FID	3336A53465	1994	new
	Hewlett Packard	5890II dual FID	3336A53727	1994	new
	Hewlett Packard	5890II dual ECD	3310A47661	1993	new
	Hewlett Packard	5890II dual ECD	3336A53325	1993	new
	Hewlett Packard	5890II PID/FID	3133A37157	1993	new
	Hewlett Packard	5890II dual ECD	3203A42206	1992	new
	Hewlett Packard	5890II dual FID	3019A28433	1991	new

Instrument Type	Manufacturer	Model Number	Serial Number	Year Put into Service	Condition When Received
	Hewlett Packard	5890II Hall/PID	3121A35782	1990	new
LC	Hewlett Packard	1100 HPLC Fluor./DAD	DE92001578	2000	new
Metals	Perkin Elmer	Elan 9000 ICP-MS	P0230202	2002	new
	Leeman	PS200 II	HG9045	2000	new
	Leeman	PS200 II	HG0033	2000	new
	Thermo Jarrell Ash	ICP61E Trace	334490	1995	new
	Thermo Jarrell Ash	ICP61E Trace	382590	1995	new

Instrument Type	Manufacturer	Model Number	Serial Number	Year Put into Service	Condition When Received
Water Quality	Konelab	20XT	E3719731	2005	new
	Thermo ECA	1200 TOX/AOX	2004.901	2004	new
	Dionex Ion Chromatograph	DX-120	20126	2004	new
	Konelab	20	S5019455	2004	new
	Glastron	CN Midi-distillation	2502	2003	new
	Glastron	Phenol Midi-distillation	2069	2003	new
	Glastron	Phenol Midi-distillation	2053	2003	new
	Labtronics	BOD Magic - Autoanalyzer	270H3XB531	2004	new
	Labtronics	BOD Magic - Autoanalyzer	270J2XB669	2003	new
	ManTech	PC Titrator	MS-OK2-607	2003	new
	HACH Spectrophotometer	DR/2500	30200004886	2003	new
	Dionex Ion Chromatograph	DX-120	2060196	2002	new
	OI Carbon Analyzer	1010 #2	H014710903	2000	new
	Spectronic Genesis	4001/4	3SGC199091	2000	new
	Lachat Quickchem	8000 Autoanalyzer	A83000-1527	2000	new
	OI Carbon Analyzer	1010 #1	H92170411	1999	new
	Lachat Quickchem	8000 Autoanalyzer	A83000-1439	1999	new
	Dionex Ion Chromatograph	DX-120	99010157	1999	new
	Orion	Ion Meter 230A	2229	1999	new

Instrument Type	Manufacturer	Model Number	Serial Number	Year Put into Service	Condition When Received
	VWR Ion	Meter 2100	1063	1997	new
	YSI	Oxygen Meter 57	93J09826	1995	new
	Lab-Line	Hi-Lo BOD chamber	391-010	1994	new
	Fischer	Accumet Ion Meter 925	860	1991	new
Sample Preparation	J2 ACCUPREP GPC		03F-10723	2003	new
	TurboVap II	TurboVap II	TV9445N5816	1996	new
	TurboVap II	TurboVap II	TV9427N4133	1996	new
	TurboVap II	TurboVap II	TV944N5819	1996	new
	TurboVap II	TurboVap II	TV944N5820	1996	new
	TurboVap II	TurboVap II	TV0024N9623	2000	new
	TurboVap II	TurboVap II	TV0022N9604	2000	new
	TurboVap II	TurboVap II	TV0312N11592	2003	new
	TurboVap II	TurboVap II	TV0312N11591	2003	new
	Organomation	Rot-X-Tractor	16902	1999	new
	Organomation	Rot-X-Tractor	16907	1999	new
	Organomation	Rot-X-Tractor	16913	1999	new
	Heat Systems Sonicator	#XL-2020	G1647/C5659	1994	new
Sample Preparation (Continued)	Heat Systems Sonicator	#XL-2020	G2665/C5674	1994	new
	Heat Systems Sonicator	#XL-2020	G2620/C5660	1994	new
	Heat Systems Sonicator	#XL-2020	G2245/C6328	1995	new
	Heat Systems Sonicator	#XL-2020	G2621/C6733	1995	new
	Heat Systems Sonicator	#XL-2020	G2713/C6732	1995	new
	Heat Systems Sonicator	#XL-2020	G1643/C6837	1995	new
	Heat Systems Sonicator	#XL-2020	G2742/C6842	1995	new

Table 20-2.

Schedule of Routine Maintenance

Instrument	Procedure	Frequency
Leeman Mercury Analyzer	Check tubing for wear	Daily
	Fill rinse tank with 10% HCl	Daily
	Change dryer tube	As Needed
	Fill reductant bottle with 10% Stannous Chloride	Daily
ICP & ICP/MS	Check pump tubing	Daily
	Check liquid argon supply	Daily
	Check fluid level in waste container	Daily
	Check re-circulator levels	Monthly
	Clean or replace filters	As required
	Check torch	Daily
	Check sample spray chamber for debris	Monthly
	Clean and align nebulizer	Monthly
	Change pump oil	Monthly
	Change Cones	As required
	Change printer cartridge	As required
	Replace pump tubing	As required
UV-Vis Spectrophotometer	Clean ambient flow cell	As required
	Precision check/alignment of flow cell	As required
	Wavelength verification check	Annually
Auto Analyzers	Clean sampler	Daily
	Check all tubing	Daily
	Clean inside of colorimeter	Daily
	Clean pump well and pump rollers	Quarterly
	Clean wash fluid receptacle	Weekly
	Oil rollers/chains/side rails	Weekly
	Clean optics and cells	Quarterly
Agilent GC/MS	Pump oil-level check	Monthly
	Pump oil changing	Annually
	Analyzer bake-out	As required
	Analyzer cleaning	As required
	Resolution adjustment	As required
	COMPUTER SYSTEM AND PRINTER:	
	Air filter cleaning	As required
	Change data system air filter	As required
	Printer head carriage lubrication	As required
	Paper sprocket cleaning	As required
	Drive belt lubrication	As required

Instrument	**Procedure**	**Frequency**
Gas Chromatograph	Compare standard response to previous day or since last initial calibration	Daily
	Check carrier gas flow rate in column	Daily via use of known compound retention
	Check temp. of detector, inlet, column oven	Daily
	Septum replacement	As required
	Glass wool replacement	As required
	Check system for gas leaks with SNOOP	W/cylinder change as required
	Check for loose/frayed power wires and insulation	As Required As Required
	Bake injector/column	As Required
	Change/remove sections of guard column	As Required
	Replace connectors/liners	As Required
	Change/replace column(s)	
Electron Capture Detector (ECD)	Detector wipe test (Ni-63)	Semi-annually
	Detector cleaning	As required
Flame Ionization Detector (FID)	Detector cleaning	As required
Photoionization Detector (PID)	Change O-rings	As required
	Clean lamp window	As required
HPLC	Change guard columns	As required
	Change lamps	As required
	Change pump seals	Semi-annually or as required
	Replace tubing	As required
	Change fuses in power supply	As required
	Filter all samples and solvents	Daily
	Change autosampler rotor/stator	As required
Vacuum Pumps/ Air Compressor	Drained	Weekly
	Belts checked	Monthly
	Lubricated	Semi-annually
Centrifuge	Check brushes and bearings	Every 6 months or as needed

Table 20-3.

Periodic Calibration

Instrument	Type of Calibration/ Number of Standards	Frequency	Acceptance Limits	Corrective Action
Analytical Balance	Accuracy determined using “S” NIST traceable weights. Minimum of 2 standards bracketing the weight of interest. Inspected and calibrated by A2LA accredited person annually.	Daily, when used Annual	± 0.2%	Clean, check level, insure lack of drafts, and that unit is warmed up, recheck. If fails, call service.
Top Loading Balance	Accuracy determined using “S” NIST traceable. Minimum of 2 standards bracketing the weight of interest. Inspected and calibrated by A2LA accredited person annually.	Daily, when used Annual	± 0.5%	Clean. Replace.
NIST Certified Weights	Accuracy determined by accredited weights and measurement laboratory.	1 year	As per certificate.	Replace.
NIST-Traceable Thermometer	Accuracy determined by accredited measurement laboratory.	5 years	As per certificate.	Replace.
Thermometer	Against NIST-traceable thermometer	Yearly at appropriate temperature range for intended use	± 1.2°C	Replace
Minimum-Maximum Thermometers	Against NIST-traceable thermometer	Yearly	± 1.5°C	Replace
InfraRed Temperature Guns	Against NIST-traceable thermometer Accuracy determined by accredited measurement laboratory.	Daily at appropriate temperature range for intended use. Annual	± 1.5°C	Repair/replace

Instrument	Type of Calibration/ Number of Standards	Frequency	Acceptance Limits	Corrective Action
Dial-type Thermometers	Against NIST-traceable thermometer	Quarterly at appropriate temperature range for intended use.	± 1.5°C	Replace
Refrigerator	Temperature checked using NIST-traceable thermometer.	Daily. If out of range, check again in two hours.	0-6°C	Adjust. Repair. While waiting for repair, seal door, attach "Out of Service" sign, move items to functional unit. Notify supervisor.
Freezer	Temperature checked using NIST-traceable thermometer	Daily. If out of range, check again in two hours.	(-10)-(-20)°C	Adjust. Repair. While waiting for repair, seal door, attach "Out of Service" sign, move items to functional unit. Notify supervisor.
Oven	Temperature checked using NIST-traceable thermometer.	When in use.	104 ± 1°C (drying) 180 ± 2°C (TDS)	Adjust. Replace.
Water Bath	Temperature checked using NIST-traceable thermometer.	When in use.	± 2°C	Adjust. Replace.
Volumetric Dispensing Devices (Eppendorf ® pipette, automatic dilutor or dispensing devices)	One delivery by weight. Using DI water or solvent of use, dispense into tared vessel. Record weight with device ID number. Calibrate using 4 replicate gravimetric measurements	Each day of use Quarterly	± 2% Calculate accuracy by dividing weight by stated volume times 100 for percent.	Adjust. Replace.
Glass Microliter Syringes	None	Accuracy must be initially demonstrated if syringe was not received with a certificate attesting to established accuracy.	± 1%	Not applicable.

Instrument	Type of Calibration/ Number of Standards	Frequency	Acceptance Limits	Corrective Action
Deionized Water	Check in-line conductivity meter on system with conductivity meter in Inorganics Department.	Daily	<1.0 µmho at 25°C	Record on log. Report discrepancies to QA Manager, Operations Manager or Technical Director.

SECTION 21

MEASUREMENT TRACEABILITY
(NELAC 5.5.6)

21.1 OVERVIEW

Traceability of measurements shall be assured using a system of documentation, calibration, and analysis of reference standards. Laboratory equipment that are peripheral to analysis and whose calibration is not necessarily documented in a test method analysis or by analysis of a reference standard shall be subject to ongoing certifications of accuracy. At a minimum, these must include procedures for checking specifications of ancillary equipment: balances, thermometers, temperature, Deionized (DI) and Reverse Osmosis (RO) water systems, automatic pipettes and other volumetric measuring devices. (Refer to Section 20.3). With the exception of Class A Glassware (including glass microliter syringes that have a certificate of accuracy), quarterly accuracy checks are performed for all mechanical volumetric devices. Wherever possible, subsidiary or peripheral equipment is checked against standard equipment or standards that are traceable to national or international standards. Class A Glassware should be routinely inspected for chips, acid etching or deformity. If the Class A glassware is suspect, the accuracy of the glassware will be assessed prior to use.

21.2 NIST-TRACEABLE WEIGHTS AND THERMOMETERS

Reference standards of measurement shall be used for calibration only and for no other purpose, unless it can be shown that their performance as reference standards would not be invalidated.

For NIST-traceable weights and thermometers, the laboratory requires that all calibrations be conducted by a calibration laboratory accredited by A2LA, NVLAP (National Voluntary Laboratory Accreditation Program), (APLAC (Asia-Pacific Laboratory Accreditation Cooperation), or EA (European Cooperation for Accreditation). A certificate and scope of accreditation is kept on file at the laboratory.

The calibration report or certificate submitted to ***TestAmerica Buffalo*** contains, in a well designed format, a traceability statement, the conditions under which the calibrations were made in the context of any potential influence, a compliance statement with an identified metrological specification and the pertinent clauses, a clearly identified record of the quantities and functional test results before and after re-calibration, and no recommendation on the calibration interval. Opinions and interpretations of results are presented along with the basis upon which they were made and identified as such. The report may be submitted by facsimile or other electronic means as long as the requirements of the International Standard are achieved. If significant amendments are made to a calibration certificate, a supplemental certificate for the serial-number-specified piece of equipment is so identified. When a new certificate is offered, it uniquely identifies and references the one it replaces. All calibration reports are filed in the QA Office.

An external certified service engineer services laboratory balances on an annual basis. This service is documented on each balance with a signed and dated certification sticker. Balance calibrations are checked each day of use. All mercury thermometers are calibrated annually against a traceable reference thermometer. Temperature readings of ovens, refrigerators, and incubators are checked on each day of use.

21.3 REFERENCE STANDARDS / MATERIALS

Reference standards/materials, where commercially available, are traceable to certified reference materials. Commercially prepared standard materials are purchased from vendors accredited by A2LA or NVLAP with an accompanying Certificate of Analysis that documents the standard purity. If a standard cannot be purchased from a vendor that supplies a Certificate of Analysis, the purity of the standard is documented by analysis. The receipt of all reference standards must be documented. Reference standards are labeled with a unique Standard Identification Number and expiration date. All documentation received with the reference standard is retained as a QC record and references the Standard Identification Number.

All reference, primary and working standards/materials, whether commercially purchased or laboratory prepared, must be checked regularly to ensure that the variability of the standard or material from the 'true' value does not exceed method requirements. The accuracy of calibration standards is checked by comparison with a standard from a second source. In cases where a second standard manufacturer is not available, a vendor certified different lot is acceptable for use as a second source. For unique situations, such as air analysis where no other source or lot is available, a standard made by a different analyst would be considered a second source. The appropriate Quality Control (QC) criteria for specific standards are defined in laboratory SOPs. In most cases, the analysis of an Initial Calibration Verification (ICV) or LCS (where there is no sample preparation) is used as the second source confirmation. These checks are generally performed as an integral part of the analysis method (e.g. calibration checks, laboratory control samples).

All standards and materials must be stored and handled according to method or manufacturer's requirements in order to prevent contamination or deterioration. Refer to the Corporate Environmental Health & Safety Manual or laboratory SOPs. Method specific information may also be found in the laboratory method SOPs in the "Standards and Reagents" sections. For safety requirements, please refer to method SOPs and the laboratory Environmental Health and Safety Manual.

21.4 DOCUMENTATION AND LABELING OF STANDARDS, REAGENTS, AND REFERENCE MATERIALS

Reagents must be at a minimum the purity required in the test method. The date of reagent receipt and the expiration date are documented. The lots for most of the common solvents and acids are tested for acceptability prior to company wide purchase. Refer to SOP No. CA-Q-S-001, Solvent and Acid Lot Testing and Approval.

All manufacturer or vendor supplied Certificate of Analysis or Purity must be retained, stored appropriately, and readily available for use and inspection. These records are maintained by each department in bound or electronic folders. Records must be kept of the date of receipt and date of expiration of standards, reagents and reference materials. In addition, records of preparation of laboratory standards, reagents, and reference materials must be retained, stored appropriately, and be readily available for use and inspection. For detailed information on documentation and labeling, please refer laboratory SOP BF-GP-019, “Standard Traceability and Preparation” and also to the method specific SOPs.

Commercial materials purchased for preparation of calibration solutions, spike solutions, etc.., are usually accompanied with an assay certificate or the purity is noted on the label. If the assay purity is 96% or better, the weight provided by the vendor may be used without correction. If the assay purity is less than 96% a correction will be made to concentrations applied to solutions prepared from the stock commercial material.

21.4.1 All standards, reagents, and reference materials must be labeled in an unambiguous manner. Standards are logged into the laboratory department’s chemical history log and are assigned a unique identification number. Preparation of working standards or reagents prepared from the stock is documented in the laboratory Department’s Standard Preparation Log. The following information is typically recorded:

- Standard ID
- Description of Standard
- Department
- Preparer’s name
- Final volume and number of vials prepared
- Solvent type and lot number
- Preparation Date
- Expiration Date
- Standard source type (stock or daughter)
- Standard type (spike, surrogate, other)
- Parent standard ID (if applicable)
- Parent Standard Analyte Concentration (if applicable)
- Parent Standard Amount used (if applicable)
- Component Analytes
- Final concentration of each analyte
- Comment section

Records are maintained for standard and reference material preparation. These records show the traceability to purchased stocks or neat compounds. These records also include method of preparation, date of preparation, expiration date and preparer’s name or initials. Preparation procedures are provided in the Method SOPs.

21.4.2 All standards, reagents, and reference materials must be clearly labeled with a minimum of the following information:

- Expiration Date
- Standard ID
- Special Health/Safety warnings if applicable

21.4.3 In addition, the following information may be helpful:

- Date of receipt for commercially purchased items or date of preparation for laboratory prepared items
- Date opened (for multi-use containers, if applicable)
- Description of standard (if different from manufacturer's label or if standard was prepared in the laboratory)
- Concentration (if applicable)
- Initials of analyst preparing standard or opening container

All containers of prepared reagents must include a preparation date, expiration date and an ID number to trace back to preparation.

Procedures for preparation of reagents can be found in the Method SOPs.

Standard ID numbers must be traceable through associated logbooks, worksheets and raw data.

All reagents and standards must be stored in accordance to the following priority: 1) with the manufacturer's recommendations; 2) with requirements in the specific analytical methods as specified in the laboratory SOPs.

SECTION 22.0

SAMPLING
(NELAC 5.5.7)

22.1 OVERVIEW

The laboratory provides sampling services. Sampling procedures are described in the following SOPs:

BF-FS-001	Chain of Custody Documentation
BF-FS-002	Sample Packaging and Shipment Off-Site
BF-FS-003	Groundwater Sampling Field Data Collection
BF-FS-004	Equipment Decontamination
BF-FS-005	Groundwater/Surface Water Sampling
BF-FS-006	Calibration of Field Meter
BF-FS-007	Low Flow Sampling Procedures
BF-FS-008	Surface and Subsurface Soil/Sediment Sampling

22.2 SAMPLING CONTAINERS

The laboratory offers clean sampling containers for use by clients. These containers are obtained from reputable container manufacturers and meet EPA specifications as required. Any certificates of cleanliness that are provided by the supplier are maintained at the laboratory.

22.2.1 Preservatives

Upon request, preservatives are provided to the client in pre-cleaned sampling containers. In some cases containers may be purchased pre-preserved from the container supplier. Whether prepared by the laboratory or bought pre-preserved, the grades of the preservatives are at a minimum:

- Hydrochloric Acid – Reagent ACS (Certified VOA Free) or equivalent
- Methanol – Purge and Trap grade
- Nitric Acid – Instra-Analyzed or equivalent
- Sodium Bisulfate – ACS Grade or equivalent
- Sodium Hydroxide – Instra-Analyzed or equivalent
- Sulfuric Acid – Instra-Analyzed or equivalent
- Sodium Thiosulfate – ACS Grade or equivalent

22.3 DEFINITION OF HOLDING TIME

The date and time of sampling documented on the chain-of-custody (COC) form establishes the day and time zero. As a general rule, when the maximum allowable holding time is expressed in “days” (e.g. 14 days, 28 days), the holding time is based on calendar day measured. Holding times expressed in “hours” (e.g. 6 hours, 24 hours, etc.) are measured from date and time zero. The first day of holding time for time critical parameters ends twenty-four hours after sampling. Holding times for analysis include any necessary reanalysis. However there are some programs that determine holding time compliance based on the date and specific time of analysis compared to the time of sampling regardless of how long the holding time is. These programs will be addressed on a case-by-case basis.

22.4 SAMPLING CONTAINERS, PRESERVATION REQUIREMENTS, HOLDING TIMES

The preservation and holding time criteria specified in the following tables are derived from the source documents for the methods. If method required holding times, this info is in the SOP or preservation requirements are not met, the reports will be qualified using a flag, footnote or case narrative. As soon as possible or “ASAP” is an EPA designation for tests for which rapid analysis is advised, but for which neither EPA nor the laboratory have a basis for a holding time.

22.5 SAMPLE ALIQUOTS / SUBSAMPLING

Taking a representative sub-sample from a container is necessary to ensure that the analytical results are representative of the sample collected in the field. The size of the sample container, the quantity of sample fitted within the container, and the homogeneity of the sample need consideration when sub-sampling for sample preparation. It is the laboratory’s responsibility to take a representative subsample or aliquot of the sample provided for analysis.

Analysts should handle each sample as if it is potentially dangerous. At a minimum, safety glasses, gloves, and lab coats must be worn when preparing aliquots for analysis.

The following information provides general guidance for homogenization and subsampling. For laboratory specific procedures refer to SOP BF-GP-005, “Sample Homogenization and Subsampling”.

SECTION 23

HANDLING OF SAMPLES
(NELAC 5.5.8)

Sample management procedures at the laboratory ensure that sample integrity and custody are maintained and documented from sampling/receipt through disposal.

23.1 CHAIN OF CUSTODY (COC)

The COC form is the written documented history of any sample and is initiated when bottles are sent to the field, or at the time of sampling. This form is completed by the sampling personnel and accompanies the samples to the laboratory where it is received and stored under the laboratory's custody. The purpose of the COC form is to provide a legal written record of the handling of samples from the time of collection until they are received at the laboratory. It also serves as the primary written request for analyses from the client to the laboratory. The COC form acts as a purchase order for analytical services when no other contractual agreement is in effect. An example of a COC form may be found in Figure 23-1.

23.1.1 Field Documentation

The information the sampler needs to provide at the time of sampling on the container label is:

- Sample identification
- Date and time
- Preservative

During the sampling process, the COC form is completed and must be legible (see Figure 23-1). This form includes information such as:

- Client name, address, phone number and fax number (if available)
- Project name and/or number
- The sample identification
- Date, time and location of sampling
- Sample collectors name
- The matrix description
- The container description
- The total number of each type of container
- Preservatives used
- Analysis requested
- Requested turnaround time (TAT)
- Any special instructions
- Purchase Order number or billing information (e.g. quote number) if available
- The date and time that each person received or relinquished the sample(s), including their signed name.

The samples are stored in a cooler with ice, as applicable, and remain solely in the possession of the client's field technician until the samples are delivered to the laboratory. The sample collector must assure that each container is in his/her physical possession or in his/her view at all times, or stored in such a place and manner to preclude tampering. The field technician relinquishes the samples in writing on the COC form to the sample control personnel at the laboratory or to a TestAmerica courier. Samples are only considered to be received by lab when personnel at the laboratory have physical contact with the samples.

Note: Independent couriers are not required to sign the COC form. The COC is usually kept in the sealed sample cooler. The shipping documents are retained with the project files.

24.1.2 Legal / Evidentiary Chain-of-Custody

If samples are identified for legal/evidentiary purposes on the COC or in the project notes, sample management will initiate Strict Chain of Custody procedures as defined in SOP BF-GP-018, "Strict Internal Chain-of-Custody".

23.2 SAMPLE RECEIPT

Samples are received at the laboratory by designated sample receiving personnel and a unique laboratory project identification number is assigned. Each sample container shall be assigned a unique sample identification number that is cross-referenced to the client identification number such that traceability of test samples is unambiguous and documented. Each sample container is affixed with a durable sample identification label. Sample acceptance, receipt, tracking and storage procedures are summarized in the following sections.

23.2.1 Laboratory Receipt

When samples arrive at the laboratory, sample receiving personnel inspect the coolers and samples. The integrity of each sample must be determined by comparing sample labels or tags with the COC and by visual checks of the container for possible damage. Any non-conformance, irregularity, or compromised sample receipt must be documented on a Login – Analytical Receipt Resolution Form and brought to the immediate attention of the client. The COC, shipping documents, documentation of any non-conformance, irregularity, or compromised sample receipt, record of client contact, and resulting instructions become part of the project record.

23.1.1.1 SAMPLE ACCEPTANCE POLICY

The laboratory has a written sample acceptance policy (Figure 23-2) that clearly outlines the circumstances under which samples shall be accepted or rejected. These include:

- a COC filled out completely;
- samples must be properly labeled;
- proper sample containers with adequate volume for the analysis (Sampling Guide) and necessary QC;

- samples must be preserved according to the requirements of the requested analytical method (Sampling Guide);
- sample holding times must be adhered to (Sampling Guide);
- the project manager will be notified if any sample is received in damaged condition.

Data from samples which do not meet these criteria are flagged and the nature of the variation from policy is defined. A copy of the sample acceptance policy is provided to each client prior to shipment of samples.

23.2.1.1 After inspecting the samples, the sample receiving personnel sign and date the COC form, make any necessary notes of the samples' conditions and store them in appropriate refrigerators or storage locations.

23.2.1.2 Any deviations from these checks described in Section 23.1.1.1 that question the suitability of the sample for analysis, or incomplete documentation as to the tests required will be resolved by consultation with the client. If the sample acceptance policy criteria are not met, the laboratory shall either:

- Retain all correspondence and/or records of communications with the client regarding the disposition of rejected samples, or

- Fully document any decision to proceed with sample analysis that does not meet sample acceptance criteria.

Once sample acceptance is verified, the samples are logged into the LIMS according SOP No. BF-SR-002.

23.3 <u>SAMPLE STORAGE</u>

In order to avoid deterioration, contamination or damage to a sample during storage and handling, from the time of receipt until all analyses are complete, samples are stored in refrigerators suitable for the sample matrix. Aqueous samples designated for metals analysis are stored at ambient temperature. In addition, samples to be analyzed for volatile organic parameters are stored in separate refrigerators designated for volatile organic parameters only. Samples are never to be stored with reagents, standards or materials that may create contamination.

To ensure the integrity of the samples during storage, refrigerator blanks are maintained in the volatile sample refrigerators and analyzed at a minimum of every two weeks.

Analysts and technicians provide a request form to the cooler custodian who then retrieves the requested samples. In the absence of the cooler custodian, the analysts may personally retrieve the sample containers allocated to their analysis from the designated refrigerator. The samples are placed on carts, transported the analytical area and analyzed. Following analysis the remaining sample is returned to the refrigerator from which it originally came. All unused portions of samples are returned to the secure sample control area. All samples are kept in the refrigerators for two to four weeks after analysis, which meets or exceeds most sample holding times. After two to four weeks the samples are moved to dry room temperature, sample archive area where they are retained a minimum of 2 weeks after the final report has been issued to the client at which time disposal occurs. Special arrangements may be made to store samples for longer periods of time. Extended archival periods allow additional metal analyses to be performed on the archived sample and assists clients in dealing with legal matters or regulatory issues.

Access to the laboratory is controlled such that sample storage need not be locked at all times unless a project specifically demands it. Samples are accessible to laboratory personnel only. Visitors to the laboratory are prohibited from entering the refrigerator and laboratory areas unless accompanied by an employee of TestAmerica.

23.4 HAZARDOUS SAMPLES AND FOREIGN SOILS

To minimize exposure to personnel and to avoid potential accidents, samples which are known or suspected to be hazardous are segregated and a notification is issued to all laboratory personnel. All hazardous samples are either returned to the client or disposed of appropriately through a hazardous waste disposal firm. All soil samples, including foreign soil samples are heat treated or incinerated in accordance with USDA permit requirements and are transported / disposed by USEPA approved facilities.

Unused portions of samples found or suspected to be hazardous according to state or federal guidelines may be returned to the client upon completion of the analytical work.

23.5 SAMPLE SHIPPING

In the event that the laboratory needs to ship samples, the samples are placed in a cooler with enough ice to ensure the samples remain just above freezing and at or below 6.0°C during transit. The samples are carefully surrounded by packing material to avoid breakage (yet maintain appropriate temperature). For sample shipments which include water/solid volatile organic analyses, a trip blank is enclosed when required by method specifications or state or regulatory programs. The chain-of-custody form is signed by the sample control technician and attached to the shipping paperwork. Samples are generally shipped overnight express or hand-delivered by a TestAmerica courier to maintain sample integrity. All personnel involved with shipping and receiving samples must be trained to maintain the proper chain-of-custody documentation and to keep the samples intact and on ice. The Environmental, Health and Safety Manual contains additional shipping requirements.

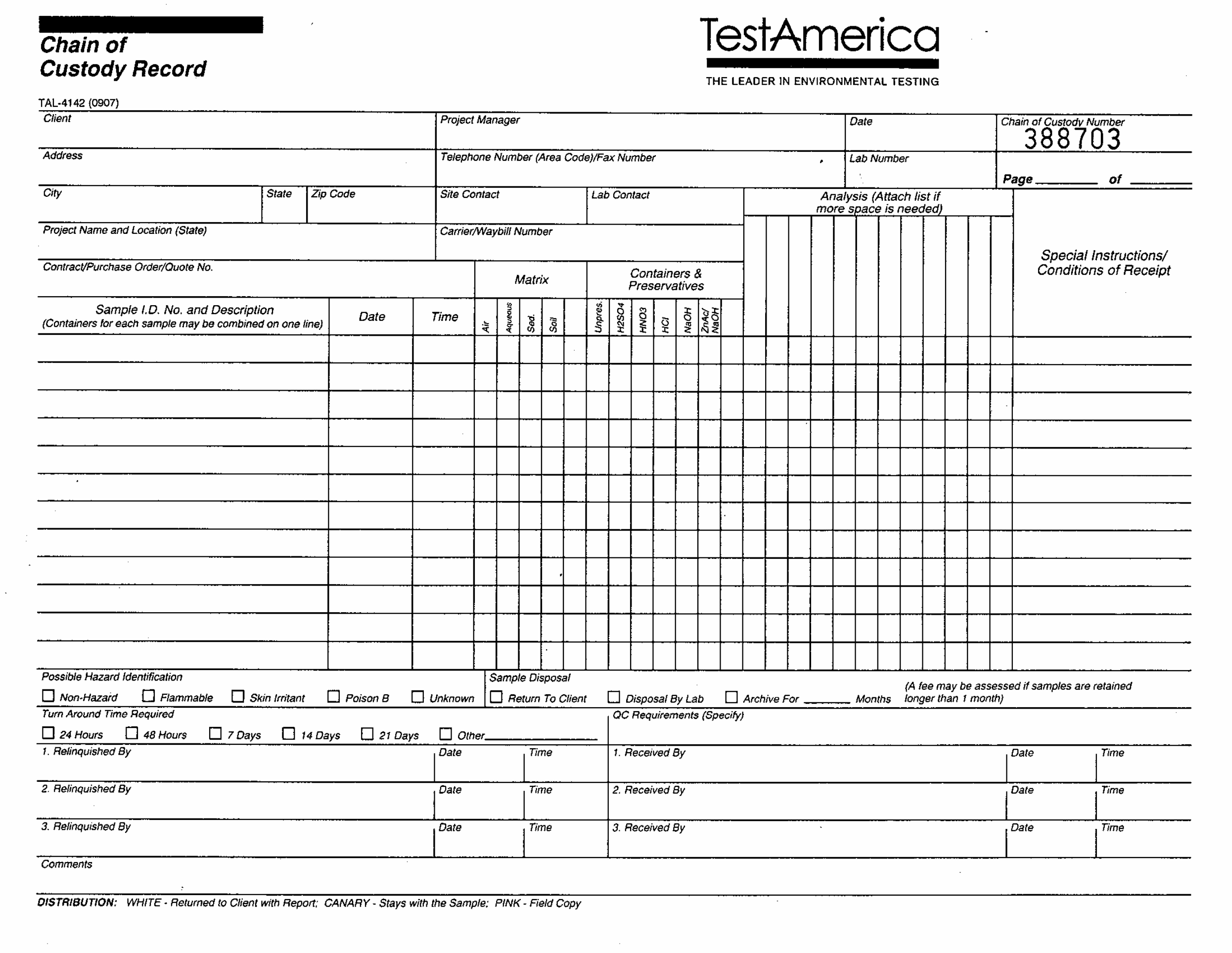
23.6 SAMPLE DISPOSAL

Samples should be retained for a minimum of 2 weeks after the project report is sent, however, provisions may be made for earlier disposal of samples once the holding time is exceeded. Some samples are required to be held for longer periods based on regulatory or client requirements (e.g., 60 days after project report is sent). The laboratory must follow the longer sample retention requirements where required by regulation or client agreement. Several possibilities for sample disposal exist: the sample may be consumed completely during analysis, the sample may be returned to the customer or location of sampling for disposal, or the sample may be disposed of in accordance with the laboratory's waste disposal procedures (SOP: BF-WM-001, "Waste Management".) All procedures in the laboratory Environmental, Health and Safety Manual are followed during disposal. Samples are normally maintained in the laboratory no longer than six weeks from receipt unless otherwise requested. Unused portions of samples found or suspected to be hazardous according to state or federal guidelines may be returned to the client upon completion of the analytical work.

If a sample is part of a known litigation, the affected legal authority, sample data user, and/or submitter of the sample may request to participate in the decision about the sample's disposal. All documentation and correspondence concerning the disposal decision process must be kept on file. Pertinent information includes the date of disposal and nature of disposal (such as sample depletion, hazardous waste facility disposal, return to client). All disposal of sample containers is accomplished through incineration. A Waste Disposal Record should be completed.

Figure 23-1.

Example: Chain of Custody (COC)



Chain of Custody Record

TestAmerica
THE LEADER IN ENVIRONMENTAL TESTING

TAL-4142 (0907)

Client
Project Manager
Date
Chain of Custody Number 388703
Address
Telephone Number (Area Code)/Fax Number
Lab Number
Page ______ of ______
City
State
Zip Code
Site Contact
Lab Contact
Project Name and Location (State)
Carrier/Waybill Number
Contract/Purchase Order/Quote No.

Sample I.D. No. and Description (Containers for each sample may be combined on one line)	Date	Time	Matrix					Containers & Preservatives							Analysis (Attach list if more space is needed)												Special Instructions/ Conditions of Receipt
			Air	Aqueous	Sed.	Soil		Unpres.	H2SO4	HNO3	HCl	NaOH	ZnAc/ NaOH														

Possible Hazard Identification
☐ Non-Hazard ☐ Flammable ☐ Skin Irritant ☐ Poison B ☐ Unknown

Sample Disposal
☐ Return To Client ☐ Disposal By Lab ☐ Archive For ______ Months (A fee may be assessed if samples are retained longer than 1 month)

Turn Around Time Required
☐ 24 Hours ☐ 48 Hours ☐ 7 Days ☐ 14 Days ☐ 21 Days ☐ Other______

QC Requirements (Specify)

1. Relinquished By	Date	Time	1. Received By	Date	Time
2. Relinquished By	Date	Time	2. Received By	Date	Time
3. Relinquished By	Date	Time	3. Received By	Date	Time

Comments

DISTRIBUTION: WHITE - Returned to Client with Report; CANARY - Stays with the Sample; PINK - Field Copy

Figure 23-2.

Example: Sample Acceptance Policy

All incoming work will be evaluated against the criteria listed below. Where applicable, data from any samples that do not meet the criteria listed below will be noted on the laboratory report defining the nature and substance of the variation. In addition the client will be notified either by telephone, fax or e-mail ASAP after the receipt of the samples.

1) Samples must arrive with labels intact with a Chain of Custody filled out completely. The following information must be recorded.
   - *Client name, address, phone number and fax number (if available)*
   - *Project name and/or number*
   - *The sample identification*
   - *Date, time and location of sampling*
   - *The collectors name*
   - *The matrix description*
   - *The container description*
   - *The total number of each type of container*
   - *Preservatives used*
   - *Analysis requested*
   - *Requested turnaround time (TAT)*
   - *Any special instructions*
   - *Purchase Order number or billing information (e.g. quote number) if available*
   - *The date and time that each person received or relinquished the sample(s), including their signed name.*
   - ***The date and time of receipt must be recorded between the last person to relinquish the samples and the person who receives the samples in the lab, and they must be exactly the same.***
   - **Information must be legible**

2) Samples must be properly labeled.
   - Use durable labels (labels provided by TestAmerica are preferred)
   - Include a unique identification number
   - Include sampling date and time & sampler ID
   - Include preservative used.
   - Use indelible ink
   - **Information must be legible**

3) Proper sample containers with adequate volume for the analysis and necessary QC are required for each analysis requested.

4) Samples must be preserved according to the requirements of the requested analytical method. See lab Sampling Guide.
   Note: Samples that are hand delivered to the laboratory immediately after collection may not have had time to cool sufficiently. In this case the samples will be considered acceptable as long as there is evidence that the chilling process has begun (arrival on ice).

- Chemical preservation (pH) will be verified prior to analysis and the project manager will be notified immediately if there is a discrepancy. If analyses will still be performed, all affected results will be flagged to indicate improper preservation.

- For Volatile Organic analyses in drinking water (Methods 502.2 or 524.2). Residual chlorine must be neutralized prior to preservation. If there is prior knowledge that the samples are not chlorinated, state it on the COC and use the VOA vials pre-preserved with HCl. The following are other options for a sampler and laboratory where the presence of chlorine is not known:
    - 1. Test for residual chlorine in the field prior to sampling.
        - If no chlorine is present, the samples are to be preserved using HCl as usual.
        - If chlorine is present, add either ascorbic acid or sodium thiosulfate prior to adding HCl.
    - 2. Use VOA vials pre-preserved with sodium thiosulfate or ascorbic acid and add HCl after filling the VOA vial with the sample.
- **FOR WATER SAMPLES TESTED FOR CYANIDE – for NPDES samples by Standard Methods or EPA 335**
    - In the Field: Samples are to be tested for Sulfide using lead acetate paper prior to the addition of Sodium Hydroxide (NaOH). If sulfide is present, the sample must be treated with Cadmium Chloride and filtered prior to the addition of NaOH.

        - If the sulfide test and treatment is not performed in the field, the lab will test the samples for sulfide using lead acetate paper at the time of receipt and if sulfide is present in the sample, the client will be notified and given the option of retaking the sample and treating in the field per the method requirements or the laboratory can analyze the samples as delivered and qualify the results in the final report.

    - It is the responsibility of the client to notify the laboratory if thiosulfate, sulfite, or thiocyanate are known or suspected to be present in the sample. This notification may be on the chain of custody. The samples may need to be subcontracted to a laboratory that performs a UV digestion. If the lab does not perform the UV digestion on samples that contain these compounds, the results must be qualified in the final report.

    - The laboratory must test the sample for oxidizing agents (e.g. Chlorine) prior to analysis and treat according to the methods prior to distillation. (ascorbic acid or sodium arsenite are the preferred choice).

5) Sample Holding Times
    - TestAmerica will make every effort to analyze samples within the regulatory holding time. Samples must be received in the laboratory with enough time to perform the sample analysis. Except for short holding time samples (< 48hr HT) sample must be received with at least 48 hrs (2 working days) remaining on the holding time to ensure analysis.

    - Analyses that are designated as “field” analyses (Odor, pH, Dissolved Oxygen, Disinfectant Residual; a.k.a. Residual Chlorine, and Redox Potential) should be analyzed ASAP by the field sampler prior to delivering to the lab (within 15 minutes). However, if the analyses are to be performed in the laboratory, TestAmerica will make every effort to analyze the samples within 24 hours from receipt of the samples in the testing laboratory. Samples for “field” analyses received after 4:00 pm on Friday or on the weekend will be analyzed no later than the next business day after receipt (Monday unless a holiday). Samples will remain refrigerated and sealed until the time of analysis.

6) All samples submitted for Volatile Organic analyses must have a Trip Blank submitted at the same time. TestAmerica will supply this blank with the bottle order.

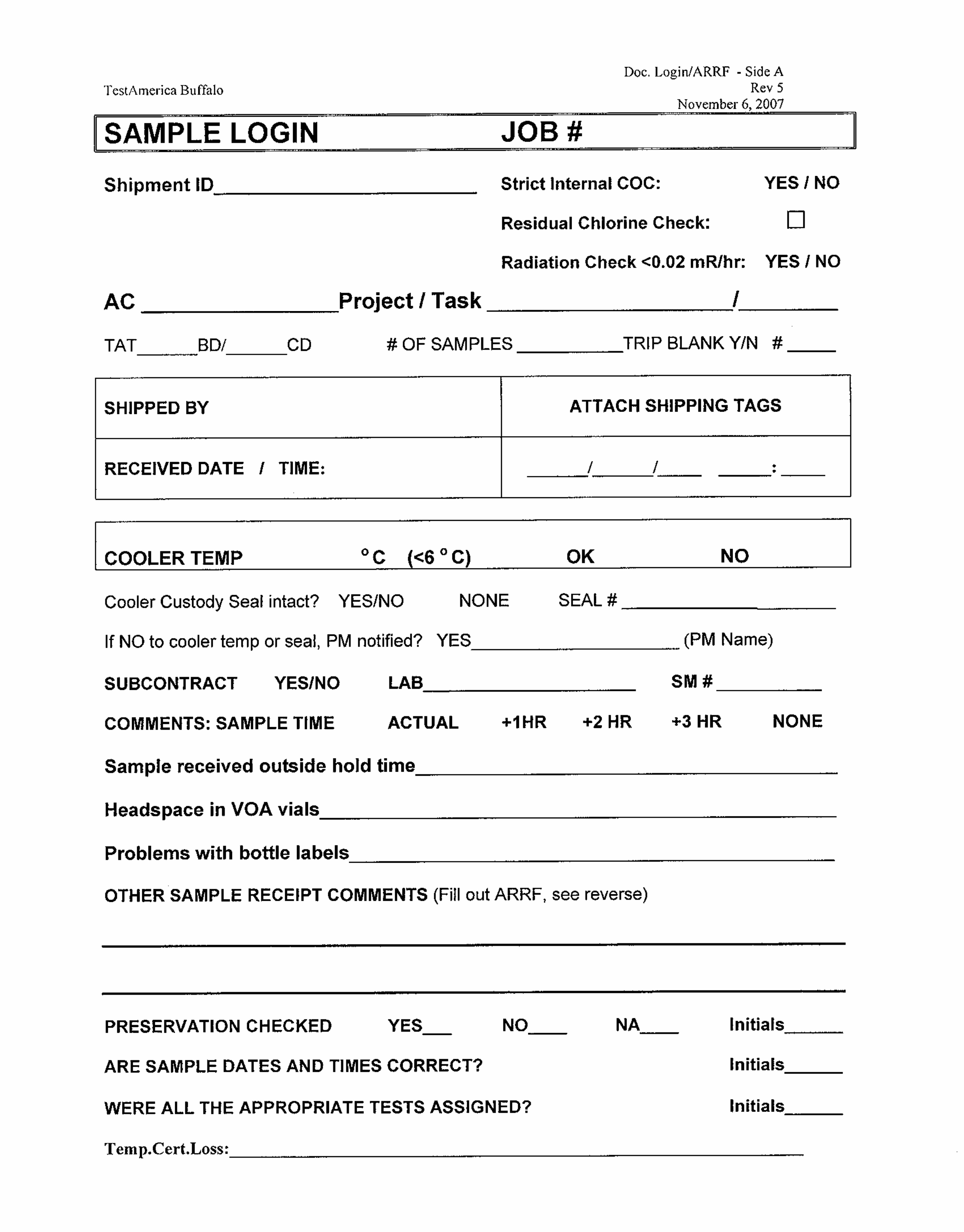
7) The project manager will be notified if any sample is received in damaged condition. TestAmerica will request that a sample be resubmitted for analysis.

8) Recommendations for packing samples for shipment.

   - Pack samples in Ice rather than “Blue” ice packs.
   - Soil samples should be placed in plastic zip-lock bags. The containers often have dirt around the top and do not seal very well and are prone to intrusion from the water from melted ice.
   - Water samples would be best if wrapped with bubble-wrap or paper (newspaper, or paper towels work) and then placed in plastic zip-lock bags.
   - Fill extra cooler space with bubble wrap.

Figure 23-3.

Example: Cooler Receipt Form



TestAmerica Buffalo

Doc. Login/ARRF - Side A
Rev 5
November 6, 2007

SAMPLE LOGIN JOB

Shipment ID______________________ **Strict Internal COC:** **YES / NO**

Residual Chlorine Check: ☐

Radiation Check <0.02 mR/hr: **YES / NO**

AC ________________**Project / Task** ____________________/________

TAT______BD/______CD # OF SAMPLES __________TRIP BLANK Y/N # _____

SHIPPED BY	**ATTACH SHIPPING TAGS**
RECEIVED DATE / TIME:	______/______/____ _____: _____

COOLER TEMP °C (<6 °C) OK NO

Cooler Custody Seal intact? YES/NO NONE SEAL # _____________________

If NO to cooler temp or seal, PM notified? YES____________________ (PM Name)

SUBCONTRACT YES/NO LAB____________________ **SM #** __________

COMMENTS: SAMPLE TIME ACTUAL +1HR +2 HR +3 HR NONE

Sample received outside hold time______________________________________

Headspace in VOA vials____

Problems with bottle labels____

OTHER SAMPLE RECEIPT COMMENTS (Fill out ARRF, see reverse)

PRESERVATION CHECKED YES___ NO___ NA___ Initials_____

ARE SAMPLE DATES AND TIMES CORRECT? Initials_____

WERE ALL THE APPROPRIATE TESTS ASSIGNED? Initials_____

Temp.Cert.Loss:____

SECTION 24.0

ASSURING THE QUALITY OF TEST RESULTS
(NELAC 5.5.9)

24.1 OVERVIEW

In order to assure our clients of the validity of their data, the laboratory continuously evaluates the quality of the analytical process. The analytical process is controlled not only by instrument calibration as discussed in Section 20, but also by routine process quality control measurements (e.g. Blanks, Laboratory Control Samples (LCS), Matrix Spikes (MS), duplicates (DUP), surrogates, Internal Standards (IS)). These quality control checks are performed as required by the method or regulations to assess precision and accuracy. In addition to the routine process quality control samples, Proficiency Testing (PT) Samples (concentrations unknown to laboratory) are analyzed to help ensure laboratory performance.

24.2 CONTROLS

Sample preparation or pre-treatment is commonly required before analysis. Typical preparation steps include homogenization, grinding, solvent extraction, sonication, acid digestion, distillation, reflux, evaporation, drying and ashing. During these pre-treatment steps, samples are arranged into discreet manageable groups referred to as preparation (prep) batches. Prep batches provide a means to control variability in sample treatment. Control samples are added to each prep batch to monitor method performance and are processed through the entire analytical procedure with investigative/field samples.

24.3 NEGATIVE CONTROLS

Table 24-1.

Control Type	Details
Method Blank (MB)	are used to assess preparation and analysis for possible contamination during the preparation and processing steps.
	The specific frequency of use for method blanks during the analytical sequence is defined in the specific standard operating procedure for each analysis. Generally it is 1 for each batch of samples; not to exceed 20 environmental samples.
	The method blank is prepared from a clean matrix similar to that of the associated samples that is free from target analytes (e.g., Reagent water, Ottawa sand, glass beads, etc.) and is processed along with and under the same conditions as the associated samples. The method blank goes through all of the steps of the process (including as necessary: filtration, clean-ups, etc.).
Calibration Blanks	are prepared and analyzed along with calibration standards where applicable. They are prepared using the same reagents that are used to prepare the standards. In some analyses the calibration blank may be included in the calibration curve.
Instrument Blanks	are blank reagents or reagent water that may be processed during an analytical sequence in order to assess contamination in the analytical system. In general, instrument blanks are used to differentiate between contamination caused by the analytical system and that caused by the sample handling or sample prep process. Instrument blanks may also be inserted throughout the analytical sequence to minimize the effect of carryover from samples with high analyte content.

Table 24-1.

Control Type	Details
Trip Blank [1]	are required to be submitted by the client with each shipment of samples requiring aqueous and solid volatiles analyses. Additionally, trip blanks may be prepared and analyzed for volatile analysis of air samples, when required by the client. A trip blank may be purchased (certified clean) or is prepared by the laboratory by filling a clean container with pure deionized water that has been purged to remove any volatile compounds. Appropriate preservatives are also added to the container. The trip blank is sent with the bottle order and is intended to reflect the environment that the containers are subjected to throughout shipping and handling and help identify possible sources if contamination is found. The field sampler returns the trip blank in the cooler with the field samples.
Field Blanks [1]	are sometimes used for specific projects by the field samplers. A field blank prepared in the field by filling a clean container with pure reagent water and appropriate preservative, if any, for the specific sampling activity being undertaken. (EPA OSWER)
Equipment Blanks [1]	are also sometimes created in the field for specific projects. An equipment blank is a sample of analyte-free media which has been used to rinse common sampling equipment to check effectiveness of decontamination procedures. (NELAC)
Holding Blanks	also referred to as refrigerator or freezer blanks, are used to monitor the sample storage units for volatile organic compounds during the storage of VOA samples in the laboratory

[1] When known, these field QC samples should not be selected for matrix QC as it does not provide information on the behavior of the target compounds in the field samples. Usually, the client sample ID will provide information to identify the field blanks with labels such as "FB", "EB", or "TB."

24.4 POSITIVE CONTROLS

Control samples (e.g., QC indicators) are analyzed with each batch of samples to evaluate data based upon (1) Method Performance (Laboratory Control Sample (LCS) or Blank Spike (BS)), which entails both the preparation and measurement steps; and (2) Matrix Effects (Matrix Spike (MS) (Matrix spikes are not applicable to air) or Sample Duplicate (MD, DUP), which evaluates field sampling accuracy, precision, representativeness, interferences, and the effect of the matrix on the method performed. Each regulatory program and each method within those programs specify the control samples that are prepared and/or analyzed with a specific batch

Note that frequency of control samples vary with specific regulatory, methodology and project specific criteria. Complete details on method control samples are as listed in each analytical SOP.

24.4.1 Method Performance Control - Laboratory Control Sample (LCS)

24.4.1.1 The LCS measures the accuracy of the method in a blank matrix and assesses method performance independent of potential field sample matrix affects in a laboratory batch.

24.4.1.2 The LCS is prepared from a clean matrix similar to that of the associated samples that is free from target analytes (for example: Reagent water, Ottawa sand, glass beads, etc.) and is processed along with and under the same conditions as the associated samples. The LCS is spiked with verified known amounts of analytes or is made of a material containing known and verified amounts of analytes, taken through all preparation and analysis steps along with the field samples. Where there is no preparation taken for an analysis (such as in aqueous volatiles), or when all samples and standards undergo the same preparation and analysis process (such as Phosphorus), a calibration verification standard may be reported as the LCS. In some instances where there is no practical clean solid matrix available, aqueous LCS's may be processed for solid matrices; final results may be calculated as mg/kg or ug/kg, assuming 100% solids and a weight equivalent to the aliquot used for the corresponding field samples, to facilitate comparison with the field samples.

24.4.1.3 Certified pre-made reference material purchased from a NIST/A2LA accredited vendor may also be used for the LCS when the material represents the sample matrix or the analyte is not easily spiked (e.g. solid matrix LCS for metals, TDS, etc.).

24.4.1.4 The specific frequency of use for LCS during the analytical sequence is defined in the specific standard operating procedure for each analysis. It is generally 1 for each batch of samples; not to exceed 20 environmental samples.

24.4.1.5 If the mandated or requested test method, or project requirements, do not specify the spiking components, the laboratory shall spike all reportable components to be reported in the Laboratory Control Sample (and Matrix Spike) where applicable (e.g. no spike of pH). However, in cases where the components interfere with accurate assessment (such as simultaneously spiking chlordane, toxaphene and PCBs in Method 608), the test method has an extremely long list of components or components are incompatible, at a minimum, a representative number of the listed components (see below) shall be used to control the test method. The selected components of each spiking mix shall represent all chemistries, elution patterns and masses, permit specified analytes and other client requested components. However, the laboratory shall ensure that all reported components are used in the spike mixture within a two-year time period.

24.4.1.5.1 For methods that have 1-10 target analytes, spike all components.

24.4.1.5.2 For methods that include 11-20 target analytes, spike at least 10 or 80%, whichever is greater.

24.4.1.5.3 For methods with more than 20 target analytes, spike at least 16 components.

24.4.1.5.4 Exception: Due to analyte incompatibility in pesticides, Toxaphene and Chlordane are only spiked at client request based on specific project needs.

24.4.1.5.5 Exception: Due to analyte incompatibility between the various PCB aroclors, aroclors 1016 and 1260 are used for spiking as they cover the range of all of the aroclors. Specific aroclors may be used by request on a project specific basis.

24.5 SAMPLE MATRIX CONTROLS

Table 24-5. Sample Matrix Control

Control Type	Details	
Matrix Spikes (MS)	Use	used to assess the effect sample matrix of the spiked sample has on the precision and accuracy of the results generated by the method used;
	Typical Frequency [1]	At a minimum, with each matrix-specific batch of samples processed, an MS is carried through the complete analytical procedure. Unless specified by the client, samples used for spiking are randomly selected and rotated between different client projects. If the mandated or requested test method does not specify the spiking components, the laboratory shall spike all reportable components to be reported in the Laboratory Control Sample and Matrix Spike. Refer to the method SOP for complete details
	Description	Essentially a sample fortified with a known amount of the test analyte(s).
Surrogate	Use	Measures method performance to sample matrix (organics only).
	Typical Frequency [1]	Are added to all samples, standards, and blanks, for all organic chromatography methods except when the matrix precludes its use or when a surrogate is not available. The recovery of the surrogates is compared to the acceptance limits for the specific method. Poor surrogate recovery may indicate a problem with sample composition and shall be reported, with data qualifiers, to the client whose sample produced poor recovery.
	Description	Are similar to matrix spikes except the analytes are compounds with properties that mimic the analyte of interest and are unlikely to be found in environment samples.
Duplicates[2]	Use	For a measure of analytical precision, with each matrix-specific batch of samples processed, a matrix duplicate (MD or DUP) sample, matrix spike duplicate (MSD), or LCS duplicate (LCSD) is carried through the complete analytical procedure.
	Typical Frequency [1]	Duplicate samples are usually analyzed with methods that do not require matrix spike analysis.
	Description	Performed by analyzing two aliquots of the same field sample independently or an additional LCS.
Internal Standards	Use	Are spiked into all environmental and quality control samples (including the initial calibration standards) to monitor the qualitative aspect of organic and some inorganic analytical measurements.
	Typical Frequency [1]	All organic and ICP methods as required by the analytical method.
	Description	Used to correct for matrix effects and to help troubleshoot variability in analytical response and are assessed after data acquisition. Possible sources of poor internal standard response are sample matrix, poor analytical technique or instrument performance.

[1] See the specific analytical SOP for type and frequency of sample matrix control samples.

[2] LCSD's are normally not performed except when regulatory agencies or client specifications require them. The recoveries for the spiked duplicate samples must meet the same laboratory established recovery limits as the accuracy QC samples. If an LCSD is analyzed both the LCS and LCSD must meet the same recovery criteria and be included in the final report. The precision measurement is reported as "Relative Percent Difference" (RPD). Poor precision between duplicates (except LCS/LCSD) may indicate non-homogeneous matrix or sampling.

24.6 ACCEPTANCE CRITERIA (CONTROL LIMITS)

24.6.1 As mandated by the test method and regulation, each individual analyte in the LCS, MS, or Surrogate Spike is evaluated against the control limits published in the test method. Where there are no established acceptance criteria, the laboratory calculates in-house control limits with the use of control charts or, in some cases, utilizes client project specific control limits. When this occurs, the regulatory or project limits will supersede the laboratory's in-house limits.

Note: For methods, analytes and matrices with very limited data (e.g., unusual matrices not analyzed often), interim limits are established using available data or by analogy to similar methods or matrices.

24.6.2 Once control limits have been established, they are verified, reviewed, and updated if necessary on an annual basis unless the method requires more frequent updating. Control limits are established per method (as opposed to per instrument) regardless of the number of instruments utilized.

24.6.3 Laboratory generated % Recovery acceptance (control) limits are generally established by taking ± 3 Standard Deviations (99% confidence level) from the average recovery of a minimum of 20-30 data points (more points are preferred).

24.6.3.1 Regardless of the calculated limit, the limit should be no tighter than the Calibration Verification (ICV/CCV). (Unless the analytical method specifies a tighter limit).

24.6.3.2 In-house limits cannot be any wider than those mandated in a regulated analytical method. Client or contract required control limits are evaluated against the laboratory's statistically derived control limits to determine if the data quality objectives (DQOs) can be achieved. If laboratory control limits are not consistent with DQOs, then alternatives must be considered, such as method improvements or use of an alternate analytical method.

24.6.3.3 The lowest acceptable recovery limit will be 10% (the analyte must be detectable). Exception: The lowest acceptable recovery limit for Benzidine will be 5% and the analyte must be detectable.

24.6.3.4 The maximum acceptable recovery limit will be 150%.

24.6.3.5 The maximum acceptable RPD limit will be 35% for waters and 40% for soils. The minimum RPD limit is 10%.

24.6.3.6 If either the high or low end of the control limit changes by ≤ 5% from previous, the data points are inspected and, using professional judgment, the limits may be left unchanged if there is no affect on laboratory ability to meet the existing limits.

24.6.4 The lab must be able to generate a current listing of their control limits and track when the updates are performed. In addition, the laboratory must be able to recreate historical control limits.

24.6.4.1 The control limits are maintained in the laboratory LIMs system. The limits for each analyte/method/matrix combination are assigned effective and expiration dates. The QA department is able to query the LIMs system and print an active list of control limits based on this database. The most current laboratory limits (based on the effective/expiration dates) are reflected on the laboratory worksheets and final reports unless superseded by project specific limits.

24.6.5 A LCS that is within the acceptance criteria establishes that the analytical system is in control and is used to validate the process. Samples that are analyzed with an LCS with recoveries outside of the acceptance limits may be determined as out of control and should be reanalyzed if possible. If reanalysis is not possible, then the results for all affected analytes for samples within the same batch must be qualified when reported. The internal corrective action process (see Section 13) is also initiated if an LCS exceeds the acceptance limits. Sample results may be qualified and reported without reanalysis if:

24.6.5.1 The analyte results are below the reporting limit and the LCS is above the upper control limit.

24.6.5.2 If the analytical results are above the relevant regulatory limit and the LCS is below the lower control limit.

24.6.5.3 Or, for NELAC and Department of Defense (DOD) work, there are an allowable number of Marginal Exceedances (ME). This information may be found in the laboratory specific SOP BF-QA-005, “Preventative and Corrective Action”.

- <11 analytes – 0 marginal exceedances are allowed.
- 11 – 30 Analytes – 1 marginal exceedance is allowed
- 31-50 Analytes – 2 marginal exceedances are allowed
- 51-70 Analytes – 3 marginal exceedances are allowed
- 71-90 Analytes – 4 marginal exceedances are allowed
- > 90 Analytes – 5 marginal exceedances are allowed

24.6.5.3.1 Marginal exceedances are recovery exceedances between 3 SD and 4 SD from the mean recovery limit (NELAC).

24.6.5.3.2 Marginal exceedances must be random. If the same analyte exceeds the LCS control limit repeatedly, it is an indication of a systematic problem. The source of the error must be located and corrective action taken.

24.6.5.3.3 Though marginal excedences may be allowed, the data must still be qualified to indicate it is outside of the normal limits.

24.6.6 If the MS/MSDs do not meet acceptance limits, the MS/MSD and the associated spiked sample is reported with a qualifier for those analytes that do not meet limits. If obvious preparation errors are suspected, or if requested by the client, unacceptable MS/MSDs are reprocessed and reanalyzed to prove matrix interference. A more detailed discussion of acceptance criteria and corrective action can be found in the lab’s method SOPs and in Section 12.

24.6.7 If a surrogate standard falls outside the acceptance limits, if there is not obvious chromatographic matrix interference, reanalyze the sample to confirm a possible matrix effect. If the recoveries confirm or there was obvious chromatographic interference, results are reported from the original analysis and a qualifier is added. If the reanalysis meets surrogate recovery criteria, the second run is reported (or both are reported if requested by the client). Under certain circumstances, where all of the samples are from the same location and share similar chromatography, the reanalysis may be performed on a single sample rather than all of the samples and if the surrogate meets the recovery criteria in the reanalysis, all of the affected samples would require reanalysis.

24.7 ADDITIONAL PROCEDURES TO ASSURE QUALITY CONTROL

24.7.1 The laboratory has written and approved method SOPs to assure the accuracy of the test method including calibration (see Section 20), use of certified reference materials (see Section 21) and use of PT samples.

24.7.2 A discussion regarding MDLs, Limit of Detection (LOD) and Limit of Quantitation (LOQ) can be found in Section 19.

24.7.3 Use of formulae to reduce data is discussed in the method SOPs and in Section 20.

24.7.4 Selection of appropriate reagents and standards is included in Section 9 and 22.

24.7.5 A discussion on selectivity of the test is included in Section 5.

24.7.6 Constant and consistent test conditions are discussed in Section 19.

24.7.7 The laboratories sample acceptance policy is included in Section 23.

SECTION 25.0

REPORTING RESULTS
(NELAC 5.5.10)

25.1 OVERVIEW

The results of each test are reported accurately, clearly, unambiguously, and objectively in accordance with State and Federal regulations as well as client requirements. A variety of report formats are available to meet specific needs. Analytical results are issued in a format that is intended to satisfy customer and laboratory accreditation requirements as well as provide the end user with the information needed to properly evaluate the results. Where there is conflict between client requests and laboratory ethics or regulatory requirements, the laboratory's ethical and legal requirements are paramount, and the laboratory will work with the client during project set up to develop an acceptable solution. Refer to Section 7.

In cases where a client asks for simplified reports, there must be a written request from the client. There still must be enough information that would show any analyses that were out of conformance (QC out of limits) and there should be a reference to a full report that is made available to the client.

Review of reported data is included in Section 19.

25.2 TEST REPORTS

Analytical results are reported in a format that is satisfactory to the client and meets all requirements of applicable accrediting authorities and agencies. A variety of report formats are available to meet specific needs. The report is printed on laboratory letterhead, reviewed, and signed by the appropriate project manager. At a minimum, the standard laboratory report shall contain the following information:

25.2.1 A report title (e.g. Analytical Report) with a “sample results” column header.

25.2.2 Each report cover page is printed on company letterhead which includes the laboratory name, address and telephone number.

25.2.3 A unique identification of the report (e.g. job number) and on each page an identification in order to ensure the page is recognized as part of the report and a clear identification of the end.

Note: Page numbers of report are represented as # / ##. Where the first number is the page number and the second is the total number of pages.

25.2.4 A copy of the chain of custody (COC).

- Any COCs involved with Subcontracting are included.

- In most cases, the applicable COC is paginated and is an integral part of the report.

- Any additional addenda to the report must be treated in a similar fashion so it is a recognizable part of the report and cannot accidentally get separated from the report (e.g. Sampling information).

25.2.5 The name and address of client and a project name/number, if applicable.

25.2.6 Client project manager or other contact

25.2.7 Description and unambiguous identification of the tested sample(s) including the client identification code.

25.2.8 Date of receipt of sample, date and time of collection, and date(s) of test preparation and performance, and time of preparation or analysis if the required holding time for either activity is less than or equal to 72 hours.

25.2.9 Date reported or date of revision, if applicable.

25.2.10 Method of analysis including method code (EPA, Standard Methods, etc).

25.2.11 Practical quantitation limits or client reporting limit.

25.2.12 Method detection limits (if requested)

25.2.13 Definition of Data qualifiers and reporting acronyms (e.g. ND).

25.2.14 Sample results.

25.2.15 QC data consisting of method blank, surrogate, LCS, and MS/MSD recoveries and control limits (if requested).

25.2.16 Condition of samples at receipt including temperature. This may be accomplished in a narrative or by attaching sample login sheets (Refer to Sec. 25.2.4 – Item 3 regarding additional addenda). Sample temperatures are recorded in the report case narrative and on the CoC. Deviations from normal conditions (e.g., preservation, breakage) are recorded in the report case narrative.

25.2.17 A statement to the effect that the results relate only to the items tested and the sample as received by the laboratory.

25.2.18 A statement that the report shall not be reproduced except in full, without prior express written approval by the laboratory coordinator.

25.2.19 A signature and title of the person(s) accepting responsibility for the content of the report and date of issue. Signatories are appointed by the Lab Director.

25.2.20 When NELAC accreditation is required, the lab shall certify that the test results meet all requirements of NELAC or provide reasons and/or justification if they do not.

25.2.21 The laboratory includes a cover letter.

25.2.22 Where applicable, a narrative to the report that explains the issue(s) and corrective action(s) taken in the event that a specific accreditation or certification requirement was not met.

25.2.23 When Soil samples are analyzed, a specific identification as to whether soils are reported on a “wet weight” or “dry weight” basis.

25.2.24 Appropriate laboratory certification number for the state of origin of the sample if applicable.

25.2.25 If only part of the report is provided to the client (client requests some results before all of it is complete), it must be clearly indicated on the report (e.g, partial report). A complete report must be sent once all of the work has been completed.

25.2.26 Any non-TestAmerica subcontracted analysis results are provided as an addendum to the report on the official letterhead of the subcontractor. All TestAmerica subcontracting is clearly identified on the report as to which laboratory performed a specific analysis.

25.3 REPORTING LEVEL OR REPORT TYPE

TestAmerica Buffalo offers four levels of quality control reporting. Each level, in addition to its own specific requirements, contains all the information provided in the preceding level. The packages provide the following information in addition to the information described above:

- Level I is a report with the features described in Section 25.2 above.
- Level II is a Level I report plus summary information, including results for the method blank, percent recovery for laboratory control samples and matrix spike samples, and the RPD values for all MSD and sample duplicate analyses.
- Level III contains all the information supplied in Level II, but presented on CLP-like summary forms, and relevant calibration information. A Level II report is not included, unless specifically requested. No raw data is provided.
- Level IV is the same as Level III with the addition of all raw supporting data.

In addition to the various levels of QC packaging, the laboratory also provides reports in diskette deliverable form. Initial reports may be provided to clients by facsimile. All faxed reports are followed by hardcopy. Procedures used to ensure client confidentiality are outlined in Section 26.7.

25.3.1 Electronic Data Deliverables (EDDs)

EDDs are routinely offered as part of TestAmerica's services. ***TestAmerica Buffalo*** offers a variety of EDD formats including Environmental Restoration Information Management System (ERPIMS), Excel, Dbase, GISKEY, and Text Files.

EDD specifications are submitted to the IT department by the PM for review and undergo the contract review process. Once the facility has committed to providing data in a specific electronic format, the coding of the format may need to be performed. This coding is documented and validated. The validation of the code is retained by the IT staff coding the EDD.

EDDs shall be subject to a review to ensure their accuracy and completeness. If EDD generation is automated, review may be reduced to periodic screening if the laboratory can demonstrate that it can routinely generate that EDD without errors. Any revisions to the EDD format must be reviewed until it is demonstrated that it can routinely be generated without errors. If the EDD can be reproduced accurately and if all subsequent EDDs can be produced error-free, each EDD does not necessarily require a review.

25.4 SUPPLEMENTAL INFORMATION FOR TEST

The lab identifies any unacceptable QC analyses or any other unusual circumstances or observations such as environmental conditions and any non-standard conditions that may have affected the quality of a result. This is typically in the form of a footnote or a qualifier and/or a narrative explaining the discrepancy in the front of the report

25.4.1 Numeric results with values outside of the calibration range, either high or low are qualified as 'estimated'.

25.4.2 Where quality system requirements are not met, a statement of compliance/non-compliance with requirements and/or specifications is required, including identification of test results derived from any sample that did not meet NELAC sample acceptance requirements such as improper container, holding time, or temperature.

25.4.3 Where applicable, a statement on the estimated uncertainty of measurements; information on uncertainty is needed when a client's instructions so require.

25.4.4 Opinions and Interpretations - The test report contains objective information, and generally does not contain subjective information such as opinions and interpretations. If such information is required by the client, the Laboratory Director will determine if a response can be prepared. If so, the Laboratory Director will designate the appropriate member of the management team to prepare a response. The response will be fully documented, and reviewed by the Laboratory Director, before release to the client. There may be additional fees charged to the client at this time, as this is a non-routine function of the laboratory.

Note: Review of data deliverable packages for submittal to regulatory authorities requires responses to non-conforming data concerning potential impact on data quality. This necessitates a limited scope of interpretation, and this work is performed by the QA Department. This is the only form of "interpretation" of data that is routinely performed by the laboratory.

When opinions or interpretations are included in the report, the laboratory provides an explanation as to the basis upon which the opinions and interpretations have been made. Opinions and interpretations are clearly noted as such and where applicable, a comment should be added suggesting that the client verify the opinion or interpretation with their regulator.

25.5 ENVIRONMENTAL TESTING OBTAINED FROM SUBCONTRACTORS

If the laboratory is not able to provide the client the requested analysis, the samples would be subcontracted following the procedures outlined in Section 8.

Data reported from analyses performed by a subcontractor laboratory are clearly identified as such on the analytical report provided to the client. Results from a subcontract laboratory outside of TestAmerica are reported to the client on the subcontract laboratory's original report stationary and the report includes any accompanying documentation.

25.6 CLIENT CONFIDENTIALITY

In situations involving the transmission of environmental test results by telephone, facsimile or other electronic means, client confidentiality must be maintained.

TestAmerica will not intentionally divulge to any person (other than the Client or any other person designated by the Client in writing) any information regarding the services provided by TestAmerica or any information disclosed to TestAmerica by the Client. Furthermore, information known to be potentially endangering to national security or an entity's proprietary rights will not be released.

Note: This shall not apply to the extent that the information is required to be disclosed by TestAmerica under the compulsion of legal process. TestAmerica will, to the extent feasible, provide reasonable notice to the client before disclosing the information.

Note: Authorized representatives of an accrediting authority are permitted to make copies of any analyses or records relevant to the accreditation process, and copies may be removed from the laboratory for purposes of assessment.

25.6.1 Report deliverable formats are discussed with each new client. If a client requests that reports be faxed or e-mailed, the reports are faxed with a cover sheet or e-mailed with the following note that includes a confidentiality statement similar to the following:

This material is intended only for the use of the individual(s) or entity to whom it is addressed, and may contain information that is privileged and confidential. It is our policy that facsimiles are intended for and should be used for business purposes only. If you are not the intended recipient, or the employee or agent responsible for delivering this material to the intended recipient, you are hereby notified that any dissemination, distribution or copying of this communication is strictly prohibited. If you have received this communication in error, please notify the sender.

25.7 FORMAT OF REPORTS

The format of reports is designed to accommodate each type of environmental test carried out and to minimize the possibility of misunderstanding or misuse.

25.8 AMENDMENTS TO TEST REPORTS

Corrections, additions, or deletions to reports are only made when justification arises through supplemental documentation. Justification is documented using the laboratory's corrective action system (refer to Section 12).

The revised report is retained on the Archive data server, as is the original report. The revised report is stored in the Archive data server under the sample number followed by "R". The revised report will have the word "revised" appended to the cover letter.

When the report is re-issued, a notation of "revised" is placed on the cover/signature page of the report. A brief explanation of reason for the re-issue is included in the report case narrative.

25.9 POLICIES ON CLIENT REQUESTS FOR AMENDMENTS

25.9.1 Policy on Data Omissions or Reporting Limit Increases

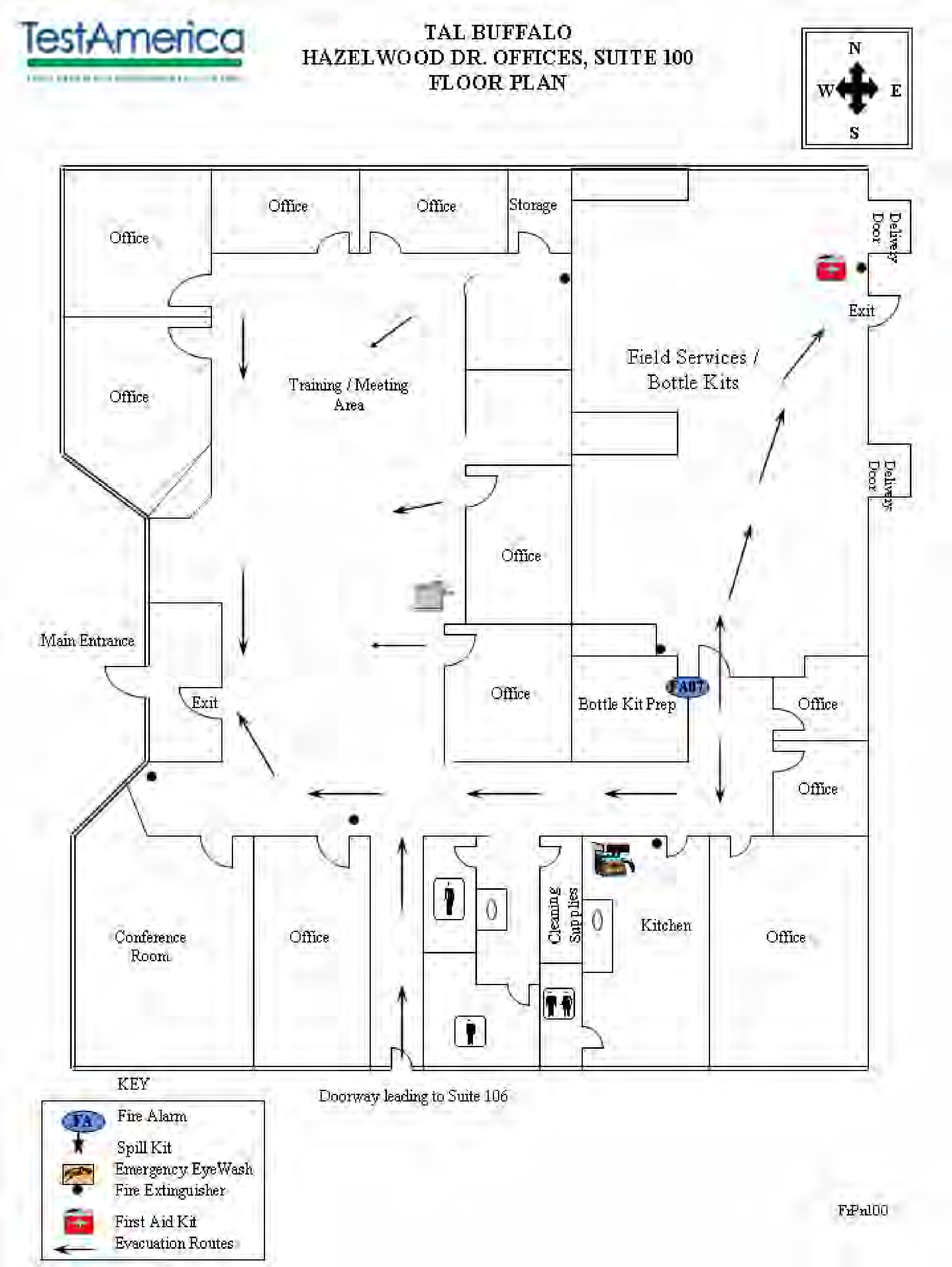
Fundamentally, our policy is simply to not omit previously reported results (including data qualifiers) or to not raise reporting limits and report sample results as ND. This policy has few exceptions. Exceptions are:

- Laboratory error.
- Sample identification is indeterminate (confusion between COC and sample labels).
- An incorrect analysis (not analyte) was requested (e.g., COC lists 8315 but client wanted 8310). A written request for the change is required.
- Incorrect limits reported based on regulatory requirements.
- The requested change has absolutely *no possible* impact on the interpretation of the analytical results and there is *no possibility* of the change being interpreted as misrepresentation by anyone inside or outside of our company.

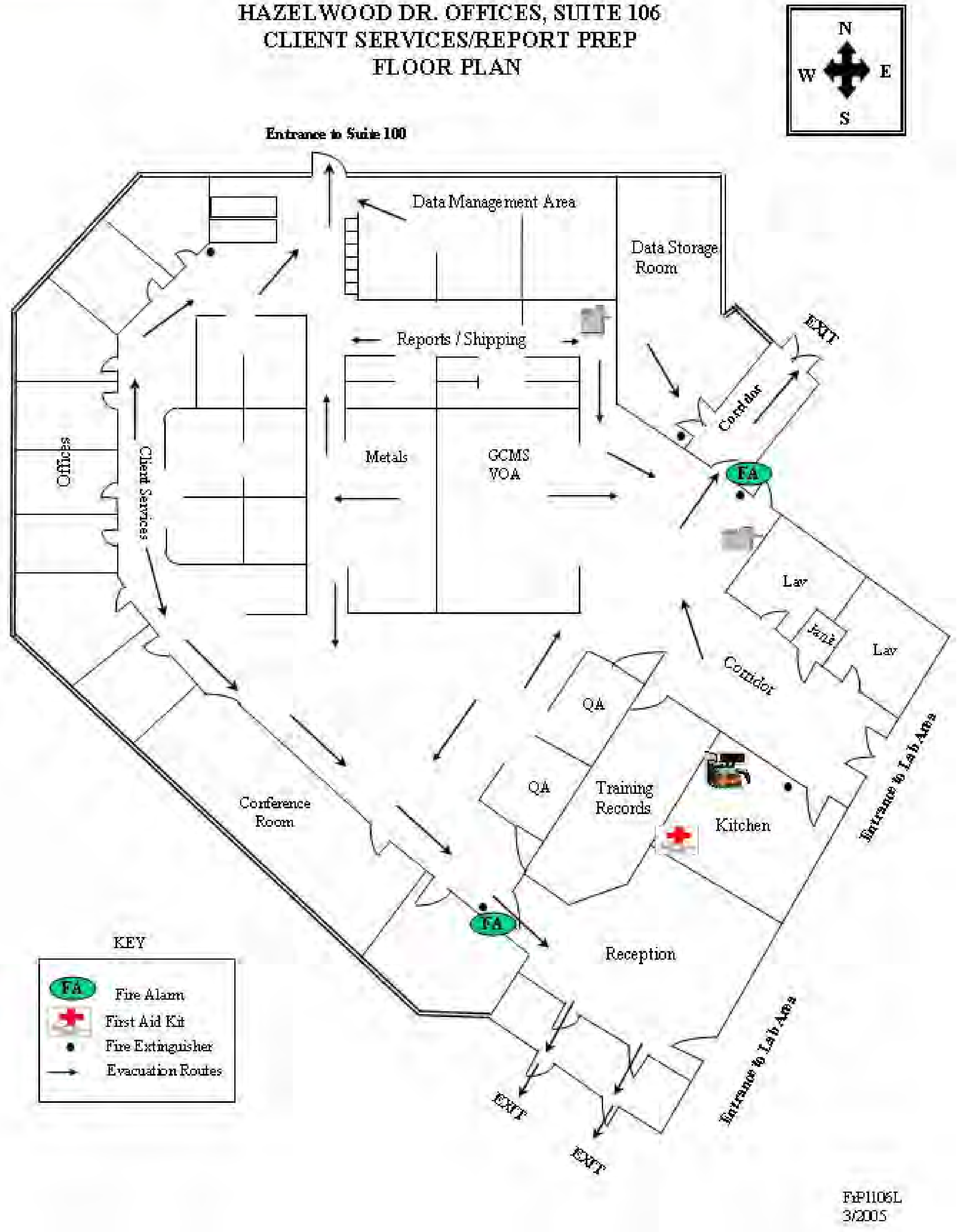
25.9.2 Multiple Reports

TestAmerica does not issue multiple reports for the same workorder where there is different information on each report (this does not refer to copies of the same report) unless required to meet regulatory needs and approved by QA.

Appendix 1.

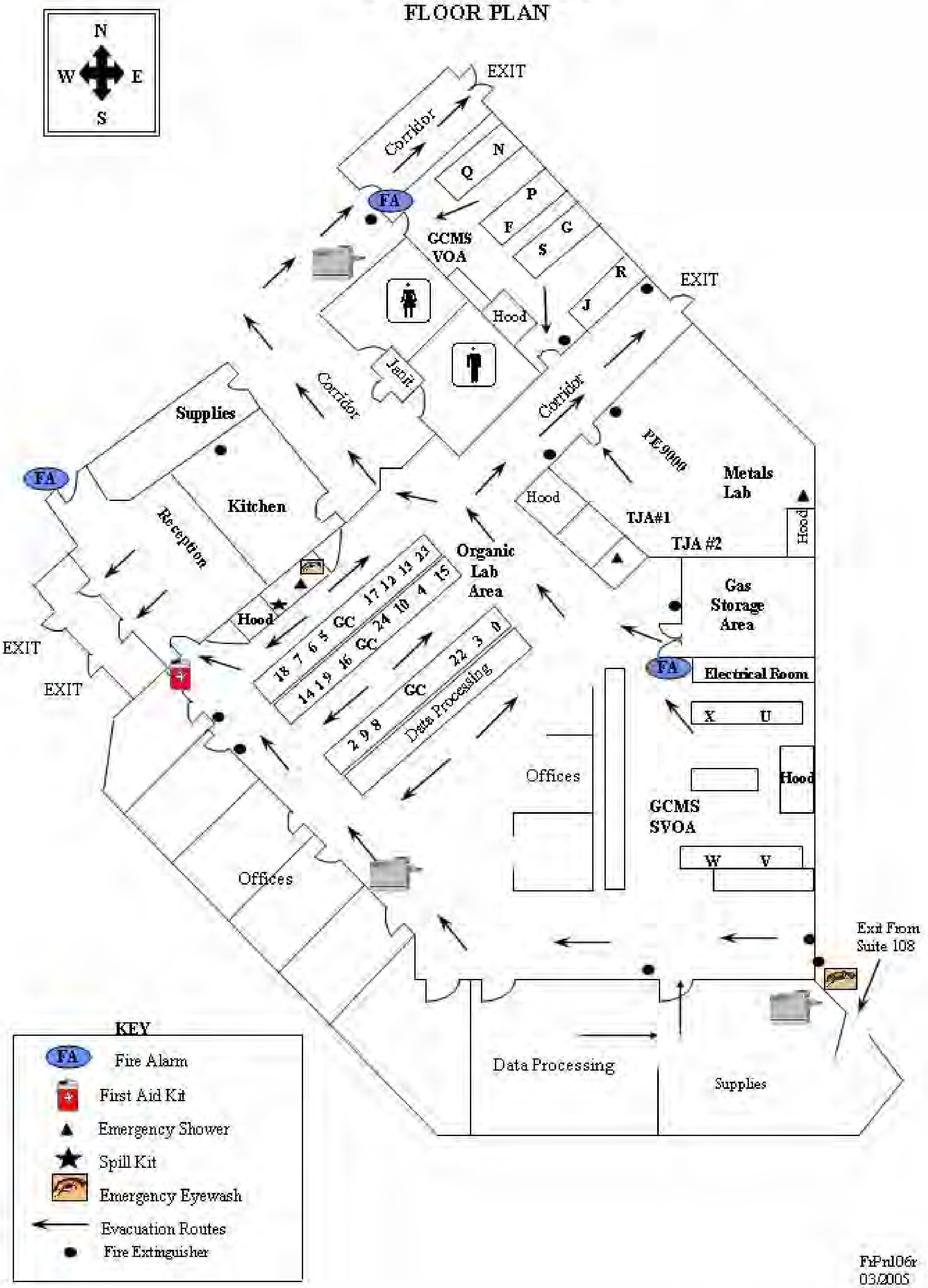

TestAmerica
TAL BUFFALO
HAZELWOOD DR. OFFICES, SUITE 100
FLOOR PLAN
N
W
E
S
Office
Office
Office
Storage
Office
Training / Meeting Area
Field Services / Bottle Kits
Delivery Door
Exit
Delivery Door
Office
Main Entrance
Exit
Office
Bottle Kit Prep
FA07
Office
Office
Conference Room
Office
Cleaning Supplies
Kitchen
Office
Doorway leading to Suite 106
KEY
FA Fire Alarm
Spill Kit
Emergency EyeWash
Fire Extinguisher
First Aid Kit
Evacuation Routes
FrPnl00

TAL BUFFALO
HAZELWOOD DR. OFFICES, SUITE 106
CLIENT SERVICES/REPORT PREP
FLOOR PLAN



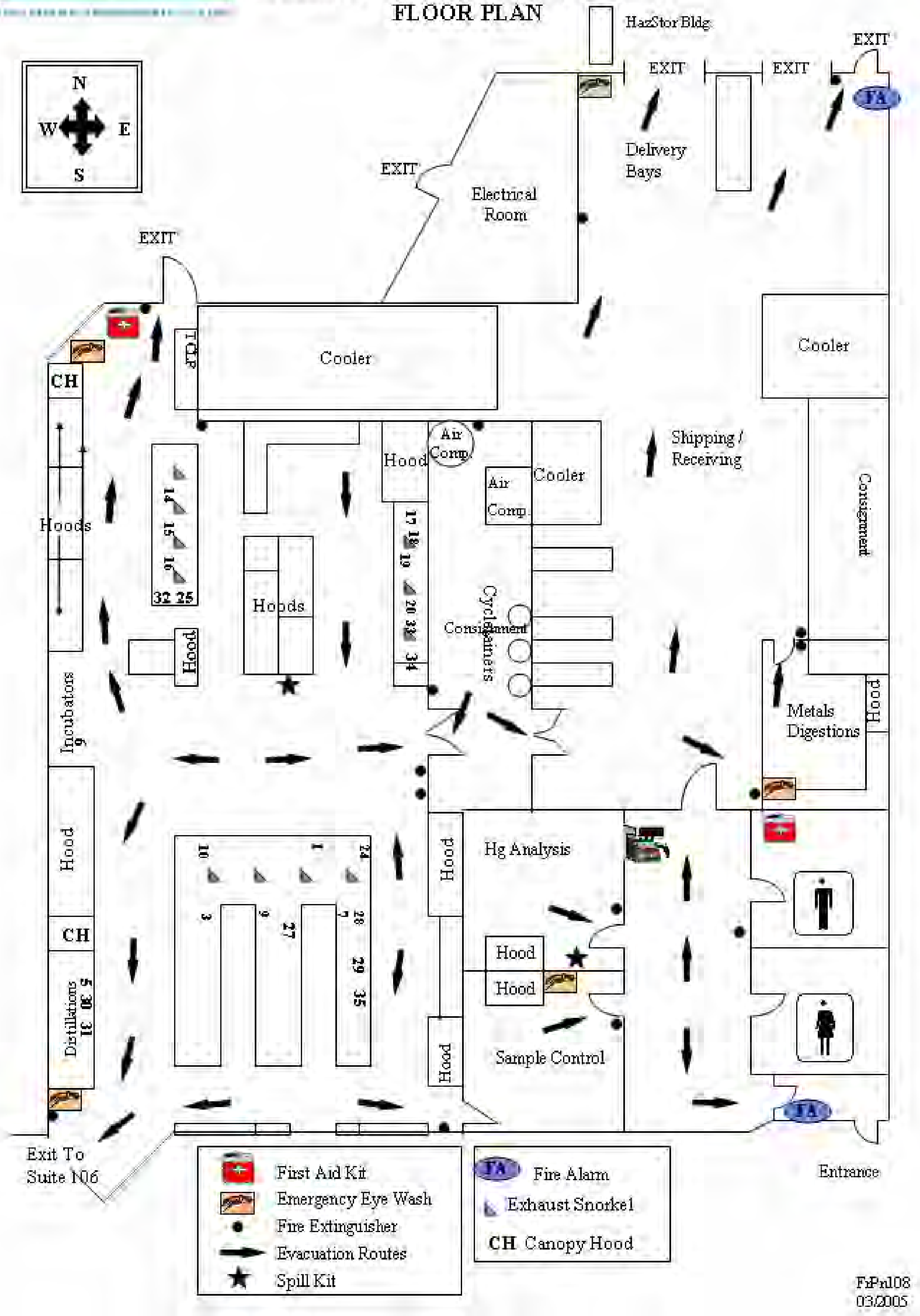
TestAmerica
THE LEADER IN ENVIRONMENTAL TESTING

TAL BUFFALO
HAZELWOOD DR. NY OFFICES, SUITE 106
LABORATORY AREA
FLOOR PLAN



FrPn106r
03/2005

TAL BUFFALO
HAZELWOOD DR. OFFICES, SUITE 108
FLOOR PLAN



Appendix 2. Glossary/Acronyms

Glossary:

Acceptance Criteria:
Specified limits placed on characteristics of an item, process, or service defined in requirement documents. (ASQC)

Accreditation:

The process by which an agency or organization evaluates and recognizes a laboratory as meeting certain predetermined qualifications or standards, thereby accrediting the laboratory. In the context of the National Environmental Laboratory Accreditation Program (NELAP), this process is a voluntary one. (NELAC)

Accrediting Authority:
The Territorial, State, or Federal Agency having responsibility and accountability for environmental laboratory accreditation and which grants accreditation (NELAC) [1.5.2.3]

Accuracy:
The degree of agreement between an observed value and an accepted reference value. Accuracy includes a combination of random error (precision) and systematic error (bias) components which are due to sampling and analytical operations; a data quality indicator. (QAMS)

Analyst:
The designated individual who performs the “hands-on” analytical methods and associated techniques and who is the one responsible for applying required laboratory practices and other pertinent quality controls to meet the required level of quality. (NELAC)

Batch:
Environmental samples which are prepared and/or analyzed together with the same process and personnel, using the same lot(s) of reagents. A preparation batch is composed of one to 20 environmental samples of the same matrix, meeting the above mentioned criteria and with a maximum time between the start of processing of the first and last sample in the batch to be 24 hours. An analytical batch is composed of prepared environmental samples (extracts, digestates or concentrates) and /or those samples not requiring preparation, which are analyzed together as a group using the same calibration curve or factor. An analytical batch can include samples originating from various environmental matrices and can exceed 20 samples. (NELAC Quality Systems Committee)

Blank:
A sample that has not been exposed to the analyzed sample stream in order to monitor contamination during sampling, transport, storage or analysis. The blank is subjected to the usual analytical and measurement process to establish a zero baseline or background value and is sometimes used to adjust or correct routine analytical results. (ASQC)

Blind Sample:

A sample for analysis with a composition known to the submitter. The analyst/laboratory may know the identity of the sample but not its composition. It is used to test the analyst's or laboratory's proficiency in the execution of the measurement process.

Calibration:
To determine, by measurement or comparison with a standard, the correct value of each scale reading on a meter, instrument, or other device. The levels of the applied calibration standard should bracket the range of planned or expected sample measurements. (NELAC)

Calibration Curve:
The graphical relationship between the known values, such as concentrations, of a series of calibration standards and their instrument response. (NELAC)

Calibration Method:
A defined technical procedure for performing a calibration. (NELAC)

Calibration Standard:
A substance or reference material used to calibrate an instrument (QAMS)

Certified Reference Material (CRM):
A reference material one or more of whose property values are certified by a technically valid procedure, accompanied by or traceable to a certificate or other documentation which is issued by a certifying body. (ISO Guide 30–2.2)

Chain of Custody:
An unbroken trail of accountability that ensures the physical security of samples and includes the signatures of all who handle the samples. (NELAC) [5.12.4]

Clean Air Act:
The enabling legislation in 42 U>S>C> 7401 et seq., Public Law 91-604, 84 Stat. 1676 Pub. L. 95-95, 91 Stat., 685 and Pub. L. 95-190, 91 Stat., 1399, as amended, empowering EPA to promulgate air quality standards, monitor and enforce them. (NELAC)

Comprehensive Environmental Response, Compensation and Liability Act (CERCLA/SUPERFUND):
The enabling legislation in 42 U.S.C. 9601-9675 et seq., as amended by the Superfund Amendments and Reauthorization Act of 1986 (SARA), 42 U.S.C. 9601 et seq., to eliminate the health and environmental threats posed by hazardous waste sites. (NELAC)

Compromised Samples:
Those samples which are improperly sampled, insufficiently documented (chain of custody and other sample records and/or labels), improperly preserved, collected in improper containers, or exceeding holding times when delivered to a laboratory. Under normal conditions, compromised samples are not analyzed. If emergency situation require analysis, the results must be appropriately qualified. (NELAC)

Confidential Business Information (CBI):
Information that an organization designates as having the potential of providing a competitor with inappropriate insight into its management, operation or products. NELAC and its

representatives agree to safeguarding identified CBI and to maintain all information identified as such in full confidentiality.

Confirmation:
Verification of the identity of a component through the use of an approach with a different scientific principle from the original method. These may include, but are not limited to:

Second column confirmation
Alternate wavelength
Derivitization
Mass spectral interpretation
Alternative detectors or
Additional Cleanup procedures
(NELAC)

Conformance:
An affirmative indication or judgment that a product or service has met the requirements of the relevant specifications, contract, or regulation; also the state of meeting the requirements. (ANSI/ASQC E4-1994)

Corrective Action:
The action taken to eliminate the causes of an existing nonconformity, defect or other undesirable situation in order to prevent recurrence. (ISO 8402)

Data Audit:
A qualitative and quantitative evaluation of the documentation and procedures associated with environmental measurements to verify that the resulting data re of acceptable quality (i.e., that they meet specified acceptance criteria). (NELAC)

Data Reduction:
The process of transforming raw data by arithmetic or statistical calculations, standard curves, concentration factors, etc., and collation into a more useable form. (EPA-QAD)

Deficiency:
An unauthorized deviation from acceptable procedures or practices, or a defect in an item. (ASQC)

Detection Limit:
The lowest concentration or amount of the target analyte that can be identified, measured, and reported with confidence that the analyte concentration is not a false positive value. See Method Detection Limit. (NELAC)

Document Control:
The act of ensuring that documents (and revisions thereto) are proposed, reviewed for accuracy, approved for release by authorized personnel, distributed properly, and controlled to ensure use of the correct version at the location where the prescribed activity if performed. (ASQC)

Duplicate Analyses:

The analyses or measurements of the variable of interest performed identically on two subsamples of the same sample. The results from duplicate analyses are used to evaluate analytical or measurement precision but not the precision of sampling, preservation or storage internal to the laboratory. (EPA-QAD)

Environmental Detection Limit (EDL):
The smallest level at which a radionuclide in an environmental medium can be unambiguously distinguished for a given confidence interval using a particular combination of sampling and measurement procedures, sample size, analytical detection limit, and processing procedure. The EDL shall be specified for the 0.95 or greater confidence interval. The EDL shall be established initially and verified annually for each test method and sample matrix. (NELAC Radioanalysis Subcommittee)

Equipment Blank:
Sample of analyte-free media which has been used to rinse common sampling equipment to check effectiveness of decontamination procedures. (NELAC)

External Standard Calibration:
Calibrations for methods that do not utilize internal standards to compensate for changes in instrument conditions.

Federal Water Pollution Control Act (Clean Water Act, CWA):
The enabling legislation under 33 U.S.C. 1251 et seq., Public Law 92-50086 Stat 816, that empowers EPA to set discharge limitations, write discharge permits, monitor, and bring enforcement action for non-compliance. (NELAC)

Field Blank:
Blank prepared in the field by filing a clean container with pure de-ionized water and appropriate preservative, if any, for the specific sampling activity being undertaken (EPA OSWER)

Field of Testing:
NELAC’s approach to accrediting laboratories by program, method and analyte. Laboratories requesting accreditation for a program-method-analyte combination or for an up-dated/improved method are required to submit to only that portion of the accreditation process not previously addressed (see NELAC, section 1.9ff). (NELAC)

Holding Times (Maximum Allowable Holding Times):
The maximum times that samples may be held prior to analyses and still be considered valid or not compromised. (40 CFR Part 136)

Internal Standard:
A known amount of standard added to a test portion of a sample and carried through the entire measurement process as a reference for evaluating and controlling the precision and bias of the applied analytical test method. (NELAC)

Internal Standard Calibration:
Calibrations for methods that utilize internal standards to compensate for changes in instrument conditions.

Instrument Blank:

A clean sample (e.g., distilled water) processed through the instrumental steps of the measurement process; used to determine instrument contamination. (EPA-QAD)

Laboratory Control Sample (however named, such as laboratory fortified blank, spiked blank, or QC check sample):
A sample matrix, free from the analytes of interest, spiked with verified known amounts of analytes or a material containing known and verified amounts of analytes, taken through all preparation and analysis steps. Where there is no preparation taken for an analysis (such as in aqueous volatiles), or when all samples and standards undergo the same preparation and analysis process (such as Phosphorus), there is no LCS. It is generally used to establish intra-laboratory or analyst specific precision and bias or to assess the performance of all or a portion of the measurement system.

An LCS shall be prepared at a minimum of 1 per batch of 20 or less samples per matrix type per sample extraction or preparation method except for analytes for which spiking solutions are not available such as total suspended solids, total dissolved solids, total volatile solids, total solids, pH, color, odor, temperature, dissolved oxygen or turbidity. The results of these samples shall be used to determine batch acceptance.

Note: NELAC standards allow a matrix spike to be used in place of this control as long as the acceptance criteria are as stringent as for the LCS. (NELAC)

Laboratory Duplicate:
Aliquots of a sample taken from the same container under laboratory conditions and processed and analyzed independently. (NELAC)

Least Squares Regression (1st Order Curve):
The least squares regression is a mathematical calculation of a straight line over two axes. The y axis represents the instrument response (or Response ratio) of a standard or sample and the x axis represents the concentration. The regression calculation will generate a correlation coefficient (r) that is a measure of the "goodness of fit" of the regression line to the data. A value of 1.00 indicates a perfect fit. In order to be used for quantitative purposes, r must be greater than or equal to 0.99 for organics and 0.995 for Inorganics.

Limit of Detection (LOD):
An estimate of the minimum amount of a substance that an analytical process can reliably detect. An LOD is analyte- and matrix-specific and may be laboratory dependent. (Analytical Chemistry, 55, p.2217, December 1983, modified) See also Method Detection Limit.

Matrix:
The component or substrate that contains the analyte of interest. For purposes of batch and QC requirement determinations, the following matrix distinctions shall be used:

> Aqueous: Any aqueous sample excluded from the definition of Drinking Water matrix or Saline/Estuarine source. Includes surface water, groundwater, effluents, and TCLP or other extracts.
>
> Drinking Water: any aqueous sample that has been designated as a potable or potential potable water source.

Saline/Estuarine: any aqueous sample from an ocean or estuary, or other salt water source such as the Great Salt Lake.

Non-aqueous Liquid: any organic liquid with <15% Settleable solids.

Biological Tissue: any sample of a biological origin such as fish tissue, shellfish, or plant material. Such samples shall be grouped according to origin.

Solids: includes soils, sediments, sludges, and other matrices with >15% Settleable solids.

Chemical Waste: a product or by-product of an industrial process that results in a matrix not previously defined.

Air: whole gas or vapor samples including those contained in flexible or rigid wall containers and the extracted concentrated analytes of interest from a gas or vapor that are collected with a sorbant tube, impinger solution, filter, or other device. (NELAC)

Matrix Spike (spiked sample or fortified sample):

Prepared by adding a known mass of target analyte to a specified amount of matrix sample for which an independent estimate of target analyte concentration is available. Matrix spikes are used, for example, to determine the effect of the matrix on a method's recovery efficiency.

Matrix spikes shall be performed at a frequency of one in 20 samples per matrix type per sample extraction or preparation method except for analytes for which spiking solutions are not available such as, total suspended solids, total dissolved solids, total volatile solids, total solids, pH, color, odor, temperature, dissolved oxygen or turbidity. The selected sample(s) shall be rotated among client samples so that various matrix problems may be noted and/or addressed. Poor performance in a matrix spike may indicate a problem with the sample composition and shall be reported to the client whose sample was used for the spike. (QAMS)

Matrix Spike Duplicate (spiked sample or fortified sample duplicate):
A second replicate matrix spike is prepared in the laboratory and analyzed to obtain a measure of the precision of the recovery for each analyte.

Matrix spike duplicates or laboratory duplicates shall be analyzed at a minimum of 1 in 20 samples per matrix type per sample extraction or preparation method. The laboratory shall document their procedure to select the use of an appropriate type of duplicate. The selected sample(s) shall be rotated among client samples so that various matrix problems may be noted and/or addressed. Poor performance in the duplicates may indicate a problem with the sample composition and shall be reported to the client whose sample was used for the duplicate. (QAMS)

Method Blank:
A sample of a matrix similar to the batch of associated samples (when available) that is free from the analytes of interest and is processed simultaneously with and under the same conditions as samples through all steps of the analytical procedures, and in which no target analytes or interferences are present at concentrations that impact the analytical results for sample analyses. (NELAC)

Method Detection Limit:
The minimum concentration of a substance (an analyte) that can be measured and reported with 99% confidence that the analyte concentration is greater than zero and is determined from analysis of a sample in a given matrix containing the analyte. (40 CFR Part 136, Appendix B)

Negative Control:
Measures taken to ensure that a test, its components, or the environment do not cause undesired effects, or produce incorrect test results. (NELAC)

Performance Audit:
The routine comparison of independently obtained qualitative and quantitative measurement system data with routinely obtained data in order to evaluate the proficiency of an analyst or laboratory. (NELAC)

Performance Based Measurement System (PBMS):
A set of processes wherein the data quality needs, mandates or limitations of a program or project are specified and serve as criteria for selecting appropriate test methods to meet those needs in a cost-effective manner. (NELAC)

Positive Control:
Measures taken to ensure that a test and/or its components are working properly and producing correct or expected results from positive test subjects. (NELAC)

Precision:
The degree to which a set of observations or measurements of the same property, obtained under similar conditions, conform to themselves; a data quality indicator. Precision is usually expressed as standard deviation, variance or range, in either absolute or relative terms. (NELAC)

Preservation:
Refrigeration and/or reagents added at the time of sample collection (or later) to maintain the chemical and/or biological integrity of the sample. (NELAC)

Proficiency Testing:
A means of evaluating a laboratory's performance under controlled conditions relative to a given set of criteria through analysis of unknown samples provided by an external source. (NELAC) [2.1]

Proficiency Testing Program:
The aggregate of providing rigorously controlled and standardized environmental samples to a laboratory for analysis, reporting of results, statistical evaluation of the results and the collective demographics and results summary of all participating laboratories. (NELAC)

Proficiency Test Sample (PT):
A sample, the composition of which is unknown to the analyst and is provided to test whether the analyst/laboratory can produce analytical results within specified acceptance criteria. (QAMS)

Quality Assurance:
An integrated system of activities involving planning, quality control, quality assessment, reporting and quality improvement to ensure that a product or service meets defined standards of quality with a stated level of confidence. (QAMS)

Quality Assurance [Project] Plan (QAPP):
A formal document describing the detailed quality control procedures by which the quality requirements defined for the data and decisions pertaining to a specific project are to be achieved. (EAP-QAD)

Quality Control:
The overall system of technical activities which purpose is to measure and control the quality of a product or service so that it meets the needs of users. (QAMS)

Quality Control Sample:
An uncontaminated sample matrix spiked with known amounts of analytes from a source independent from the calibration standards. It is generally used to establish intra-laboratory or analyst specific precision and bias or to assess the performance of all or a portion of the measurement system. (EPA-QAD)

Quality Manual:
A document stating the management policies, objectives, principles, organizational structure and authority, responsibilities, accountability, and implementation of an agency, organization, or laboratory, to ensure the quality of its product and the utility of its product to its users. (NELAC)

Quality System:
A structured and documented management system describing the policies, objectives, principles, organizational authority, responsibilities, accountability, and implementation plan of an organization for ensuring quality in its work processes, products (items), and services. The quality system provides the framework for planning, implementing, and assessing work performed by the organization and for carrying out required QA and QC (ANSI/ASQC-E-41994)

Quantitation Limits:
The maximum or minimum levels, concentrations, or quantities of a target variable (e.g., target analyte) that can be quantified with the confidence level required by the data user. (NELAC)

Range:
The difference between the minimum and the maximum of a set of values. (EPA-QAD)

Reagent Blank (method reagent blank):
A sample consisting of reagent(s), without the target analyte or sample matrix, introduced into the analytical procedure at the appropriate point and carried through all subsequent steps to determine the contribution of the reagents and of the involved analytical steps. (QAMS)

Reference Material:
A material or substance one or more properties of which are sufficiently well established to be used for the calibration of an apparatus, the assessment of a measurement method, or for assigning values to materials. (ISO Guide 30-2.1)

Reference Standard:
A standard, generally of the highest metrological quality available at a given location, from which measurements made at that location are derived. (VIM-6.0-8)

Replicate Analyses:
The measurements of the variable of interest performed identically on two or more sub-samples of the same sample within a short time interval. (NELAC)

<u>Report Limit (RL):</u>
The laboratory nominal Quantitation Limit (QL) or the level of sensitivity required by the client but not lower than the LOD.

Resource Conservation and Recovery Act (RCRA):
The enabling legislation under 42 USC 321 et seq. (1976), that gives EPA the authority to control hazardous waste from the “cradle-to-grave”, including its generation, transportation, treatment, storage, and disposal. (NELAC)

Safe Drinking Water Act (SDWA):
The enabling legislation, 42 USC 300f et seq. (1974), (Public Law 93-523), that requires the EPA to protect the quality of drinking water in the U.S. by setting maximum allowable contaminant levels, monitoring, and enforcing violations. (NELAC)

Sample Duplicate:
Two samples taken from and representative of the same population and carried through all steps of the sampling and analytical procedures in an identical manner. Duplicate samples are used to assess variance of the total method including sampling and analysis. (EPA-QAD)

Second Order Polynomial Curve (Quadratic): The 2nd order curves are a mathematical calculation of a slightly curved line over two axis. The y axis represents the instrument response (or Response ratio) of a standard or sample and the x axis represents the concentration. The 2nd order regression will generate a coefficient of determination (COD or r^2) that is a measure of the "goodness of fit" of the quadratic curvature the data. A value of 1.00 indicates a perfect fit. In order to be used for quantitative purposes, r^2 must be greater than or equal to 0.99.

Selectivity:
(Analytical chemistry) the capability of a test method or instrument to respond to a target substance of constituent in the presence of non-target substances. (EPA-QAD)

Sensitivity:

The capability of a method or instrument to discriminate between measurement responses representing different levels (e.g., concentrations) of a variable of interest. (NELAC)

Spike:
A known mass of target analyte added to a blank, sample or sub-sample; used to determine recovery efficiency or for other quality control purposes.

If the mandated or requested test method does not specify the spiking components, the laboratory shall spike all reportable components to be reported in the Laboratory Control Sample and Matrix Spike. However, in cases where the components interfere with accurate assessment (such as simultaneously spiking chlordane, toxaphene and PCBs in Method 608), the test method has an extremely long list of components or components are incompatible, a representative number (at a minimum 10%) of the listed components may be used to control the test method. The selected components of each spiking mix shall represent all chemistries, elution patterns and masses permit specified analytes and other client requested components. However, the laboratory shall ensure that all reported components are used in the spike mixture within a two-year time period.. (NELAC)

Standard:
The document describing the elements of laboratory accreditation that has been developed and established within the consensus principles of NELAC and meets the approval requirements of NELAC procedures and policies. (ASQC)

Standard Operating Procedures (SOPs):
A written document which details the method of an operation, analysis, or action whose techniques and procedures are thoroughly prescribed and which is accepted as the method for performing certain routine or repetitive tasks. (QAMS)

Standardized Reference Material (SRM):
A certified reference material produced by the U.S. National Institute of Standards and Technology or other equivalent organization and characterized for absolute content, independent of analytical method. (EPA-QAD)

Surrogate:
A substance with properties that mimic the analyte of interest. It is unlikely to be found in environment samples and is added to them for quality control purposes.

Surrogate compounds must be added to all samples, standards, and blanks, for all organic chromatography methods except when the matrix precludes its use or when a surrogate is not available. Poor surrogate recovery may indicate a problem with sample composition and shall be reported to the client whose sample produced poor recovery. (QAMS)

Systems Audit (also Technical Systems Audit):
A thorough, systematic, qualitative on-site assessment of the facilities, equipment, personnel, training, procedures, record keeping, data validation, data management, and reporting aspects of a total measurement system. (EPA-QAD)

Toxic Substances Control Act (TSCA):

The enabling legislation in 15 USC 2601 et seq., (1976) that provides for testing, regulating, and screening all chemicals produced or imported into the United States for possible toxic effects prior to commercial manufacture. (NELAC)

Traceability:
The property of a result of a measurement whereby it can be related to appropriate standards, generally international or national standards, through an unbroken chain of comparisons. (VIM-6.12)

Uncertainty:
A parameter associated with the result of a measurement that characterizes the dispersion of the value that could reasonably be attributed to the measured value.

Acronyms:

BS – Blank Spike
BSD – Blank Spike Duplicate
CAR – Corrective Action Report
CCV – Continuing Calibration Verification
CF – Calibration Factor
CFR – Code of Federal Regulations
COC – Chain of Custody
CRS – Change Request Form
DOC – Demonstration of Capability
DQO – Data Quality Objectives
DU – Duplicate
DUP - Duplicate
EHS – Environment, Health and Safety
EPA – Environmental Protection Agency
GC - Gas Chromatography
GC/MS - Gas Chromatography/Mass Spectrometry
HPLC - High Performance Liquid Chromatography
ICP - Inductively Coupled Plasma Atomic Emission Spectroscopy
ICV – Initial Calibration Verification
IDL – Instrument Detection Limit
IH – Industrial Hygiene
IS – Internal Standard
LCS – Laboratory Control Sample
LCSD – Laboratory Control Sample Duplicate
LIMS – Laboratory Information Management System
MDL – Method Detection Limit
MS – Matrix Spike
MSD – Matrix Spike Duplicate
MSDS - Material Safety Data Sheet
NELAC - National Environmental Laboratory Accreditation Conference
NELAP - National Environmental Laboratory Accreditation Program
PT – Performance Testing
QAM – Quality Assurance Manual
QA/QC – Quality Assurance / Quality Control
QAPP – Quality Assurance Project Plan
RF – Response Factor
RPD – Relative Percent Difference
RSD – Relative Standard Deviation
SD – Standard Deviation
SOP: Standard Operating Procedure
TAT – Turn-Around-Time
VOA – Volatiles
VOC – Volatile Organic Compound

Appendix 3.

Laboratory Certifications, Accreditations, Validations

TestAmerica Buffalo maintains certifications, accreditations, certifications, and validations with numerous state and national entities. Programs vary but may include on-site audits, reciprocal agreements with another entity, performance testing evaluations, review of the QA Manual, Standard Operating Procedures, Method Detection Limits, training records, etc. At the time of this QA Manual revision, the laboratory has accreditation/certification/licensing with the following organizations:

State	***Program***	***Certification Number***
Arkansas	*SDWA, CWA, RCRA, SOIL*	*88-0686*
California*	*NELAP CWA, RCRA*	*01169CA*
Connecticut	*SDWA, CWA, RCRA, SOIL*	*PH-0568*
Florida*	*NELAP CWA, RCRA*	*E87672*
Georgia*	*SDWA,NELAP CWA, RCRA*	*956*
Illinois*	*NELAP SDWA, CWA, RCRA*	*200003*
Iowa	*SW/CS*	*374*
Kansas*	*NELAP SDWA, CWA, RCRA*	*E-10187*
Kentucky	*SDWA*	*90029*
Kentucky UST	*UST*	*30*
Louisiana*	*NELAP CWA, RCRA*	*2031*
Maine	*SDWA, CWA*	*NY0044*
Maryland	*SDWA*	*294*
Massachusetts	*SDWA, CWA*	*M-NY044*
Michigan	*SDWA*	*9937*
Minnesota	*SDWA,CWA, RCRA*	*036-999-337*
New Hampshire*	*NELAP SDWA, CWA*	*233701*
New Jersey*	*NELAP,SDWA, CWA, RCRA,*	*NY455*
New York*	*NELAP, AIR, SDWA, CWA, RCRA,CLP*	*10026*
Oklahoma	*CWA, RCRA*	*9421*
Pennsylvania*	*Registration, NELAP CWA,RCRA*	*68-00281*
Tennessee	*SDWA*	*02970*
USDA	*FOREIGN SOIL PERMIT*	*S-41579*
USDOE	*Department of Energy*	*DOECAP-STB*
Virginia	*SDWA*	*278*
Washington	*CWA,RCRA*	*C1677*
West Virginia	*CWA,RCRA*	*252*
Wisconsin	*CWA, RCRA*	*998310390*

The certificates and parameter lists (which may differ) for each organization may be found on the corporate web site, the laboratory's public server, and in the QA official.

ARCADIS

Attachment 3

Field Standard Operating Procedures

Attachment 3A
Chain-of-Custody, Handling, Packing, and Shipping

ARCADIS
Infrastructure, environment, buildings

Imagine the result

Chain-of-Custody, Handling, Packing and Shipping

Rev. #: 2

Rev Date: March 6, 2009

Approval Signatures

Prepared by: Caron Koll Date: 3/6/09
Caron Koll

Reviewed by: Date: 3/6/09
Jane Kennedy(Technical Expert)

I. Scope and Application

This Standard Operating Procedure (SOP) describes the chain-of-custody, handling, packing, and shipping procedures for the management of samples to decrease the potential for cross-contamination, tampering, mis-identification, and breakage, and to insure that samples are maintained in a controlled environment from the time of collection until receipt by the analytical laboratory.

II. Personnel Qualifications

ARCADIS field sampling personnel will have current health and safety training, including 40-hour HAZWOPER training, Department of Transportation (DOT) training, site supervisor training, and site-specific training, as needed. In addition, ARCADIS field sampling personnel will be versed in the relevant SOPs and possess the skills and experience necessary to successfully complete the desired field work.

III. Equipment List

The following list provides materials that may be required for each project. Project documents and sample collection requirements should be reviewed prior to initiating field operations:

- indelible ink pens (black or blue);
- polyethylene bags (resealable-type);
- clear packing tape, strapping tape, duct tape;
- chain of custody
- DOT shipping forms, as applicable
- custody seals or tape;
- appropriate sample containers and labels,;
- insulated coolers of adequate size for samples and sufficient ice to maintain 4°C during collection and transfer of samples;
- wet ice;
- cushioning and absorbent material (i.e., bubble wrap or bags);

- temperature blank
- sample return shipping papers and addresses; and
- field notebook.

IV. Cautions

Review project requirements and select appropriate supplies prior to field mobilization.

Insure that appropriate sample containers with applicable preservatives, coolers, and packing material have been supplied by the laboratory.

Understand the offsite transfer requirements for the facility at which samples are collected.

If overnight courier service is required schedule pick-up or know where the drop-off service center is located and the hours of operation. Prior to using air transportation, confirm air shipment is acceptable under DOT and International Air Transport Association (IATA) regulation

Schedule pick-up time for laboratory courier or know location of laboratory/service center and hours of operation.

Understand DOT and IATA shipping requirements and evaluate dangerous goods shipping regulations relative to the samples being collected (i.e. complete an ARCADIS shipping determination). Review the ARCADIS SOPs for shipping, packaging and labeling of dangerous goods. Potential samples requiring compliance with this DOT regulation include:

- Methanol preservation for Volatile Organic Compounds in soil samples
- Non-aqueous phase liquids (NAPL)

V. Health and Safety Considerations

Follow health and safety procedures outlined in the project/site Health and Safety Plan (HASP).

Use caution and appropriate cut resistant gloves when tightening lids to 40 mL vials. These vials can break while tightening and can lacerate hand. Amber vials (thinner glass) are more prone to breakage.

Some sample containers contain preservatives.

- The preservatives must be retained in the sample container and should in no instance be rinsed out.
- Preservatives may be corrosive and standard care should be exercised to reduce potential contact to personnel skin or clothing. Follow project safety procedures if spillage is observed.
- If sample container caps are broken discard the bottle. Do not use for sample collection.

VI. Procedure

Chain-of-Custody Procedures

1. Prior to collecting samples, complete the chain-of-custody record header information by filling in the project number, project name, and the name(s) of the sampling technician(s) and other relevant project information. Attachment 1 provides an example chain-o- custody record

2. Chain-of-custody information MUST be printed legibly using indelible ink (black or blue).

3. After sample collection, enter the individual sample information on the chain-of-custody:

   a. Sample Identification indicates the well number or soil location that the sample was collected from. Appropriate values for this field include well locations, grid points, or soil boring identification numbers (e.g., MW-3, X-20, SB-30). When the depth interval is included, the complete sample ID would be “SB-30 (0.5-1.0) where the depth interval is in feet. Please note it is very important that the use of hyphens in sample names and depth units (i.e., feet or inches) remain consistent for all samples entered on the chain-of-custody form. DO NOT use the apostrophe or quotes in the sample ID. Sample names may also use the abbreviations “FB,” “TB,” and “DUP” as prefixes or suffixes to indicate that the sample is a field blank, trip blank, or field duplicate, respectively. NOTE: The sample nomenclature may be dictated by the project database and require unique identification for each sample collected for the project. Consult the project data management plan for additional information regarding sample identification.

b. List the date o sample collection. The date format to be followed should be mm/dd/yy (e.g., 03/07/09) or mm/dd/yyyy (e.g. 03/07/2009).

c. List the time that the sample was collected. The time value should be presented using military format. For example, 3:15 P.M. should be entered as 15:15.

d. The composite field should be checked if the sample is a composite over a period of time or from several different locations and mixed prior to placing in sample containers.

e. The “Grab”. field should be marked with an “X” if the sample was collected as an individual grab sample. (e.g. monitoring well sample or soil interval).

f. Any sample preservation should be noted.

g. The analytical parameters that the samples are being analyzed for should be written legibly on the diagonal lines. As much detail as possible should be presented to allow the analytical laboratory to properly analyze the samples. For example, polychlorinated biphenyl (PCB) analyses may be represented by entering “PCBs” or “Method 8082.” Multiple methods and/or analytical parameters may be combined for each column (e.g., PCBs/VOCs/SVOCs or 8082/8260/8270). These columns should also be used to present project-specific parameter lists (e.g., Appendix IX+3 target analyte list. Each sample that requires a particular parameter analysis will be identified by placing the number of containers in the appropriate analytical parameter column. For metals in particular, indicate which metals are required.

h. Number of containers for each method requested. This information may be included under the parameter or as a total for the sample based on the chain of custody form used.

i. Note which samples should be used for site specific matrix spikes.

j. Indicate any special project requirements.

k. Indicate turnaround time required.

l. Provide contact name and phone number in the event that problems are encountered when samples are received at the laboratory.

m. If available attach the Laboratory Task Order or Work Authorization forms

n. The remarks field should be used to communicate special analytical requirements to the laboratory. These requirements may be on a per sample basis such as “extract and hold sample until notified,” or may be used to inform the laboratory of special reporting requirements for the entire sample delivery group (SDG). Reporting requirements that should be specified in the remarks column include: 1) turnaround time; 2) contact and address where data reports should be sent; 3) name of laboratory project manager; and 4) type of sample preservation used.

o. The “Relinquished By” field should contain the signature of the sampling technician who relinquished custody of the samples to the shipping courier or the analytical laboratory.

p. The “Date” field following the signature block indicates the date the samples were relinquished. The date format should be mm/dd/yyyy (e.g., 03/07/2005).

q. The “Time” field following the signature block indicates the time that the samples were relinquished. The time value should be presented using military format. For example, 3:15 P.M. should be entered as 15:15.

r. The “Received By” section is signed by sample courier or laboratory representative who received the samples from the sampling technician or it is signed upon laboratory receipt from the overnight courier service.

3. Complete as many chain-of-custody forms as necessary to properly document the collection and transfer of the samples to the analytical laboratory.

4. Upon completing the chain-of-custody forms, forward two copies to the analytical laboratory and retain one copy for the field records.

5. If electronic chain-of-custody forms are utilized, sign the form and make 1 copy for ARCADIS internal records and forward the original with the samples to the laboratory.

Handling Procedures

1. After completing the sample collection procedures, record the following information in the field notebook with indelible ink:

   - project number and site name;
   - sample identification code and other sample identification information, if appropriate;
   - sampling method;
   - date;
   - name of sampler(s);
   - time;
   - location (project reference);
   - location of field duplicates and both sample identifications;
   - locations that field QC samples were collected including equipment blanks, field blanks and additional sample volume for matrix spikes; and
   - any comments.

2. Complete the sample label with the following information in indelible ink:

   - sample type (e.g., surface water);
   - sample identification code and other sample identification information, if applicable;
   - analysis required;
   - date;
   - time sampled; and
   - initials of sampling personnel;

- sample matrix; and
- preservative added, if applicable.

3. Cover the label with clear packing tape to secure the label onto the container and to protect the label from liquid.

4. Confirm that all caps on the sample containers are secure and tightly closed.

5. In some instances it may be necessary to wrap the sample container cap with clear packing tape to prevent it from becoming loose.

6. For some projects individual custody seals may be required. Custody seal evidence tape may be placed on the shipping container or they may be placed on each sample container such that the cooler or cap cannot be opened without breaking the custody seal. The custody seal should be initialed and dated prior to relinquishing the samples.

Packing Procedures

Following collection, samples must be placed on wet ice to initiate cooling to 4°C immediately. Retain samples on ice until ready to pack for shipment to the laboratory.

1. Secure the outside and inside of the drain plug at the bottom of the cooler being used for sample transport with “Duct” tape.

2. Place a new large heavy duty plastic garbage bag inside each cooler

3. Place each sample bottle wrapped in bubble wrap inside the garbage bag. VOC vials may be grouped by sample in individual resealable plastic bags). If a cooler temperature blank is supplied by the laboratory, it should be packaged following the same procedures as the samples. If the laboratory did not include a temperature blank, do not add one. Place 1 to 2 inches of cushioning material (i.e., vermiculite) at the bottom of the cooler.

4. Place the sealed sample containers upright in the cooler.

5. Package ice in large resealable plastic bags and place inside the large garbage bag in the cooler. Samples placed on ice will be cooled to and maintained at a temperature of approximately 4°C.

6. Fill the remaining space in the cooler with cushioning material such as bubble wrap. The cooler must be securely packed and cushioned in an upright position and be surrounded (Note: to comply with 49 CFR 173.4, filled cooler must not exceed 64 pounds).

7. Place the completed chain-of-custody record(s) in a large resealable bag and tape the bag to the inside of the cooler lid.

8. Close the lid of the cooler and fasten with packing tape.

9. Wrap strapping tape around both ends of the cooler.

10. Mark the cooler on the outside with the following information: shipping address, return address, “Fragile, Handle with Care” labels on the top and on one side, and arrows indicating “This Side Up” on two adjacent sides.

11. Place custody seal evidence tape over front right and back left of the cooler lid, initial and date, then cover with clear plastic tape.

Note: Procedure numbers 2, 3, 5, and 6 may be modified in cases where laboratories provide customized shipping coolers. These cooler types are designed so the sample bottles and ice packs fit snugly within preformed styrofoam cushioning and insulating packing material.

Shipping Procedures

1. All samples will be delivered by an express carrier within 48 hours of sample collection. Alternatively, samples may be delivered directly to the laboratory or laboratory service center or a laboratory courier may be used for sample pickup.

2. If parameters with short holding times are required (e.g., VOCs [EnCore™ Sampler], nitrate, nitrite, ortho-phosphate and BOD), sampling personnel will take precautions to ship or deliver samples to the laboratory so that the holding times will not be exceeded.

3. Samples must be maintained at 4°C+2°C until shipment and through receipt at the laboratory

4. All shipments must be in accordance with DOT regulations and ARCADIS dangerous goods shipping SOPs.

5. When the samples are received by the laboratory, laboratory personnel will complete the chain-of-custody by recording the date and time of receipt of samples, measuring and recording the internal temperature of the shipping container, and checking the sample identification numbers on the containers to ensure they correspond with the chain-of-custody forms.

Any deviations between the chain-of-custody and the sample containers, broken containers, or temperature excursions will be communicated to ARCADIS immediately by the laboratory.

VII. Waste Management

Not applicable

VIII. Data Recording and Management

Chain-of-custody records will be transmitted to the ARCADIS PM or designee at the end of each day unless otherwise directed by the ARCADIS PM. The sampling team leader retains copies of the chain-of-custody forms for filing in . the project file. Record retention shall be in accordance with project requirements.

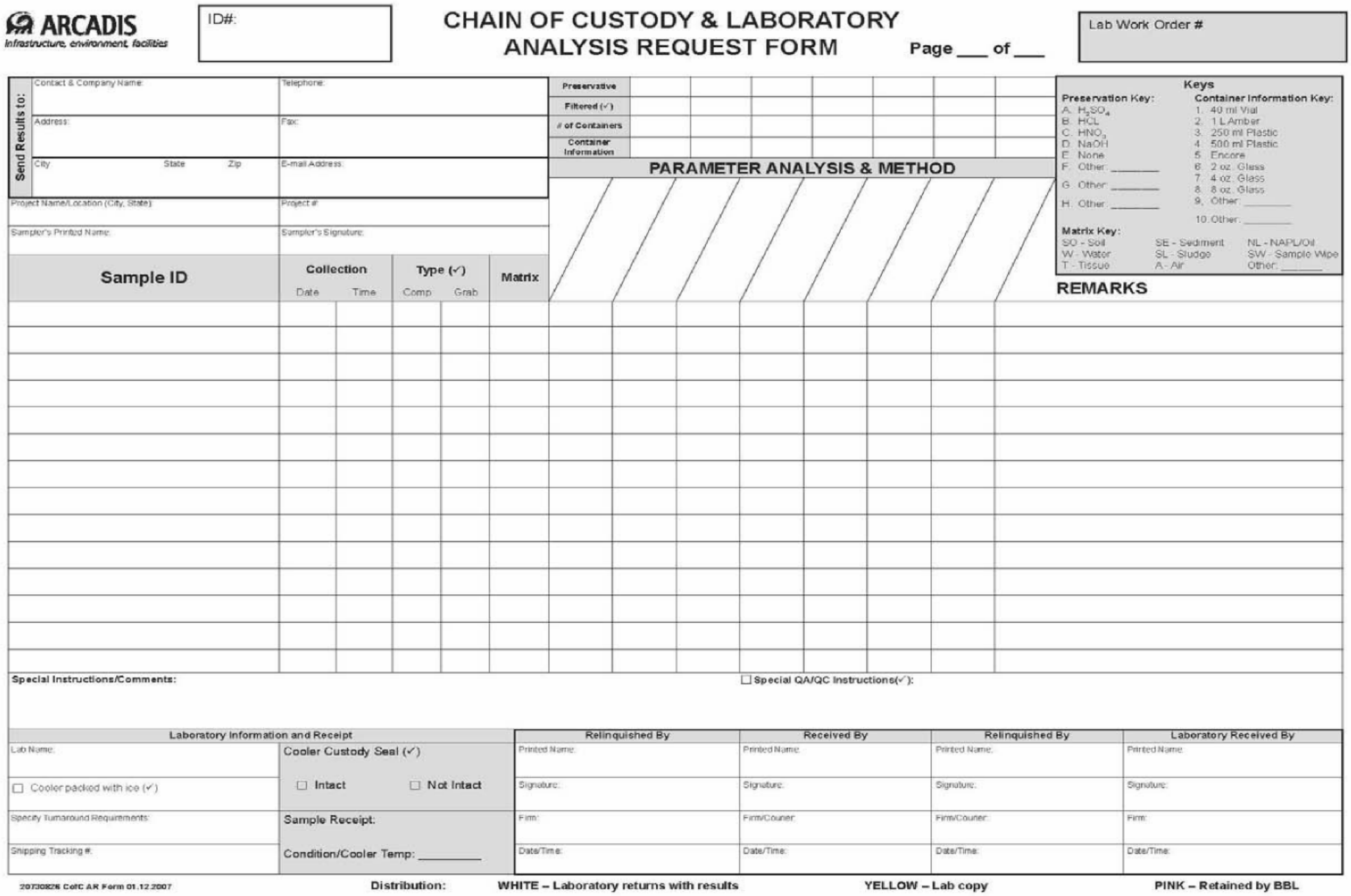
IX. Quality Assurance

Chain-of-custody forms will be legibly completed in accordance with the applicable project documents such as Sampling and Analysis Plan (SAP), Quality Assurance Project Plan (QAPP), Work Plan, or other project guidance documents. A copy of the completed chain-of-custody form will be sent to the ARCADIS Project Manager or designee for review.

X. References

Not Applicable

Attachment 1



ARCADIS
Infrastructure, environment, facilities

ID#:

CHAIN OF CUSTODY & LABORATORY ANALYSIS REQUEST FORM

Page ___ of ___

Lab Work Order #

Send Results to:

Contact & Company Name:	Telephone:
Address:	Fax:
City State Zip	E-mail Address:
Project Name/Location (City, State):	Project #:
Sampler's Printed Name:	Sampler's Signature:

Preservative							
Filtered (✓)							
# of Containers							
Container Information							

PARAMETER ANALYSIS & METHOD

Keys

Preservation Key:
A. H_2SO_4
B. HCL
C. HNO_3
D. NaOH
E. None
F. Other: ________
G. Other: ________
H. Other: ________

Container Information Key:
1. 40 ml Vial
2. 1 L Amber
3. 250 ml Plastic
4. 500 ml Plastic
5. Encore
6. 2 oz. Glass
7. 4 oz. Glass
8. 8 oz. Glass
9. Other: ________
10. Other: ________

Matrix Key:

SO - Soil	SE - Sediment	NL - NAPL/Oil
W - Water	SL - Sludge	SW - Sample Wipe
T - Tissue	A - Air	Other: ________

Sample ID	**Collection** Date	Time	**Type (✓)** Comp	Grab	**Matrix**								**REMARKS**

Special Instructions/Comments:

☐ **Special QA/QC Instructions(✓):**

Laboratory Information and Receipt		**Relinquished By**	**Received By**	**Relinquished By**	**Laboratory Received By**
Lab Name:	Cooler Custody Seal (✓)	Printed Name:	Printed Name:	Printed Name:	Printed Name:
☐ Cooler packed with ice (✓)	☐ Intact ☐ Not Intact	Signature:	Signature:	Signature:	Signature:
Specify Turnaround Requirements:	Sample Receipt:	Firm:	Firm/Courier:	Firm/Courier:	Firm:
Shipping Tracking #:	Condition/Cooler Temp: _________	Date/Time:	Date/Time:	Date/Time:	Date/Time:

20730826 CofC AR Form 01.12.2007

Distribution: **WHITE – Laboratory returns with results** **YELLOW – Lab copy** **PINK – Retained by BBL**

Attachment 3B
Field Equipment Decontamination

ARCADIS
Infrastructure, environment, facilities

Imagine the result

Field Equipment Decontamination

Rev. #: 3

Rev Date: April 26, 2010

Approval Signatures

Prepared by: ______________________ Date: 4/26/2010
Keith Shepherd

Reviewed by: ______________________ Date: 4/26/2010
Richard Murphy (Technical Expert)

I. Scope and Application

Equipment decontamination is performed to ensure that sampling equipment that contacts a sample, or monitoring equipment that is brought into contact with environmental media to be sampled, is free from analytes of interest and/or constituents that would interfere with laboratory analysis for analytes of interest. Equipment must be cleaned prior to use for sampling or contact with environmental media to be sampled, and prior to shipment or storage. The effectiveness of the decontamination procedure should be verified by collecting and analyzing equipment blank samples.

The equipment cleaning procedures described herein includes pre-field, in the field, and post-field cleaning of sampling tools which will be conducted at an established equipment decontamination area (EDA) on site (as appropriate). Equipment that may require decontamination at a given site includes: soil sampling tools; groundwater, sediment, and surface-water sampling devices; water testing instruments; down-hole instruments; and other activity-specific sampling equipment. Non-disposable equipment will be cleaned before collecting each sample, between sampling events, and prior to leaving the site. Cleaning procedures for sampling equipment will be monitored by collecting equipment blank samples as specified in the applicable work plan or field sampling plan. Dedicated and/or disposable (not to be re-used) sampling equipment will not require decontamination.

II. Personnel Qualifications

ARCADIS field sampling personnel will have current health and safety training, including 40-hour HAZWOPER training, site supervisor training, and site-specific training, as needed. In addition, ARCADIS field sampling personnel will be versed in the relevant SOPs and possess the skills and experience necessary to successfully complete the desired fieldwork. The project HASP and other documents will identify any other training requirements such as site specific safety training or access control requirements.

III. Equipment List

- health and safety equipment, as required in the site Health and Safety Plan (HASP)
- distilled water

- Non-phosphate detergent such as Alconox or, if sampling for phosphorus phosphorus-containing compounds, Luminox (or equivalent).
- tap water
- rinsate collection plastic containers
- DOT-approved waste shipping container(s), as specified in the work plan or field sampling plan (if decontamination waste is to be shipped for disposal)
- brushes
- large heavy-duty garbage bags
- spray bottles
- (Optional) – Isoprophyl alcohol (free of ketones) or methanol
- Ziploc-type bags
- plastic sheeting

IV. Cautions

Rinse equipment thoroughly and allow the equipment to dry before re-use or storage to prevent introducing solvent into sample medium. If manual drying of equipment is required, use clean lint-free material to wipe the equipment dry.

Store decontaminated equipment in a clean, dry environment. Do not store near combustion engine exhausts.

If equipment is damaged to the extent that decontamination is uncertain due to cracks or dents, the equipment should not be used and should be discarded or submitted for repair prior to use for sample collection.

A proper shipping determination will be performed by a DOT-trained individual for cleaning materials shipped by ARCADIS.

V. Health and Safety Considerations

Review the material safety data sheets (MSDS) for the cleaning materials used in decontamination. If solvent is used during decontamination, work in a well-ventilated area and stand upwind while applying solvent to equipment. Apply solvent in a manner that minimizes potential for exposure to workers. Follow health and safety procedures outlined in the HASP.

VI. Procedure

A designated area will be established to clean sampling equipment in the field prior to sample collection. Equipment cleaning areas will be set up within or adjacent to the specific work area, but not at a location exposed to combustion engine exhaust. Detergent solutions will be prepared in clean containers for use in equipment decontamination.

Cleaning Sampling Equipment

1. Wash the equipment/pump with potable water.

2. Wash with detergent solution (Alconox, Liquinox or equivalent) to remove all visible particulate matter and any residual oils or grease.

3. If equipment is very dirty, precleaning with a brush and tap water may be necessary.

4. (Optional) – Flush with isopropyl alcohol (free of ketones) or with methanol. This step is optional but should be considered when sampling in highly impacted media such as non-aqueous phase liquids or if equipment blanks from previous sampling events showed the potential for cross contamination of organics.

5. Rinse with distilled/deionized water.

Decontaminating Submersible Pumps

Submersible pumps may be used during well development, groundwater sampling, or other investigative activities. The pumps will be cleaned and flushed before and between uses. This cleaning process will consist of an external detergent solution wash and tap water rinse, a flush of detergent solution through the pump, followed by a flush of potable water through the pump. Flushing will be accomplished by using an appropriate container filled with detergent solution and another contained filled with potable water. The pump will run long enough to effectively flush the pump housing and hose (unless new, disposable hose is used). Caution should be exercised to avoid contact with the pump casing and water in the container while the pump is running (do not use metal drums or garbage cans) to avoid electric shock. Disconnect the pump from the power source before handling. The pump and hose should be placed on or in clean polyethylene sheeting to avoid contact with the ground surface.

VII. Waste Management

Equipment decontamination rinsate will be managed in conjunction with all other waste produced during the field sampling effort. Waste management procedures are outlined in the work plan or Waste Management Plan (WMP).

VIII. Data Recording and Management

Equipment cleaning and decontamination will be noted in the field notebook. Information will include the type of equipment cleaned, the decontamination location and any deviations from this SOP. Specific factors that should be noted include solvent used (if any), and source of water.

Any unusual field conditions should be noted if there is potential to impact the efficiency of the decontamination or subsequent sample collection.

An inventory of the solvents brought on site and used and removed from the site will be maintained in the files. Records will be maintained for any solvents used in decontamination, including lot number and expiration date.

Containers with decontamination fluids will be labeled.

IX. Quality Assurance

Equipment blanks should be collected to verify that the decontamination procedures are effective in minimizing potential for cross contamination. The equipment blank is prepared by pouring deionized water over the clean and dry tools and collecting the deionized water into appropriate sample containers. Equipment blanks should be analyzed for the same set of parameters that are performed on the field samples collected with the equipment that was cleaned. Equipment blanks are collected per equipment set, which represents all of the tools needed to collect a specific sample.

X. References

USEPA Region 9, Field Sampling Guidance #1230, Sampling Equipment Decontamination.

USEPA Region 1, Low Stress (low flow) Purging and Sampling Procedure for the Collection of Groundwater Samples from Monitoring Wells.

Attachment 3C
Soil Drilling and Sample Collection

ARCADIS
Infrastructure, environment, facilities

Imagine the result

Soil Drilling and Sample Collection

Rev. #: 1

Rev Date: March 3, 2009

Approval Signatures

Prepared by: Caron Koll Date: 3/3/09

Reviewed by: Michael J Gefell Date: 3/3/09
(Technical Expert)

I. Scope and Application

Overburden drilling is commonly performed using the hollow-stem auger drilling method. Other drilling methods suitable for overburden drilling, which are sometimes necessary due to site-specific geologic conditions, include: drive-and-wash, spun casing, Rotasonic, dual-rotary (Barber Rig), and fluid/mud rotary. Direct-push techniques (e.g., Geoprobe or cone penetrometer) may also be used. The drilling method to be used at a given site will be selected based on site-specific consideration of anticipated drilling depths, site or regional geologic knowledge, types of sampling to be conducted, required sample quality and volume, and cost.

No oils or grease will be used on equipment introduced into the boring (e.g., drill rod, casing, or sampling tools).

II. Personnel Qualifications

The Project Manager (a qualified geologist, environmental scientist, or engineer) will identify the appropriate soil boring locations, depth and soil sample intervals in a written plan.

Personnel responsible for overseeing drilling operations must have at least 16 hours of prior training overseeing drilling activities with an experienced geologist, environmental scientist, or engineer with at least 2 years of prior experience.

III. Equipment List

The following materials will be available during soil boring and sampling activities, as required:

- Site Plan with proposed soil boring/well locations;
- Work Plan or Field Sampling Plan (FSP), and site Health and Safety Plan (HASP);
- personal protective equipment (PPE), as required by the HASP;
- drilling equipment required by the American Society for Testing and Materials (ASTM) D 1586, when performing split-spoon sampling;
- disposable plastic liners, when drilling with direct-push equipment;
- appropriate soil sampling equipment (e.g., stainless steel spatulas, knife);

- equipment cleaning materials;
- appropriate sample containers and labels;
- chain-of-custody forms;
- insulated coolers with ice, when collecting samples requiring preservation by chilling;
- photoionization detector (PID) or flame ionization detector (FID); and
- field notebook and/or personal digital assistant (PDA).

IV. Cautions

Prior to beginning field work, underground utilities in the vicinity of the drilling areas will be identified by one of the following three actions (lines of evidence):

- Contact the State One Call
- Obtain a detailed site utility plan drawn to scale, preferably an “as-built” plan
- Conduct a detailed visual site inspection

In the event that one or more of the above lines of evidence cannot be conducted, or if the accuracy of utility location is questionable, a minimum of one additional line of evidence will be utilized as appropriate or suitable to the conditions. Examples of additional lines of evidence include but are not limited to:

- Private utility locating service
- Research of state, county or municipal utility records and maps including computer drawn maps or geographical information systems (GIS)
- Contact with the utility provider to obtain their utility location records
- Hand augering or digging
- Hydro-knife
- Air-knife

- Radio Frequency Detector (RFD)
- Ground Penetrating Radar (GPR)
- Any other method that may give ample evidence of the presence or location of subgrade utilities.

Overhead power lines also present risks and the following safe clearance must be maintained from them.

Power Line Voltage Phase to Phase (kV)	**Minimum Safe Clearance (feet)**
50 or below	10
Above 50 to 200	15
Above 200 to 350	20
Above 350 to 500	25
Above 500 to 750	35
Above 750 to 1,000	35

ANSI Standard B30.5-1994, 5-3.4.5

Avoid using drilling fluids or materials that could impact groundwater or soil quality, or could be incompatible with the subsurface conditions.

Water used for drilling and sampling of soil or bedrock, decontamination of drilling/sampling equipment, or grouting boreholes upon completion will be of a quality acceptable for project objectives. Testing of water supply should be considered.

Specifications of materials used for backfilling borehole will be obtained, reviewed and approved to meet project quality objectives.

V. Health and Safety Considerations

Field activities associated with overburden drilling and soil sampling will be performed in accordance with a site-specific HASP, a copy of which will be present on site during such activities.

VI. Procedure

Drilling Procedures

The drilling contractor will be responsible for obtaining accurate and representative samples; informing the supervising geologist of changes in drilling pressure; and keeping a separate general log of soils encountered, including blow counts (i.e., the number of blows from a soil sampling drive weight [140 pounds] required to drive the split-barrel sampler in 6-inch increments). Records will also be kept of occurrences of premature refusal due to boulders or construction materials that may have been used as fill. Where a boring cannot be advanced to the desired depth, the boring will be abandoned and an additional boring will be advanced at an adjacent location to obtain the required sample. Where it is desirable to avoid leaving vertical connections between depth intervals, the borehole will be sealed using cement and/or bentonite. Multiple refusals may lead to a decision by the supervising geologist to abandon that sampling location.

Soil Sampling Procedures

Samples of subsurface materials encountered while drilling soil borings will be collected using one of the following methods:

- 2-inch split-barrel (split-spoon) sampler, if using the ASTM D 1586 - Standard Test Method for Penetration Test and Split-Barrel Sampling of Soils

- Plastic internal soil sample sleeves if using direct-push drilling.

Soil samples are typically field screened with an FID or PID at sites where volatile organic compounds are present in the subsurface. Field screening is performed using one of the following methods:

- Upon opening the sampler, the soil is split open and the PID or FID probe is placed in the opening and covered with a gloved hand. Such readings should be obtained at several locations along the length of the sample

- A portion of the collected sample is placed in a jar, which is covered with aluminum foil, sealed, and allowed to warm to room temperature. After warming, the cover is removed, the foil is pieced with the FID or PID probe, and a reading is obtained.

Samples selected for laboratory analysis will be handled, packed, and shipped in accordance with the procedures outlined in the Work Plan, FSP, or Chain-of-Custody, Handling, Packing, and Shipping SOP.

A geologist will be onsite during drilling and sampling operations to describe each soil sample on the soil boring log, including:

- percent recovery;
- structure and degree of sample disturbance;
- soil type;
- color;
- moisture condition;
- density;
- grain-size;
- consistency; and
- other observations, particularly relating to the presence of waste materials

Further details regarding geologic description of soil samples are presented in the Soil Description SOP.

Particular care will be taken to fully describe any sheens observed, oil saturation, staining, discoloration, evidence of chemical impacts, or unnatural materials.

VII. Waste Management

Water generated during cleaning procedures will be collected and contained onsite in appropriate containers for future analysis and appropriate disposal.

PPE (such as gloves, disposable clothing, and other disposable equipment) resulting from personnel cleaning procedures and soil sampling/handling activities will be placed in plastic bags. These bags will be transferred into appropriately labeled 55-gallon drums or a covered roll-off box for appropriate disposal.

Soil materials will be placed in sealed 55-gallon steel drums or covered roll-off boxes and stored in a secured area. Once full, the material will be analyzed to determine the appropriate disposal method.

VIII. Data Recording and Management

The supervising geologist or scientist will be responsible for documenting drilling events using a bound field notebook and/or PDA to record all relevant information in a clear and concise format. The record of drilling events will include:

- start and finish dates of drilling;
- name and location of project;
- project number, client, and site location;
- sample number and depths;
- blow counts and recovery;
- depth to water;
- type of drilling method;
- drilling equipment specifications, including the diameter of drilling tools;
- documentation of any elevated organic vapor readings;
- names of drillers, inspectors, or other people onsite; and
- weather conditions.

IX. Quality Assurance

Equipment will be cleaned prior to use onsite, between each drilling location, and prior to leaving the site. Drilling equipment and associated tools, including augers, drill rods, sampling equipment, wrenches, and other equipment or tools that may have come in contact with soils and/or waste materials will be cleaned with high-pressure steam-cleaning equipment using a potable water source. The drilling equipment will be cleaned in an area designated by the supervising engineer or geologist that is located outside of the work zone. More elaborate cleaning procedures may be required for reusable soil samplers (split-spoons) when soil samples are obtained for laboratory analysis of chemical constituents.

X. References

American Society of Testing and Materials (ASTM) D 1586 - *Standard Test Method for Penetration Test and Split-Barrel Sampling of Soils*.

Attachment 3D
Field Log Book Entries

ARCADIS
Infrastructure, environment, facilities

Imagine the result

Field Log Book Entries

Rev. #: 0

Rev Date: 11 August 2009

Approval Signatures

Prepared by: Andrew Karnik Date: 8/11/09

Reviewed by: Michael J Gefell Date: 8/11/09
(Technical Expert)

I. Scope and Application

This ARCADIS Standard Operating Procedure covers the entries needed in a field log book for environmental investigations.

This SOP does not address all of the entries that may be needed for a specific project, and does not address health and safety, equipment decontamination, field parameter measurements, sample preservation, chain-of-custody, or laboratory analysis. For direction on requirements in these areas, refer to other ARCADIS SOPs, the project work plans including the quality assurance project plan, sampling plan, and health and safety plan, as appropriate.

II. Personnel Qualifications

ARCADIS personnel participating in fieldwork and making entries into the field log book should have a minimum of one (1) year of field experience (or be under the supervision and accompanied in the field by someone who does) and current health and safety training including 40-hour HAZWOPER training, site supervisor training, site-specific training, first aid, and CPR, as needed. Field personnel will also be compliant with client-specific training requirements. In addition, ARCADIS field sampling personnel will be versed in the relevant SOPs and posses the required skills and experience necessary to successfully complete the desired field work.

III. Equipment List

- Field Log Book
- Ball point (medium point) pen with blue or black ink (black preferred). A fine point Sharpie pen may be used if the ink does not bleed through the page and become visible on back side of the page. If weather conditions prevent the use of a pen, indicate so in the log and use an alternate writing instrument .
- Zip-lock baggie or other weather-proof container to protect the field log book from the elements.

IV. Cautions

All entries in the field log must be legible and archivable. Do not leave the field log book exposed to the elements or other conditions that might moisten the pages and smear/dissolve the entries. When not in the field, the log book should be stored in a location that is easily accessible to field crews.

V. Health and Safety Considerations

ARCADIS field personnel will be familiar and compliant with Client-specific health and safety requirements.

VI. Procedure

- Print legibly. Do not use cursive writing.

- The name of the project, project number and project location should be written in indelible ink on the outside of the field log book.

- On the inside of the front cover, write “If Found, Please Return to ARCADIS” and include the appropriate address and phone number, the name of the person to which the book is assigned, and the name of the project manager.

- Reserve the first page of the book for a Table of Contents.

- Reserve the last five (5) pages of the book for important contacts, notes, reminders, etc.

- Each day of field work, the following should be recorded in the field log book as applicable:

  a) Project Name

  b) Date and time arrived

  c) Work Site Location

  d) Names of people on-site related to the project including ARCADIS employees, visitors, subcontractor employees, agency personnel, client representative, etc.

  e) Describe the work to be performed briefly, and list the equipment on-site

  f) Indicate the health and safety (H&S) level to be used

  g) Record instrument calibrations and checks

  h) Record time and general content of H&S briefing

  i) Describe the weather conditions, including temperature, precipitation, and wind speed and direction

  j) List periodic time entries in the far left hand column of each page

  k) Minimize unused space on each page

- The tailgate meeting must be recorded in the log book and the tailgate form completed. If H&S monitoring is performed, record the time and results of initial and followup monitoring.

- Note factual observations including collection of QA/QC samples, delays, well damage, accidents, work plan deviations, instrument problems, and problem resolutions.

- Describe work performed and how documented such as photographs, sample core logs, water sampling logs, etc.

- Describe bases for field decisions including pertinent conversations with visitors, regulators, or project personnel.

- Note final instrument calibrations and checks.

- Sign the log book at the end of each day at a minimum. Draw a line to the end of the page to indicate no further entries on that page. Sign the bottom of each page if possible.

- If an entry to the log book is changed, strike out the deleted text or item with a single line such that the entry remains legible, and initial and date the change. Such changes should only be made by the same person that made the initial entry.

- Field log book entries must be made in the field at the site, not at a later time at a different location. Supplemental entries to the log book may be made at a later date. The supplemental entry must be clearly identified as such and the entry must be signed and dated as described in this SOP.

- Problems noted in the field log book must be brought to the attention of the project manager and task manager in a timely fashion. Problems may be reported in person, on the telephone, or in a written daily log form. If daily logs are prepared and you will not be able to personally give the daily log to the project manager, send the daily log via FAX or overnight courier to the project manager and task manager.

VII. Waste Management

Investigation-derived waste will be managed as described in the Investigation-Derived Waste Handling and Storage SOP. A drum/waste inventory should be maintained on a pre-designated page in the field log book.

VIII. Data Recording and Management

Each page of the field log book should be scanned for electronic/digital archiving at periodic intervals. This will ensure that copies of the field notes are available in the event the field book is lost or damaged, and that field data can be easily disseminated to others without the risk of physically sending the field log book. Field log books that are full should be archived with the project files, and readily retrievable.

IX. Quality Assurance

Be mindful that the field log book may be produced in court. All entries should be legible (as discussed above). Entries should also be in English, unless working in a country where English is not the predominant language or you are directed otherwise by the project manager.

X. References

Not Applicable

Attachment 3E
Low-Flow Groundwater Purging and Sampling Procedures for Monitoring Wells

Low-Flow Groundwater Purging and Sampling Procedures for Monitoring Wells

Rev. #: 3

Rev Date: March 9, 2009

Approval Signatures

Prepared by: David S. Lipson Date: 3/9/2009

Reviewed by: Michael J Gefell Date: 3/9/2009
(Technical Expert)

I. Scope and Application

Groundwater samples will be collected from monitoring wells to evaluate groundwater quality. The protocol presented in this standard operating procedure (SOP) describes the procedures to be used to purge monitoring wells and collect groundwater samples. This protocol has been developed in accordance with the United States Environmental Protection Agency (USEPA) Region I Low Stress (Low Flow) Purging and Sampling Procedures for the Collection of Groundwater Samples from Monitoring Wells (USEPA SOP No. GW0001; July 30, 1996). Both filtered and unfiltered groundwater samples may be collected using this low-flow sampling method. Filtered samples will be obtained using a 0.45-micron disposable filter. No wells will be sampled until well development has been performed in accordance with the procedures presented in the SOP titled Monitoring Well Development, unless that well has been sampled or developed within the prior 1-year time period. Groundwater samples will not be collected within 1 week following well development.

II. Personnel Qualifications

ARCADIS personnel directing, supervising, or leading groundwater sample collection activities should have a minimum of 2 years of previous groundwater sampling experience. ARCADIS personnel providing assistance to groundwater sample collection and associated activities should have a minimum of 6 months of related experience or an advanced degree in environmental sciences, engineering, hydrogeology, or geology.

The supervisor of the groundwater sampling team will have at least 1 year of previous supervised groundwater sampling experience.

Prior to mobilizing to the field, the groundwater sampling team should review and be thoroughly familiar with relevant site-specific documents including but not limited to the site work plan, field sampling plan, QAPP, HASP, and historical information. Additionally, the groundwater sampling team should review and be thoroughly familiar with documentation provided by equipment manufacturers for all equipment that will be used in the field prior to mobilization.

III. Equipment List

Specific to this activity, the following materials (or equivalent) will be available:

- Health and safety equipment (as required in the site Health and Safety Plan [HASP]).

- Site Plan, well construction records, prior groundwater sampling records (if available).
- Sampling pump, which may consist of one or more of the following:
  - submersible pump (e.g., Grundfos Redi-Flo 2);
  - peristaltic pump (e.g., ISCO Model 150); and/or
  - bladder pump (e.g., Marschalk System 1, QED Well Wizard, etc.).
- Appropriate controller and power source for pump:
  - Submersible and peristaltic pumps require electric power from either a generator or a deep cell battery.
  - Submersible pumps such as Grundfos require a pump controller to run the pump
  - Bladder pumps require a pump controller and a gas source (e.g., air compressor or compressed N_2 or CO_2 gas cylinders).
- Teflon® tubing or Teflon®-lined polyethylene tubing of an appropriate size for the pump being used. For peristaltic pumps, dedicated Tygon® tubing (or other type as specified by the manufacturer) will also be used through the pump apparatus.
- Water-level probe (e.g., Solinist Model 101).
- Water-quality (temperature/pH/specific conductivity/ORP/turbidity/dissolved oxygen) meter and flow-through measurement cell. Several brands may be used, including:
  - YSI 6-Series Multi-Parameter Instrument;
  - Hydrolab Series 3 or Series 4a Multiprobe and Display; and/or
  - Horiba U-10 or U-22 Water Quality Monitoring System.
- Supplemental turbidity meter (e.g., Horiba U-10, Hach 2100P, LaMotte 2020). Turbidity measurements collected with multi-parameter meters have been shown to sometimes be unreliable due to fouling of the optic lens of the

turbidity meter within the flow-through cell. A supplemental turbidity meter will be used to verify turbidity data during purging if such fouling is suspected. Note that industry improvements may eliminate the need for these supplemental measurements in the future.

- Appropriate water sample containers (supplied by the laboratory).
- Appropriate blanks (trip blank supplied by the laboratory).
- 0.45-micron disposable filters (if field filtering is required).
- Large glass mixing container (if sampling with a bailer).
- Teflon® stirring rod (if sampling with a bailer).
- Cleaning equipment.
- Groundwater sampling log (attached) or bound field logbook.

Note that in the future, the client may acquire different makes/models of some of this equipment if the listed makes/models are no longer available, or as a result of general upgrades or additional equipment acquisitions. In the event that the client uses a different make/model of the equipment listed, the client will use an equivalent type of equipment (e.g., pumps, flow-through analytical cells) and note the specific make/model of the equipment used during a sampling event on the groundwater sampling log. In addition, should the client desire to change to a markedly different sampling methodology (e.g., discrete interval samplers, passive diffusion bags, or a yet to be developed technique), the client will submit a proposed SOP for the new methodology for USEPA approval prior to implementing such a change.

The maintenance requirements for the above equipment generally involve decontamination or periodic cleaning, battery charging, and proper storage, as specified by the manufacturer. For operational difficulties, the equipment will be serviced by a qualified technician.

IV. Cautions

If heavy precipitation occurs and no cover over the sampling area and monitoring well can be erected, sampling must be discontinued until adequate cover is provided. Rain water could contaminate groundwater samples.

Do not use permanent marker or felt-tip pens for labels on sample container or sample coolers – use indelible ink. The permanent markers could introduce volatile constituents into the samples.

It may be necessary to field filter some parameters (e.g., metals) prior to collection, depending on preservation, analytical method, and project quality objectives.

Store and/or stage empty and full sample containers and coolers out of direct sunlight.

To mitigate potential cross-contamination, groundwater samples are to be collected in a pre-determined order from least impacted to impacted based on previous analytical data. If no analytical data are available, samples are collected in order of upgradient, then furthest downgradient to source area locations.

Be careful not to over-tighten lids with Teflon liners or septa (e.g., 40 mL vials). Over-tightening can cause the glass to shatter or impair the integrity of the Teflon seal.

V. Health and Safety Considerations

Use caution and appropriate cut resistant gloves when tightening lids to 40 mL vials. These vials can break while tightening and can lacerate hand. Amber vials (thinner glass) are more prone to breakage.

If thunder or lighting is present, discontinue sampling and take cover until 30 minutes have passed after the last occurrence of thunder or lighting.

Use caution when removing well caps as well may be under pressure, cap can dislodge forcefully and cause injury.

Use caution when opening protective casing on stickup wells as wasps frequently nest inside the tops of the covers. Also watch for fire ant mounds near well pads when sampling in the south or western U.S.

VI. Procedure

Groundwater will be purged from the wells using an appropriate pump. Peristaltic pumps will initially be used to purge and sample all wells when applicable. If the depth to water is below the sampling range of a peristaltic pump (approximately 25 feet), submersible pumps or bladder pumps will be used provided the well is constructed with a casing diameter greater than or equal to 2 inches (the minimum well diameter capable of accommodating such pumps). Bladder pumps are preferred over peristaltic and submersible pumps if sampling of VOCs is required to prevent volatilization. For smaller diameter wells where the depth to water is below the sampling range of a peristaltic pump, alternative sampling methods (i.e., bailing or small diameter bladder pumps) will be used to purge and sample the groundwater. Purge water will be collected and containerized.

1. Calibrate field instruments according to manufacturer procedures for calibration.

2. Measure initial depth to groundwater prior to placement of pumps.

3. Prepare and install pump in well: For submersible and non-dedicated bladder pumps, decontaminate pump according to site decontamination procedures. Non-dedicated bladder pumps will require a new Teflon® bladder and attachment of an air line, sample discharge line, and safety cable prior to placement in the well. Attach the air line tubing to the air port on the top of the bladder pump. Attach the sample discharge tubing to the water port on the top of the bladder pump. Care should be taken not to reverse the air and discharge tubing lines during bladder pump set-up as this could result in bladder failure or rupture. Attach and secure a safety cable to the eyebolt on the top of bladder pump (if present, depending on pump model used). Slowly lower pump, safety cable, tubing, and electrical lines into the well to a depth corresponding to the approximate center of the saturated screen section of the well. Take care to avoid twisting and tangling of safety cable, tubing, and electrical lines while lowering pump into well; twisted and tangled lines could result in the pump becoming stuck in the well casing. Also, make sure to keep tubing and lines from touching the ground or other surfaces while introducing them into the well as this could lead to well contamination. If a peristaltic pump is being used, slowly lower the sampling tubing into the well to a depth corresponding to the approximate center of the saturated screen section of the well. The pump intake or sampling tube must be kept at least 2 feet above the bottom of the well to prevent mobilization of any sediment present in the bottom of the well.

4. Connect the pump to other equipment. If using a bladder pump, the discharge water line should be connected to the bottom inlet port on the flow-through cell connected to the water quality meter. Connect the air line to the pump controller output port. The pump controller should then be connected to a supply line from an air compressor or compressed gas cylinder using an appropriate regulator and air hose. Take care to tighten the regulator connector onto the gas cylinder (if used) to prevent leaks. Teflon tape may be used on the threads of the cylinder to provide a tighter seal. Once the air compressor or gas cylinder is connected to the pump controller, turn on the compressor or open the valve on the cylinder to begin the gas flow. Turn on the pump controller if an on/off switch

is present and verify that all batteries are charged and fully operating before beginning to pump.

5. Measure the water level again with the pump in the well before starting the pump. Start pumping the well at 200 to 500 milliliters (mL) per minute (or at lower site-specific rate if specified). The pump rate should be adjusted to cause little or no water level drawdown in the well (less than 0.3 feet below the initial static depth to water measurement) and the water level should stabilize. The water level should be monitored every 3 to 5 minutes (or as appropriate, lower flow rates may require longer time between readings) during pumping if the well diameter is of sufficient size to allow such monitoring. Care should be taken not to break pump suction or cause entrainment of air in the sample. Record pumping rate adjustments and depths to water. If necessary, pumping rates should be reduced to the minimum capabilities of the pump to avoid pumping the well dry and/or to stabilize indicator parameters. A steady flow rate should be maintained to the extent practicable. Groundwater sampling records from previous sampling events (if available) should be reviewed prior to mobilization to estimate the optimum pumping rate and anticipated drawdown for the well in order to more efficiently reach a stabilized pumping condition.

   If the recharge rate of the well is very low, alternative purging techniques should be used, which will vary based on the well construction and screen position. For wells screened across the water table, the well should be pumped dry and sampling should commence as soon as the volume in the well has recovered sufficiently to permit collection of samples. For wells screened entirely below the water table, the well should be pumped until a stabilized level (which may be below the maximum displacement goal of 0.3 feet) can be maintained and monitoring for stabilization of field indicator parameters can commence. If a lower stabilization level cannot be maintained, the well should be pumped until the drawdown is at a level slightly higher than the bentonite seal above the well screen. Sampling should commence after one well volume has been removed and the well has recovered sufficiently to permit collection of samples.

   During purging, monitor the field indicator parameters (e.g., turbidity, temperature, specific conductance, pH, etc.) every 3 to 5 minutes (or as appropriate). Field indicator parameters will be measured using a flow-through analytical cell or a clean container such as a glass beaker. Record field indicator parameters on the groundwater sampling log. The well is considered stabilized and ready for sample collection when turbidity values remain within 10% (or within 1 NTU if the turbidity reading is less than 10 NTU), the specific conductance and temperature values remain within 3%, and pH remains within 0.1 units for three consecutive readings collected at 3- to 5-minute intervals (or other appropriate interval, alternate stabilization goals may exist in different geographic regions, consult the site-specific Work Plan for stabilization criteria). If the field indicator parameters do not stabilize within 1 hour of the start of purging, but the groundwater turbidity is below the goal of 50 NTU and the values for all other parameters are within 10%, the well can be sampled. If the parameters have stabilized but the turbidity is not in the range of the 50 NTU goal, the pump flow rate should be decreased to a minimum rate of 100 mL/min to reduce turbidity levels as low as possible. If dissolved oxygen values are not within acceptable range for the temperature of groundwater (Attachment 1), then check for and remove air bubbles on probe or in tubing. If the dissolved oxygen value is 0.00 or less, then the meter should be serviced and re-calibrated.

During extreme weather conditions, stabilization of field indicator parameters may be difficult to obtain. Modifications to the sampling procedures to alleviate these conditions (e.g., measuring the water temperature in the well adjacent to the pump intake) will be documented in the field notes. If other field conditions exist that preclude stabilization of certain parameters, an explanation of why the parameters did not stabilize will also be documented in the field logbook.

6. Complete the sample label and cover the label with clear packing tape to secure the label onto the container.

7. After the indicator parameters have stabilized, collect groundwater samples by diverting flow out of the unfiltered discharge tubing into the appropriate labeled sample container. If a flow-through analytical cell is being used to measure field parameters, the flow-through cell should be disconnected after stabilization of the field indicator parameters and prior to groundwater sample collection. Under no circumstances should analytical samples be collected from the discharge of the flow-through cell. When the container is full, tightly screw on the cap. Samples should be collected in the following order: VOCs, TOC, SVOCs, metals and cyanide, and others (or other order as defined in the site-specific Work Plan).

8. If sampling for total and filtered metals and/or PCBs, a filtered and unfiltered sample will be collected. Install an in-line, disposable 0.45-micron particle filter on the discharge tubing after the appropriate unfiltered groundwater sample has been collected. Continue to run the pump until an initial volume of “flush” water has been run through the filter in accordance with the manufacturer’s directions (generally 100 to 300 mL). Collect filtered groundwater sample by diverting flow out of the filter into the appropriately labeled sample container. When the container is full, tightly screw on the cap.

9. Secure with packing material and store at 4°C in an insulated transport container provided by the laboratory.

10. Record on the groundwater sampling log or bound field logbook the time sampling procedures were completed, any pertinent observations of the sample (e.g., physical appearance, and the presence or lack of odors or sheens), and the values of the stabilized field indicator parameters as measured during the final reading during purging (Attachment 2 – Example Sampling Log).

11. Turn off the pump and air compressor or close the gas cylinder valve if using a bladder pump set-up. Slowly remove the pump, tubing, lines, and safety cable from the well. Do not allow the tubing or lines to touch the ground or any other surfaces which could contaminate them. .

12. If tubing is to be dedicated to a well, it should be folded to a length that will allow the well to be capped and also facilitate retrieval of the tubing during later sampling events. A length of rope or string should be used to tie the tubing to the well cap. Alternatively, if tubing and safety line are to be saved and reused for sampling the well at a later date they may be coiled neatly and placed in a clean plastic bag that is clearly labeled with the well ID. Make sure the bag is tightly sealed before placing it in storage.

13. Secure the well and properly dispose of personal protective equipment (PPE) and disposable equipment.

14. Complete the procedures for packaging, shipping, and handling with associated chain-of-custody.

15. Complete decontamination procedures for flow-through analytical cell and submersible or bladder pump, as appropriate.

16. At the end of the day, perform calibration check of field instruments.

If it is not technically feasible to use the low-flow sampling method, purging and sampling of monitoring wells may be conducted using the bailer method as outlined below:

1. Don appropriate PPE (as required by the HASP).

2. Place plastic sheeting around the well.

3. Clean sampling equipment.

4. Open the well cover while standing upwind of the well. Remove well cap and place on the plastic sheeting. Insert PID probe approximately 4 to 6 inches into the casing or the well headspace and cover with gloved hand. Record the PID reading in the field log. If the well headspace reading is less than 5 PID units, proceed; if the headspace reading is greater than 5 PID units, screen the air within the breathing zone. If the breathing zone reading is less than 5 PID units, proceed. If the PID reading in the breathing zone is above 5 PID units, move upwind from well for 5 minutes to allow the volatiles to dissipate. Repeat the breathing zone test. If the reading is still above 5 PID units, don appropriate respiratory protection in accordance with the requirements of the HASP. Record all PID readings. For wells that are part of the regular weekly monitoring program and prior PID measurements have not resulted in a breathing zone reading above 5 PID units, PID measurements will be taken monthly.

5. Measure the depth to water and determine depth of well by examining drilling log data or by direct measurement. Calculate the volume of water in the well (in gallons) by using the length of the water column (in feet), multiplying by 0.163 for a 2-inch well or by 0.653 for a 4-inch well. For other well diameters, use the formula:

   Volume (in gallons) = • TIMES well radius (in feet) squared TIMES length of water column (in feet) TIMES 7.481 (gallons per cubic foot)

6. Measure a length of rope or twine at least 10 feet greater than the total depth of the well. Secure one end of the rope to the well casing and secure the other end to the bailer. Test the knots and make sure the rope will not loosen. Check bailers so that all parts are intact and will not be lost in the well.

7. Lower bailer into well and remove one well volume of water. Contain all water in appropriate containers.

8. Monitor the field indicator parameters (e.g., turbidity, temperature, specific conductance, and pH). Measure field indicator parameters using a clean container such as a glass beaker or sampling cups provided with the instrument. Record field indicator parameters on the groundwater sampling log.

9. Repeat Steps 7 and 8 until three or four well volumes have been removed. Examine the field indicator parameter data to determine if the parameters have stabilized. The well is considered stabilized and ready for sample collection when turbidity values remain within 10% (or within 1 NTU if the turbidity reading is less than 10 NTU), the specific conductance and temperature values remain

within 3%, and pH remains within 0.1 units for three consecutive readings collected once per well volume removed.

10. If the field indicator parameters have not stabilized, remove a maximum of five well volumes prior to sample collection. Alternatively, five well volumes may be removed without measuring the field indicator parameters.

11. If the recharge rate of the well is very low, wells screened across the water table may be bailed dry and sampling should commence as soon as the volume in the well has recovered sufficiently to permit collection of samples. For wells screened entirely below the water table, the well should only be bailed down to a level slightly higher than the bentonite seal above the well screen. The well should not be bailed completely dry, to maintain the integrity of the seal. Sampling should commence as soon as the well volume has recovered sufficiently to permit sample collection.

12. Following purging, allow water level in well to recharge to a sufficient level to permit sample collection.

13. Complete the sample label and cover the label with clear packing tape to secure the label onto the container.

14. Slowly lower the bailer into the screened portion of the well and carefully retrieve a filled bailer from the well causing minimal disturbance to the water and any sediment in the well.

15. The sample collection order (as appropriate) will be as follows:

    a. VOCs;

    b TOC;

    c. SVOCs;

    d. metals and cyanide; and

    e. others.

16. When sampling for volatiles, collect water samples directly from the bailer into 40-mL vials with Teflon®-lined septa.

17. For other analytical samples, remove the cap from the large glass mixing container and slowly empty the bailer into the large glass mixing container. The sample for dissolved metals and/or filtered PCBs should either be placed directly from the bailer into a pressure filter apparatus or pumped directly from the bailer with a peristaltic pump, through an in-line filter, into the pre-preserved sample bottle.

18. Continue collecting samples until the mixing container contains a sufficient volume for all laboratory samples.

19. Mix the entire sample volume with the Teflon® stirring rod and transfer the appropriate volume into the laboratory jar(s). Secure the sample jar cap(s) tightly.

20. If sampling for total and filtered metals and/or PCBs, a filtered and unfiltered sample will be collected. Sample filtration for the filtered sample will be performed in the field using a peristaltic pump prior to preservation. Install new medical-grade silicone tubing in the pump head. Place new Teflon® tubing into the sample mixing container and attach to the intake side of pump tubing. Attach (clamp) a new 0.45-micron filter (note the filter flow direction). Turn the pump on and dispense the filtered liquid directly into the laboratory sample bottles.

21. Secure with packing material and store at 4°C in an insulated transport container provided by the laboratory.

22. After sample containers have been filled, remove one additional volume of groundwater. Measure the pH, temperature, turbidity, and conductivity. Record on the groundwater sampling log or bound field logbook the time sampling procedures were completed, any pertinent observations of the sample (e.g., physical appearance, and the presence or lack of odors or sheens), and the values of the field indicator parameters.

23. Remove bailer from well, secure well, and properly dispose of PPE and disposable equipment.

24. If a bailer is to be dedicated to a well, it should be secured inside the well above the water table, if possible. Dedicated bailers should be tied to the well cap so that inadvertent loss of the bailer will not occur when the well is opened.

25. Complete the procedures for packaging, shipping, and handling with associated chain-of-custody.

VII. Waste Management

Materials generated during groundwater sampling activities, including disposable equipment, will be placed in appropriate containers. Containerized waste will be disposed of by the client consistent with the procedures identified in the HASP.

VIII. Data Recording and Management

Initial field logs and chain-of-custody records will be transmitted to the ARCADIS PM at the end of each day unless otherwise directed by the PM. The groundwater team leader retains copies of the groundwater sampling logs.

IX. Quality Assurance

In addition to the quality control samples to be collected in accordance with this SOP, the following quality control procedures should be observed in the field:

- Collect samples from monitoring wells in order of increasing concentration, to the extent known based on review of historical site information if available.

- Equipment blanks should include the pump and tubing (if using disposable tubing) or the pump only (if using tubing dedicated to each well).

- Collect equipment blanks after wells with higher concentrations (if known) have been sampled.

- Operate all monitoring instrumentation in accordance with manufacturer’s instructions and calibration procedures. Calibrate instruments at the beginning of each day and verify the calibration at the end of each day. Record all calibration activities in the field notebook.

- Clean all groundwater sampling equipment prior to use in the first well and after each subsequent well using procedures for equipment decontamination.

X. References

United States Environmental Protection Agency (USEPA). 1986. RCRA Groundwater Monitoring Technical Enforcement Guidance Document (September 1986).

USEPA Region II. 1998. *Ground Water Sampling Procedure Low Stress (Low Flow) Purging and Sampling*.

USEPA. 1991. Handbook Groundwater, Volume II Methodology, Office of Research and Development, Washington, DC. USEPN62S, /6-90/016b (July, 1991).

U.S. Geological Survey (USGS). 1977. National Handbook of Recommended Methods for Water-Data Acquisition: USGS Office of Water Data Coordination. Reston, Virginia.

Attachment 1

Groundwater Sampling Log

Page _____ of _____

ARCADIS

Infrastructure, environment, facilities

Low-Flow Groundwater Sampling Log

Project ______________________

Project Number ______________ **Site Location** ______________ **Well ID** ______________

Date ______________ **Sampled By** ______________

Sampling Time ______________ **Recorded By** ______________

Weather ______________ **Coded Replicate No.** ______________

Instrument Identification

Water Quality Meter(s) ______________ **Serial #** ______________

Casing Material ______________ **Purge Method** ______________

Casing Diameter ______________ **Screen Interval (ft bmp)** **Top** ______________ **Bottom** ______________

Sounded Depth (ft bmp) ______________ **Pump Intake Depth (ft bmp)** ______________ ______________

Depth to Water (ft bmp) ______________ **Purge Time** **Start** ______________ **Finish** ______________

Field Parameter Measurements During Purging

Time	Minutes Elasped	Flow Rate (mL/min)	Volume Purged	Temp (°C)	pH (s.u.)	Conductivity (umhos or mS/cm) 1)	ORP (mV)	DO (mg/L)	Turbidity (NTU)	Depth to Water (ft bmp)

Collected Sample Condition **Color**__________________ **Odor**__________________ **Appearance**__________________

Parameter	Container	No.	Preservative

PID Reading ______________

Comments ______________________

____

____

____

1) Circle one unit type

C:\Documents and Settings\ljohnson2\Local Settings\Temporary Internet Files\Content.Outlook\9VNLKRXU\lowflowsampform.xls - Sheet1

Attachment 2

Oxygen Solubility in Fresh Water

Temperature (degrees C)	**Dissolved Oxygen** (mg/L)
0	14.6
1	14.19
2	13.81
3	13.44
4	13.09
5	12.75
6	12.43
7	12.12
8	11.83
9	11.55
10	11.27
11	11.01
12	10.76
13	10.52
14	10.29
15	10.07
16	9.85
17	9.65
18	9.45
19	9.26
20	9.07
21	8.9
22	8.72
23	8.56
24	8.4
25	8.24
26	8.09
27	7.95
28	7.81
29	7.67
30	7.54
31	7.41
32	7.28
33	7.16
34	7.05
35	6.93

Reference: Vesilind, P.A., *Introduction to Environmental Engineering*, PWS Publishing Company, Boston, 468 pages (1996).

Attachment 3F
Investigation-Derived Waste Handling and Storage

ARCADIS
Infrastructure, environment, buildings

Imagine the result

Investigation-Derived Waste Handling and Storage

Rev. #: 2

Rev Date: March 6, 2009

Approval Signatures

Prepared by: Andrew Karnik Date: 3/6/09

Reviewed by: Liz Marsh Date: 3/6/09
(Technical Expert)

I. Scope and Application

The objective of this Standard Operating Procedure (SOP) is to describe the procedures to manage investigation-derived wastes (IDW), both hazardous and non-hazardous, generated during site activities, which may include, but are not limited to - drilling, trenching/excavation, construction, demolition, monitoring well sampling, soil sampling, decontamination and remediation. Please note that this SOP is intended for materials that have been deemed a solid waste as defined by 40 CFR § 261.2 (which may includes liquids, solids, and sludges). In some cases, field determinations will be made based on field screening or previous data that materials are not considered a solid waste. IDW may include soil, groundwater, drilling fluids, decontamination liquids, personal protective equipment (PPE), sorbent materials, construction and demolition debris, and disposable sampling materials that may have come in contact with potentially impacted materials. IDW will be collected and staged at the point of generation. Quantities small enough to be containerized in 55-gallon drums will be taken to a designated temporary storage area (discussed in further detail under Drum Storage) onsite pending characterization and disposal. Waste materials will be analyzed for constituents of concern to evaluate proper disposal methods. PPE and disposable sampling equipment will be placed in DOT-approved drums prior to disposal and typically does not require laboratory analysis. This SOP describes the necessary equipment, field procedures, materials, regulatory references, and documentation procedures necessary for proper handling and storage of IDW up to the time it is properly disposed. The procedures for handling IDW are based on the United States Environmental Protection Agency's Guide to Management of Investigation Derived Wastes (USEPA, 1992). IDW is assumed to be contaminated with the site constituents of concern (COCs) until analytical evidence indicates otherwise. IDW will be managed to ensure the protection of human health and the environment and will comply with all applicable or relevant and appropriate requirements (ARAR). The following Laws and Regulations on Hazardous Waste Management are potential ARAR for this site.

State Laws and Regulations

- To Be Determined Based on Location of Site and Location of Treatment, Storage, and/or Disposal Facility (TSDF) to be utilized

Federal Laws and Regulations

- Resource Conservation and Recovery Act (RCRA) 42 USC § 6901-6987
- Comprehensive Environmental Response, Compensation and Liability Act (CERCLA) 42 USC § 9601-9675

- Superfund Amendments and Reauthorization Act (SARA)
- Department of Transportation (DOT) Hazardous Materials Transportation

Pending characterization, IDW will be stored appropriately within each area of contamination (AOC). Under RCRA, “storage” is defined as the holding of hazardous waste for a temporary period, at the end of which the hazardous waste is treated, disposed of, or stored elsewhere” (40 CFR § 260.10). The onsite waste staging area will be in a secure and controlled area. Waste characterization can either be based on generator knowledge, such as using materials safety data sheets (MSDS’), or can be based upon analytical results. The laboratory used for waste characterization analysis must have the appropriate state and federal certifications and be approved by ARCADIS and Client. IDW will be classified as RCRA hazardous or non-regulated under RCRA based on the waste characterization.

If IDW is characterized as RCRA hazardous waste, RCRA and DOT requirements must be followed for packaging, labeling, transporting, storing, and record keeping as described in 40 CFR § 262 and 49 CFR § 171-178. Wastes judged to potentially meet the criteria for hazardous wastes shall be stored in DOT approved packaging. Waste material classified as RCRA non-hazardous may be handled and disposed of as an industrial waste.

Liquid wastes judged to potentially meet the criteria for hazardous wastes shall be stored in DOT approved 55 gallon drums or other approved containers that are compatible with the type of material stored therein. Solid materials deemed to potentially meet hazardous criteria will be drummed where practicable. Large quantities of potentially hazardous solid materials must be containerized (such as in a roll-off box) for up to a maximum of 90 or 180 days as described in the Excavated Solids Section. Waste material classified as non-hazardous may be handled and disposed of as an industrial waste and is not subject to the 90-day or 180-day on-site storage limitation.

This is a standard (i.e., typically applicable) operating procedure which may be varied or changed as required, dependent upon site conditions, equipment limitations, or limitations imposed by the procedure. The ultimate procedure employed will be documented in the project work plans or reports. If changes to the sampling procedures are required due to unanticipated field conditions, the changes will be discussed with the Project Manager and Client as soon as practicable and documented in the report.

II. Personnel Qualifications

ARCADIS field sampling personnel will have current health and safety training including 40-hour HAZWOPER training, site supervisor training, site-specific training, first aid, and CPR, as needed. ARCADIS personnel may sign manifests on a case-to-case basis for clients, provided the appropriate agreement is in place between ARCADIS and the client documenting that ARCADIS is not the generator, but is acting as authorized representative for the generator. ARCADIS personnel who sign hazardous waste manifests will have the current DOT hazardous materials transportation training according to 49 CFR § 172.704. ARCADIS field personnel will also comply with client-specific training such as LPS. In addition, ARCADIS field sampling personnel will be versed in the relevant SOPs and posses the required skills and experience necessary to successfully complete the desired field work.

III. Equipment List

The following materials, as required, shall be available for IDW handling and storage:

Appropriate personal protective equipment as specified in the Site Health and Safety Plan

- 55-gallon steel drums, DOT 1A2 or equivalent
- ¾ -inch socket wrench
- Hammer
- Leather gloves
- Drum dolly
- Appropriate drum labels (outdoor waterproof self adhesive)
- Polyethylene storage tank
- Appropriate labeling, packing, chain-of-custody forms, and shipping materials as specified in the *Chain-of-Custody* SOP and *Field Sampling Handling, Packing, and Shipping* SOP.
- Indelible ink and/or permanent marking pens
- Plastic sheeting

- Appropriate sample containers, labels, and forms
- Stainless-steel bucket auger
- Stainless steel spatula or knife
- Stainless steel hand spade
- Stainless steel scoop
- Digital camera
- Field logbook.

IV. Cautions

- Filled drums can be very heavy, always use appropriate moving techniques and equipment.
- Similar media will be stored in the same drums to aid in sample analysis and disposal.
- Drum lids must be secured to prevent rainwater from entering the drums.
- Drums containing solid material may not contain any free liquids.
- Waste containers stored for extended periods of time may be subject to deterioration. Drum over packs may be used as secondary containment.
- All drums must be in good condition to prevent potential leakage and facilitate subsequent disposal. Inspect the drums for dents and rust, and verify the drum has a secure lid prior to use.

V. Health and Safety Considerations

- Appropriate personal protective equipment must be worn by all field personnel within the designated work area.
- Air monitoring may be required during certain field activities as required in the Site Health and Safety Plan.

- If excavating in potentially hazardous areas is possible, contingency plans should be developed to address the potential for encountering gross contamination or non-aqueous phase liquids.

- ARCADIS field personnel will be familiar and compliant with Client-specific health and safety requirements such as Chevron’s hand safety policy including the prohibition of fixed and/or folding blade knives.

VI. Procedure

Waste storage and handling procedures to be used depend upon the type of generated waste. For this reason, IDW should be stored in a secure location onsite in separate 55-gallon storage drums, solids can be stockpiled onsite (if non-hazardous), and purge water may be stored in polyethylene tanks. Waste materials such as broken sample bottles or equipment containers and wrappings will be stored in 55-gallon drums unless they were not in contact with sample media.

Management of IDW

Minimization of IDW should be considered by the Project Manager during all phases of the project. Site managers may want to consider techniques such as replacing solvent-based cleaners with aqueous-based cleaners for decontamination of equipment, reuse of equipment (where it can be decontaminated), limitation of traffic between exclusion and support zones, and drilling methods and sampling techniques that generate little waste. Alternative drilling and subsurface sampling methods may include the use of small diameter boreholes, as well as borehole testing methods such as a core penetrometer or direct push technique instead of coring (EPA, 1993).

Drum Storage

Drums containing hazardous waste shall be stored in accordance with the requirements of 40 CFR 265 Subpart I (for containers) and 265 Subpart DD (for containment buildings). All 55-gallon drums will be stored at a secure, centralized on-site location that is readily accessible for vehicular pick-up. Drums confirmed as, or believed to contain hazardous waste will be stored over an impervious surface provided with secondary containment. The storage location will, for drums containing liquid, have a containment system that can contain at least the larger of 10% of the aggregate volume of staged materials or 100% of the volume of the largest container. Drums will be closed during storage and be in good condition in accordance with the Guide to Management of Investigation-Derived Wastes (USEPA, 1992).

Hazardous Waste Determination

Waste material must be characterized to determine if it meets any of the federal definitions of hazardous waste as required by 40 CFR § 262.11. If the waste does not meet any of the federal definitions, it must then be established if any state-specific hazardous waste criteria exist/apply.

Generator Status

Once hazardous waste determination has been made, the generator status will be determined. Large quantity generators (LQG) are generators who generate more than 1,000 kilograms of hazardous waste in a calendar month. Small quantity generators (SQG) of hazardous waste are generators who generate greater than 100 kilograms but less than 1,000 kilograms of hazardous waste in a calendar month. Conditionally exempt small quantity generators (CESQG) are generators who generate less than 100 kilograms of hazardous waste per month. Please note that a generator status may change from month to month and that a notice of this change is usually required by the generator's state agency.

Accumulation Time for Hazardous Waste

A LQG may accumulate hazardous waste on site for 90 days or less without a permit and without having interim status provided that such accumulation is in compliance with specifications in 40 CFR § 262.34. A SQG may accumulate hazardous waste on site for 180 days or less without a permit or without having interim status subject to the requirements of 40 CFR § 262.34(d). CESQG requirements are found in 40 CFR § 261.5. **NOTE**: The CESQG and SQG provisions of 40 CFR § 261.5, 262.20(e), 262.42(b) and 262.44 may not be recognized by some states (e.g. Rhode Island). **State-specific regulations must be reviewed and understood prior to the generation of hazardous waste.**

Satellite Accumulation of Hazardous Waste

Satellite accumulation (SAA) shall mean the accumulation of as much as fifty-five (55) gallons of hazardous waste, or the accumulation of as much as one quart of acutely hazardous waste, in containers at or near any point of generation where the waste initially accumulates, which is under the control of the operator of the process generating the waste, without a permit or interim status and without complying with the requirements of 40 CFR § 262.34(a) and without any storage time limit, provided that the generator complies with 40 CFR § 262.34(c)(1)(i).

Once more than 55 gallons of hazardous waste accumulates in SAA, the generator has three days to move this waste into storage.

Storage recommendations for hazardous waste include:

- Ignitable Hazardous wastes must be >50 feet from the property line per 40 CFR § 265.176 (LQG generators only).
- Hazardous waste must be stored on a concrete slab (asphalt is acceptable if there are no free liquids in the waste) per 40 CFR § 265.176.
- Drainage must be directed away from the accumulation area.
- Area must be properly vented.
- Area must be secure.

Drum/Container Labeling

Drums will be labeled on both the side and lid of the drum using a permanent marking pen. Old drum labels must be removed to the extent possible, descriptions crossed out should any information remain, and new labels affixed on top of the old labels. Other containers used to store various types of waste (polyethylene tanks, roll-off boxes, end-dump trailers, etc.) will be labeled with an appropriate "Waste Container" or “Testing in Progress” label pending characterization. Drums and containers will be labeled as follows:

- Appropriate waste characterization label (Testing In Progress, Hazardous, or Non-Hazardous)
- Waste generator's name (e.g., client name)
- Project name
- Name and telephone number of ARCADIS project manager
- Composition of contents (e.g., used oil, acetone 40%, toluene 60%)
- Media (e.g., solid, liquid)
- Accumulation start date

- Drum number of total drums as reconciled with the Drum Inventory maintained in the field log book.

IDW containers will remain closed except when adding or removing waste. Immediately upon beginning to place waste into the drum/container, a “Waste Container” or “Testing in Progress” label will be filled out to include the information specified above, and affixed to the container. Once the contents of the container are identified as either non-hazardous or hazardous, the following additional labels will be applied. Containers with waste determined to be non-hazardous will be labeled with a green and white "Non-Hazardous Waste" label over the "Waste Container" label. Containers with waste determined to be hazardous will be stored in an onsite storage area and will be labeled with the "Hazardous Waste" label and affixed over the "Waste Container" label. The ACCUMULATION DATE for the hazardous waste is the date the waste is first placed in the container and is the same date as the date on the "Waste Container" label. DOT hazardous class labels must be applied to all hazardous waste containers for shipment offsite to an approved disposal or recycling facility. In addition a DOT proper shipping name shall be included on the hazardous waste label. The transporter should be equipped with the appropriate DOT placards. However, placarding or offering placards to the initial transporter is the responsibility of the generator per 40 CFR § 262.33.

Inspections and Documentation

All IDW will be documented as generated on a Drum Inventory Log maintained in the field log book. The Drum Inventory will record the generation date, type, quantity, matrix and origin (e.g. Boring-1, Test Pit 3, etc) of materials in every drum, as well as a unique identification number for each drum. The drum inventory will be used during drum pickup to assist with labeling of drums. The drum storage area and any other areas of temporarily staged waste, such as soil/debris piles, will be inspected weekly. The weekly inspections will be recorded in the field notebook or on a Weekly Inspection Log. Digital photographs will be taken upon the initial generation and drumming/staging of waste, and final labeling after characterization to document compliance with labeling and storage protocols, and condition of the container. Evidence of damage, tampering or other discrepancy should be documented photographically.

Emergency Response and Notifications

Specific procedures for responding to site emergencies will be detailed in the HASP. If the generator is designated as a LQG, a Contingency Plan will need to be prepared to include emergency response and notification procedures per 40 CFR § 265 Subpart D. In the event of a fire, explosion, or other release which could threaten human health outside of the site or when Client or ARCADIS has knowledge of a spill that has reached surface water, Client or ARCADIS must immediately notify the National Response Center (800-424-8802) in accordance with 40 CFR § 262.34. Other notifications to state agencies may also be necessary.

Drilling Soil Cuttings and Muds

Soil cuttings are solid to semi-solid soils generated during trenching activities, subsurface soil sampling, or installation of monitoring wells. Depending on the drilling method, drilling fluids known as "muds" may be used to remove soil cuttings. Drilling fluids flushed from the borehole must be directed into a settling section of a mud pit. This allows reuse of the decanted fluids after removal of the settled sediments. Soil cuttings will be labeled and stored in 55-gallon drums with bolt-sealed lids.

Excavated Solids

Excavated solids may include, but are not limited to soil, fill and construction and demolition debris. Excavated solids may be temporarily stockpiled onsite as long as the material is a RCRA non-hazardous waste and the solids will be treated onsite pursuant to a certified, authorized, or permitted treatment method, or properly disposed off-site. Stockpiled materials characterized as hazardous must be immediately containerized and removed from the site within 90 days of generation (except for soils using satellite accumulation). Excavated solids should be stockpiled and maintained in a secure area onsite. At a minimum, the floor of the stockpile area will be covered with a 20-mil high density polyethylene liner that is supported by a foundation or at least a 60-mil high density polyethylene liner that is not supported by a foundation. The excavated material will not contain free liquids. The owner/operator will provide controls for windblown dispersion, run-on control, and precipitation runoff. The run-on control system will prevent flow onto the active portion of the pile during peak discharge from at least a 25-year storm and the run-off management system will collect and control at least the water volume resulting from a 24-hour, 25-year storm (EPA, 1992). Additionally, the stockpile area will be inspected on a weekly basis and after storm events. Individual states may require that the stockpile be inspected/certified by a licensed professional engineer. Stockpiled material will be covered with a 6-mil polyvinyl chloride (PVC) liner. The stockpile cover will be secured in place with appropriate material (concrete blocks, weights, etc.) to prevent the movement of the cover. Excavated solids may also be placed in roll off containers and covered with a 6-mil PVC liner pending results for waste characterization.

Decontamination Solutions

Decontamination solutions are generated during the decontamination of personal protective equipment and sampling equipment. Decontamination solutions may range from detergents, organic solvents and acids used to decontaminate small field sampling equipment to steam cleaning rinsate used to wash heavy field equipment. These solutions are to be labeled and stored in 55-gallon drums with bolt-sealed lids.

Disposable Equipment

Disposable equipment includes personal protective equipment (tyvek coveralls, gloves, booties and APR cartridges) and disposable sampling equipment such as trowels or disposable bailers. If the media sampled exhibits hazardous characteristics per results of waste characterization sampling, disposable equipment will also be disposed of as a hazardous waste. These materials will be stored onsite in labeled 55-gallon drums pending analytical results for waste characterization.

Purge Water

Purge water includes groundwater generated during well development, groundwater sampling, or aquifer testing. The volume of groundwater generated will dictate the appropriate storage procedure. Monitoring well development and groundwater sampling may generate three well volumes of groundwater or more. This volume will be stored in labeled 55-gallon drums. Aquifer tests may generate significantly greater volumes of groundwater depending on the well yield and the duration of the test. Therefore, large-volume portable polyethylene tanks will be considered for temporary storage pending groundwater-waste characterization.

Purged Water Storage Tank Decontamination and Removal

The following procedures will be used for inspection, cleaning, and offsite removal of storage tanks used for temporary storage of purge water. These procedures are intended to be used for rented portable tanks such as Baker Tanks or Rain for Rent containers. Storage tanks will be made of inert polyethylene materials.

The major steps for preparing a rented tank for return to a vendor include characterizing the purge water, disposing of the purge water, decontaminating the tank, final tank inspection, and mobilization. Decontamination and inspection procedures are describe in further detail below.

- Tank Cleaning: Most vendors require that tanks be free of any sediment and water before returning, a professional cleaning service may be required. Each specific vendor should be consulted concerning specific requirements for returning tanks.

- Tank Inspection: After emptying the tank, purged water storage tanks should be inspected for debris, chemical staining, and physical damage. The vendors require that tanks be returned in the original condition (i.e., free of sediment, staining and no physical damage).

VII. Waste Characterization Sampling and Shipping

Soil/Solids Characterization

Waste characterization will be conducted in accordance with waste hauler, waste handling facility, and state/federal requirements. In general, RCRA hazardous wastes are those solid wastes determined by a Toxicity Characteristic Leaching Procedure (TCLP) test or to contain levels of certain toxic metals, pesticides, or other organic chemicals above specific federally regulated thresholds. If the one or more of 40 toxic compounds listed in Table I of 40 CFR § 261.24 are detected in the sample at levels above the maximum unregulated concentrations, the waste must be characterized as a toxic hazardous waste. Wastes can also be considered “listed” hazardous waste depending on site-specific processes.

Composite soil samples will be collected at a frequency of one sample per 10 cubic yard basis for stockpiled soil or one per 55-gallon drum for containerized. A four point composite sample will be collected per 10 cubic yards of stockpiled material and for each drum. Sample and composite frequencies may be adjusted in accordance with the waste handling facility’s requirements. Waste characterization samples may be analyzed for the TCLP volatile organic compounds (VOCs), TCLP semi-volatile organic compounds (SVOCs), TCLP RCRA metals, and polychlorinated biphenyls, as well as corrosivity (pH), reactivity and flammability (flashpoint). Additional samples may be collected and analyzed by the laboratory on a contingency basis.

Wastewater Characterization

Waste characterization will be conducted in accordance with the requirements of the waste hauler, waste handling facility, and state/federal governments. In general, purge water should be analyzed by methods appropriate for the known contaminants, if any, that have been historically detected in the monitoring wells. Samples will be collected and analyzed in accordance with the requirements of the waste disposal facility.

Wastewater characterization samples may be analyzed for TCLP volatile organic compounds (VOCs), TCLP semi-volatile organic compounds (SVOCs), TCLP RCRA metals, and polychlorinated biphenyls, as well as corrosivity (pH), reactivity and flammability (flashpoint). Additional samples may be collected and analyzed by the laboratory on a contingency basis.

Sample Handling and Shipping

All samples will be appropriately labeled, packed, and shipped, and the chain-of-custody will be filled out in accordance with the Chain-of-Custody SOP and Field Sampling Handling, Packing, and Shipping SOP and Hazardous Materials Packaging and Shipping SOP.

It should be noted that additional training is required for packaging and shipping of hazardous and/or dangerous materials. Please reference the following ARCADIS intranet team page for more information: http://team/sites/hazmat/default.aspx.

Preparing Waste Shipment Documentation (Hazardous and Non-Hazardous)

Waste profiles will be prepared by the ARCADIS PM and forwarded, along with laboratory analytical data to the Client PM for approval/signature. The Client PM will then return the profile to ARCADIS who will then forward to the waste removal contractor for preparation of a manifest. The manifest will be reviewed by ARCADIS prior to forwarding to the Client PM for approval. Upon approval of the manifest, the Client PM will return the original signed manifest directly to the waste contractor or to the ARCADIS PM for forwarding to the waste contractor.

Final drum labeling and pickup will be supervised by an ARCADIS representative who is experienced with waste labeling procedures. The ARCADIS representative will have a copy of the drum inventory maintained in the field book and will reconcile the drum inventory with the profile numbers on the labels and on the manifest. Different profile numbers will be generated for different matrices or materials in the drums. For example, the profile number for drill cuttings will be different than the profile number for purge water. **When there are multiple profiles it is critical that the proper label, with the profile number appropriate to a specific material be affixed to the proper drums.** A copy of the ARCADIS drum inventory will be provided to the waste transporter during drum pickup and to the facility receiving the waste.

VIII. Data Recording and Management

Waste characterization sample handling, packing, and shipping procedures will be documented in accordance with the *Quality Assurance Project Plan*, if one exists. Copies of the chains-of-custody forms will be maintained in the project file.

Following waste characterization, IDW containers will be re-labeled with the appropriate waste hazardous or non-hazardous waste labels and the client will initiate disposal at the appropriate waste disposal facility.

IX. Quality Assurance

The chain-of-custody and sample labels for waste characterization samples will be filled out in accordance with the *Quality Assurance Project Plan*.

X. References

United States Environmental Protection Agency (USEPA). 1992. Guide to Management of Investigation-Derived Wastes. Office of Remedial and Emergency Response. Hazardous Site Control Division. January 1992.

USEPA. 1991. *Guide to Discharging CERCLA Aqueous Wastes to Publicly Owned Treatment Works (POTWs)*. Office of Remedial and Emergency Response. Hazardous Site Control Division 0S-220W. March 1991.

Attachment 3G
Monitoring Well Installation

ARCADIS
Infrastructure, environment, facilities

Imagine the result

Monitoring Well Installation

Rev. #: 2

Rev Date: August 22, 2008

Approval Signatures

Prepared by: Sonya A Cadle Date: 8/25/08

Reviewed by: Michael J Gefell Date: 8/25/08
(Technical Expert)

I. Scope and Application

The procedures set out herein are designed to produce standard groundwater monitoring wells suitable for: (1) groundwater sampling, (2) water level measurement, (3) bulk hydraulic conductivity testing of formations adjacent to the open interval of the well.

Monitoring well boreholes in unconsolidated (overburden) materials are typically drilled using the hollow-stem auger drilling method. Other drilling methods that are also suitable for installing overburden monitoring wells, and are sometimes necessary due to site-specific geologic conditions, include: drive-and-wash, spun casing, Rotasonic, dual-rotary (Barber Rig), and fluid/mud rotary with core barrel or roller bit. Direct-push techniques (e.g., Geoprobe or cone penetrometer) and driven well points may also be used in some cases within the overburden. Monitoring wells within consolidated materials such as bedrock are commonly drilled using water-rotary (coring or tri-cone roller bit), air rotary or Rotasonic methods. The drilling method to be used at a given site will be selected based on site-specific consideration of anticipated drilling/well depths, site or regional geologic knowledge, type of monitoring to be conducted using the installed well, and cost.

No oils or grease will be used on equipment introduced into the boring (e.g., drill rod, casing, or sampling tools). No coated bentonite pellets will be used in the well drilling or construction process. Specifications of materials to be installed in the well will be obtained prior to mobilizing onsite, including:

- well casing;
- bentonite;
- sand; and
- grout.

Well materials will be inspected and, if needed, cleaned prior to installation.

II. Personnel Qualifications

Monitoring well installation activities will be performed by persons who have been trained in proper well installation procedures under the guidance of an experienced field geologist, engineer, or technician. Where field sampling is performed for soil or bedrock characterization, field personnel will have undergone in-field training in soil or bedrock description methods, as described in the appropriate SOP(s) for those activities.

III. Equipment List

The following materials will be available during soil boring and monitoring well installation activities, as required:

- Site Plan with proposed soil boring/well locations;
- Work Plan or Field Sampling Plan (FSP), and site Health and Safety Plan (HASP);
- personal protective equipment (PPE), as required by the HASP;
- traffic cones, delineators, caution tape, and/or fencing as appropriate for securing the work area, if such are not provided by drillers;
- appropriate soil sampling equipment (e.g., stainless steel spatulas, knife);
- soil and/or bedrock logging equipment as specified in the appropriate SOPs;
- appropriate sample containers and labels;
- drum labels as required for investigation derived waste handling;
- chain-of-custody forms;
- insulated coolers with ice, when collecting samples requiring preservation by chilling;
- photoionization detector (PID) or flame ionization detector (FID);
- ziplock style bags;
- water level or oil/water interface meter;
- locks and keys for securing the well after installation;
- decontamination equipment (bucket, distilled or deionized water, cleansers appropriate for removing expected chemicals of concern, paper towels);

- field notebook.

Prior to mobilizing to the site, ARCADIS personnel will contact the drilling subcontractor or in-house driller (as appropriate) to confirm that appropriate sampling and well installation equipment will be provided. Specifications of the sampling and well installation equipment are expected to vary by project, and so communication with the driller will be necessary to ensure that the materials provided will meet the project objectives. Equipment typically provided by the driller could include:

- drilling equipment required by the American Society of Testing and Materials (ASTM) D 1586, when performing split-spoon sampling;
- disposable plastic liners, when drilling with direct-push equipment;
- drums for investigation derived waste;
- drilling and sampling equipment decontamination materials;
- decontamination pad materials, if required; and
- well construction materials.

IV. Cautions

Prior to beginning field work, underground utilities in the vicinity of the drilling areas will be delineated by the drilling contractor or an independent underground utility locator service. See separate SOP for utility clearance.

Some regulatory agencies require a minimum annular space between the well or permanent casing and the borehole wall. When specified, the minimum clearance is typically 2 inches on all sides (e.g., a 2-inch diameter well requires a 6-inch diameter borehole). In addition, some regulatory agencies have specific requirements regarding grout mixtures. Determine whether the oversight agency has any such requirements prior to finalizing the drilling and well installation plan.

If dense non-aqueous phase liquids (DNAPL) are known or expected to exist at the site, refer to the DNAPL Contingency Plan SOP for additional details regarding drilling and well installation to reduce the potential for inadvertent DNAPL remobilization.

Avoid using drilling fluids or materials that could impact groundwater or soil quality, or could be incompatible with the subsurface conditions.

Similarly, consider the material compatibility between the well materials and the surrounding environment. For example, PVC well materials are not preferred when DNAPL is present. In addition, some groundwater conditions leach metals from stainless steel.

Water used for drilling and sampling of soil or bedrock, decontamination of drilling/sampling equipment, or grouting boreholes upon completion will be of a quality acceptable for project objectives. Testing of water supply should be considered.

Specifications of materials used for backfilling bore hole will be obtained, reviewed and approved to meet project quality objectives. Bentonite is not recommended where DNAPLs are likely to be present. In these situations, neat cement grout is preferred.

No coated bentonite pellets will be used in monitoring well construction, as the coating could impact the water quality in the completed well.

Monitoring wells may be installed with Schedule 40 polyvinyl chloride (PVC) to a maximum depth of 200 feet below ground surface (bgs). PVC monitoring wells between 200 and 400 feet total depth will be constructed using Schedule 80 PVC. Monitoring wells deeper than 400 feet will be constructed using steel.

V. Health and Safety Considerations

Field activities associated with monitoring well installation will be performed in accordance with a site-specific HASP, a copy of which will be present on site during such activities.

VI. Procedures

The procedures for installing groundwater monitoring wells are presented below:

Hollow-Stem Auger, Drive-and-Wash, Spun Casing, Fluid/Mud Rotary, Rotasonic, and Dual-Rotary Drilling Methods

1. Locate boring/well location, establish work zone, and set up sampling equipment decontamination area.

2. Advance boring to desired depth. Collect soil and/or bedrock samples at appropriate interval as specified in the Work Plan and/or FSP. Collect, document, and store samples for laboratory analysis as specified in the Work Plan and/or FSP. Decontaminate equipment between samples in accordance with the Work Plan and/or FSP. A common sampling method that produces

high-quality soil samples with relatively little soil disturbance is the ASTM D 1586 - Standard Test Method for Penetration Test and Split-Barrel Sampling of Soils. Split-spoon samples are obtained during drilling using hollow-stem auger, drive-and-wash, spun casing, and fluid/mud rotary. Rotasonic drilling produces large-diameter soil cores that tend to be more disturbed than split-spoon samples due to the vibratory action of the drill casing. Dual-rotary removes cuttings by compressed air and allows only a general assessment of geology. High-quality bedrock samples can be obtained by coring.

3. Describe each soil or bedrock sample as outlined in the appropriate SOP. Record descriptions in the field notebook and/or personal digital assistant (PDA). It should be noted that PDA logs must be electronically backed up and transferred to a location accessible to other project team members as soon as feasible to retain and protect the field data. During soil boring advancement, document all drilling events in field notebook, including blow counts (number of blows required to advance split-spoon sampler in 6-inch increments) and work stoppages. Blow counts will not be available if Rotasonic, dual-rotary, or direct-push methods are used. When drilling in bedrock, the rate of penetration (minutes per foot) is recorded.

4. If it is necessary to install a monitor well into a permeable zone below a confining layer, particularly if the deeper zone is believed to have water quality that differs significantly from the zone above the confining layer, then a telescopic well construction should be considered. In this case, the borehole is advanced approximately 3 to 5 feet into the top of the confining layer, and a permanent casing (typically PVC, black steel or stainless steel) is installed into the socket drilled into the top of the confining layer. The casing is then grouted in place. The preferred methods of grouting telescoping casings include: pressure-injection grouting using an inflatable packer installed temporarily into the base of the casing, such that grout is injected out the bottom of the casing until it is observed at ground surface outside the casing; displacement-method grouting (also known as the Halliburton method), which entails filling the casing with grout and displacing the grout out the bottom of the casing by pushing a drillable plug, typically made of wood to the bottom of the casing, following by tremie grouting the remainder of the annulus outside the casing; or tremie grouting the annulus surrounding the casing using a tremie pipe installed to the base of the borehole. In all three cases, the casing is grouted to the ground surface, and the grout is allowed to set prior to drilling deeper through the casing. Site-specific criteria and work plans should be created for the completion of non-standard monitoring wells, including telescopic wells.

5. In consolidated formations such as competent bedrock, a monitoring well may be completed with an open borehole interval without a screen and sandpack. In these cases, the borehole is advanced to the targeted depth of the top of the open interval. A permanent casing is then grouted in place following the procedures described in Step 4 above. After the grout sets, the borehole is advanced by drilling through the permanent casing to the targeted bottom depth of the open interval, which then serves as the monitoring interval for the well. If open-borehole interval stability is found to be questionable or if a specific depth interval is later selected for monitoring, a screened monitoring well may later be installed within the open-borehole interval, depending on the annular space and well diameter requirements.

6. Prior to screened well installation or after the completion of an open-bedrock well, the water level or oil/water interface probe should be used to determine the static water level in the borehole in relation to the proposed well screen or open-interval location. If necessary, an open-bedrock well may be drilled deeper to intersect the water table or a permeable water-bearing zone.

7. Upon completing the borehole to the desired depth, if a screened well construction is desired, install the monitoring well by lowering the screen and casing assembly with sump through the augers or casing. Monitoring wells typically will be constructed of 2-inch-diameter, flush-threaded PVC or stainless steel slotted well screen and blank riser casing. Smaller diameters may be used if wells are installed using direct-push methodology or if multiple wells are to be installed in a single borehole. The screen length will be specified in the Work Plan or FSP based on regulatory requirements and specific monitoring objectives. Monitoring well screens are usually 5 to 10 feet long, but may be up to 25 feet long in very low permeability, thick geologic formations. The screen length will depend on the purpose for the well and the objectives of the groundwater investigation. Typically, the slot size will be 0.010 inch and the sand pack will be 20-40, Morie No. 0, or equivalent. In very fine-grained formations where sample turbidity needs to be minimized, it may be preferred to use a 0.006-inch slot size and 30-65, Morie No. 00, or equivalent sand pack. Alternatively, where monitoring wells are installed in coarse-grained deposits and higher well yield is required, a 0.020-inch slot size and 10-20, Morie No. 1, or equivalent sand pack may be preferred. To the extent practicable, the slot size and sand pack gradation may be predetermined in the Work Plan or FSP based on site-specific grain-size analysis or other geologic considerations or monitoring objectives. A blank sump may be attached below the well screen if the well is being installed for DNAPL recovery/monitoring purposes. If so, the annular space around the sump will be backfilled with neat cement grout to the bottom of the well screen prior to placing the sand pack around the screen. A

blank riser will extend from the top of the screen to approximately 2.5 feet above grade or, if necessary, just below grade where conditions warrant a flush-mounted monitoring well. For wells greater than 50 feet deep, centralizers may be desired to assist in centralizing the monitoring well in the borehole during construction.

8. When the monitoring well assembly has been set in place and the grout has been placed around the sump (if any), place a washed silica sand pack in the annular space from the bottom of the boring to a height of 1 to 2 feet above the top of the well screen. The sand pack is placed and drilling equipment extracted in increments until the top of the sand pack is at the appropriate depth. The sand pack will be consistent with the screen slot size and the soil particle size in the screened interval, as specified in the Work Plan or FSP. A hydrated bentonite seal (a minimum of 2 feet thick) will then be placed in the annular space above the sand pack. If non-hydrated bentonite is used, the bentonite should be permitted to hydrate in place for a minimum of 30 minutes before proceeding. No coated bentonite pellets will be used in monitoring well drilling or construction. Potable water may be added to hydrate the bentonite if the seal is above the water table. Monitor the placement of the sand pack and bentonite with a weighted tape measure. During the extraction of the augers or casing, a cement/bentonite or neat cement grout will be placed in the annular space from the bentonite seal to a depth approximately 2 feet bgs.

9. Place a locking, steel protective casing (extended at least 1.5 feet below grade and 2 feet above grade) over the riser casing and secure with a neat cement seal. Alternatively, for flush-mount completions, place a steel curb box with a bolt-down lid over the riser casing and secure with a neat cement seal. In either case, the cement seal will extend approximately 1.5 to 2.0 feet below grade and laterally at least 1 foot in all directions from the protective casing, and should slope gently away to promote drainage away from the well. Monitoring wells will be labeled with the appropriate designation on both the inner and outer well casings or inside of the curb box lid.

   When an above-grade completion is used, the PVC riser will be sealed using an expandable locking plug and the top of the well will be vented by drilling a small-diameter (1/8 inch) hole near the top of the well casing or through the locking plug, or by cutting a vertical slot in the top of the well casing. When a flush-mount installation is used, the PVC riser will be sealed using an unvented, expandable locking plug.

10. During well installation, record construction details and actual measurements relayed by the drilling contractor and tabulate materials used (e.g., screen and riser footages; bags of bentonite, cement, and sand) in the field notebook.

11. After completing the well installation, lock the well, clean the area, and dispose of materials in accordance with the procedures outlined in Section VII below.

Direct-Push Method

The direct-push drilling method may also be used to complete soil borings and install monitoring wells. Examples of this technique include the Diedrich ESP vibratory probe system, GeoProbe®, or AMS Power Probe® dual-tube system. Environmental probe systems typically use a hydraulically operated percussion hammer. Depending on the equipment used, the hammer delivers 140- to 350-foot pounds of energy with each blow. The hammer provides the force needed to penetrate very stiff/medium dense soil formations. The hammer simultaneously advances an outer steel casing that contains a dual-tube liner for sampling soil. The outside diameter (OD) of the outer casing ranges from 1.75 to 2.4 inches and the OD of the inner sampling tube ranges from 1.1 to 1.8 inches. The outer casing isolates shallow layers and permits the unit to continue to probe at depth. The double-rod system provides a borehole that may be tremie-grouted from the bottom up. Alternatively, the inside diameter (ID) of the steel casing provides clearance for the installation of small-diameter (e.g., 0.75- to 1-inch ID) micro-wells. The procedures for installing monitoring wells in soil using the direct-push method are described below.

1. Locate boring/well location, establish work zone, and set up sample equipment decontamination area.

2. Advance soil boring to designated depth, collecting samples at intervals specified in the Work Plan. Samples will be collected using dedicated, disposable, plastic liners. Describe samples in accordance with the procedures outlined in Step 3 above. Collect samples for laboratory analysis as specified in the Work Plan and/or FSP.

3. Upon advancing the borehole to the desired depth, install the micro-well through the inner drill casing. The micro-well will consist of approximately 1-inch ID PVC or stainless steel slotted screen and blank riser. The sand pack, bentonite seal, and cement/bentonite grout will be installed as described, where applicable, in Step 7 and 8 above.

4. Install protective steel casing or flush-mount, as appropriate, as described in Step 9 above. During well installation, record construction details and tabulate materials used.

5. After completing the well installation, lock the well, clean the area, and dispose of materials in accordance with the procedures outlined in Section VII below.

Driven Well Point Installation

Well points will be installed by pushing or driving using a drilling rig or direct-push rig, or hand-driven where possible. The well point construction materials will consist of a 1- to 2-inch-diameter threaded steel casing with either 0.010- or 0.020-inch slotted stainless steel screen. The screen length will vary depending on the hydrogeologic conditions of the site. The casings will be joined together with threaded couplings and the terminal end will consist of a steel well point. Because they are driven or pushed to the desired depth, well points do not have annular backfill materials such as sand pack or grout.

VII. Waste Management

Investigation-derived wastes (IDW), including soil cuttings and excess drilling fluids (if used), decontamination liquids, and disposable materials (well material packages, PPE, etc.), will be placed in clearly labeled, appropriate containers, or managed as otherwise specified in the Work Plan, FSP, and/or IDW management SOP.

VIII. Data Recording and Management

Drilling activities will be documented in a field notebook. Pertinent information will include personnel present on site, times of arrival and departure, significant weather conditions, timing of well installation activities, soil descriptions, well construction specifications (screen and riser material and diameter, sump length, screen length and slot size, riser length, sand pack type), and quantities of materials used. In addition, the locations of newly-installed wells will be documented photographically or in a site sketch. If appropriate, a measuring wheel or engineer’s tape will be used to determine approximate distances between important site features.

The well or piezometer location, ground surface elevation, and inner and outer casing elevations will be surveyed using the method specified in the site Work Plan. Generally, a local baseline control will be set up. This local baseline control can then be tied into the appropriate vertical and horizontal datum, such as the National Geodetic Vertical Datum of 1929 or 1988 and the State Plane Coordinate System. At a minimum, the elevation of the top of the inner casing used for water-level measurements should be measured to the nearest 0.01 foot. Elevations will be established in relation to the National Geodetic Vertical Datum of 1929. A permanent mark will be placed on top of the inner casing to mark the point for water-level measurements.

IX. Quality Assurance

All drilling equipment and associated tools (including augers, drill rods, sampling equipment, wrenches, and any other equipment or tools) that may have come in contact with soil will be cleaned in accordance with the procedures outlined in the appropriate SOP. Well materials will also be cleaned prior to well installation.

X. References

American Society of Testing and Materials (ASTM) D 1586 - *Standard Test Method for Penetration Test and Split-Barrel Sampling of Soils*.

Attachment 3H
Monitoring Well Integrity Survey

ARCADIS
Infrastructure, environment, buildings

Imagine the result

Monitoring Well Integrity Survey

Rev. #: 0

Rev Date: February 24, 2009

Approval Signatures

Prepared by: Michael J Gefell Date: 2/24/09
Michael Gefell

Reviewed by: Michael J Gefell Date: 2/24/09
Michael Gefell (Technical Expert)

I. Scope and Application

This Standard Operating Procedure (SOP) specifies the procedures for performing inventories of existing monitoring wells. This SOP also applies to piezometers that are constructed analogous to monitoring wells. For simplicity, such piezometers are also referred to as monitoring wells for the remainder of this document.

Monitoring well inventories are periodically conducted to assess the integrity of existing monitoring wells and to identify the need for repairs, replacement of parts, or replacement of wells that are determined to no longer be usable. A well inventory involves an inspection of the overall condition of the well, comparison of measurable quantities (e.g., riser stickup relative to grade and total depth), general verification of survey coordinates and elevation, and measurement of depth to water in the well.

II. Personnel Qualifications

All personnel shall meet the requirements of the site-specific Health and Safety Plan (HASP).

The Project Manager is responsible for ensuring that the activities described herein are conducted in accordance with this SOP and any other appropriate procedures. This will be accomplished through staff training and by maintaining quality assurance/quality control (QA/QC).

The Field Manager is responsible for periodic observation of field activities and review of field generated documentation associated with this SOP. The Field Manager is also responsible for implementation of corrective action if well conditions necessitate them.

III. Equipment List

The following materials will be available, as required, during performance of a monitoring well inventory:

- Health and safety equipment (as required by the site-specific Health and Safety Plan)
- Ruler or tape measure
- Water level indicator and/or interface probe
- Indelible pen

- Paint pen
- Well keys
- Wrenches for accessing flush-mount well covers
- Cleaning equipment
- Well construction information
- Field notebook or Personal Digital Assistant (PDA)

If feasible, a supply of typical replacement parts (e.g., locks, bolts, and well caps) should be available to enable immediate usage as necessary.

IV. Cautions

It is important to confirm the correct identity of wells, particularly when they are installed in a cluster. In these cases, however, the wells usually differ significantly in terms of depth below grade. During the well integrity survey, verify that all wells are properly labeled by comparing their measured depth to the reported depth as installed. If the well identity is incorrectly labeled or not labeled, provide a clear, correct label using an indelible pen on the inside of the steel protective cover for the well, or on the outside of the steel protective cover using a paint pen.

V. Health and Safety Considerations

Field activities associated with monitoring well installation will be performed in accordance with a site-specific HASP, a copy of which will be present on site during such activities. Care should be taken using tools to access flush-mount curb boxes. Wells in or near roadways must not be accessed without proper traffic cones and flagging. Access to wells containing chemicals of concern may pose a hazard of chemical exposure.

VI. Procedure

The typical procedure for assessing the integrity of a monitoring well is outlined below.

Step 1. Prior to mobilizing in the field, obtain a list of wells/piezometers to be inventoried and available information concerning their location and physical characteristics.

Step 2. Identify site and well identification number on the Well Integrity Assessment Form (Attachment 1). Record all observations on this form, supplemented by notes in the field notebook if necessary.

Step 3. Examine the well for the presence of an identification label. If absent, label the well with the appropriate well number after measuring the total depth of the well to verify that the depth matches the well number (see Step 8 below). If the well identity is incorrectly labeled or not labeled, provide a clear, correct label using an indelible pen on the inside of the steel protective cover for the well, and on the outside of the steel protective cover using a paint pen.

Step 4. Examine the surface condition of the well. Record the type of well (i.e., flush mount or above-grade stickup), condition of the well cover and surface seal. Confirm the protective casing is not bent, the PVC casing is not broken or chipped, and there is no evidence of frost heaving.

Step 5. Unlock and open the well. Record the type (e.g., PVC or stainless steel), dimensions (i.e., casing diameter and stickup relative to grade), condition of the well casing, and type of well cap. If well cap is missing, replace with available parts or record the type of cap required.

Step 6. Measure the above-grade portion of the well riser stickup and compare to the known length of the stickup measured during well installation (surveyed top of inner casing elevation minus ground surface elevation). If the difference between the observed stickup length and the known stickup length is greater than 0.1 foot, the monitoring well location and elevation should be re-surveyed.

Step 7. Locate the marked measuring point along the top of the well casing. If no mark is visible, add a mark at the highest point of the casing using an indelible pen

Step 8. Measure the depth to water and total depth of the well. For total depth measurements, account for any difference in calibration of the measuring tape on the probe (i.e., distance from part of probe that measures depth to water and the physical bottom of the probe which will measure total depth of the well). Record any obstructions encountered and a description of the feel of the well bottom (i.e., soft due to sediment or hard).

Step 9. Compare all observations concerning the measured dimensions of the well with the listed values. Based on these results, as well as other observations concerning the condition of the well, record any appropriate recommendations on the Monitoring Well Integrity Assessment form (Attachment 1). Perform any recommended maintenance activities that can be accomplished with available equipment.

Step 10. Remove all equipment from the well. If no additional maintenance activities are to be performed, close the well and collect all personal protection equipment (PPE) and other wastes generated for disposal (see Section V below).

VII. Follow-up Activities

Depending on the results of the well inventory, several additional activities may be warranted prior to future usage of the well. Typical follow-up activities include replacement of missing parts, removal of sediment from the base of the well, re-surveying of the well, or complete replacement if the well is determined to be unusable. These activities are briefly discussed below.

As stated above, a supply of locks, bolts, and well caps should be available for immediate usage during performance of the well inventories. However, it may not be feasible to maintain a supply of all potential replacement parts due to the variety of well types in use. Therefore, a list of required replacement parts should be compiled during the performance of a well inventory event. At the conclusion of the event, the necessary replacement parts for all wells should be obtained and installed.

Sediment accumulation occurs to some degree in all monitoring wells, particularly those that are not pumped on a routine basis. If a sufficient quantity of sediment which may adversely impact future groundwater sampling activities is observed during a well inventory (i.e., a sediment accumulation of greater than one-half foot above the bottom of the well screen), activities should be taken to remove the sediment. These activities will involve the removal of sediment by either pumping or bailing the well, followed by re-measurement of the total depth of the well to confirm that the total depth is near the reported values. The removed sediment should be inspected for the presence of filter pack materials which may indicate that the well screen has been damaged. If initial efforts are unsuccessful in clearing the sediment accumulations, the well may need to be re-developed or replaced.

The measuring points marked on the well risers are utilized as a base datum in the determination of groundwater elevations. The distance of these markers from the ground surface are verified against listed values during well inventory activities. Minor variations between listed and measured values may be attributed to an uneven ground surface around the well or to enhancements to the ground surface such as paving or grading activities which may have been performed since installation of the well. Therefore, minor variations (i.e., less than 3 inches) will be discounted and existing survey information for the measuring point on the well will be assumed to be accurate. Greater discrepancies may be attributed to damage or modifications to the well, such as cutting or lengthening the well riser. In these situations, the well should be re-surveyed to establish a new datum for future groundwater elevation measurements.

Replacement or decommissioning of a well may be warranted if the well is broken, obstructed, or otherwise compromised. If the well cannot be adequately repaired and is required for future monitoring purposes, a replacement well should be installed if no suitable alternate wells are located in the vicinity.

VII. Waste Management

Materials generated during well inventory activities, including disposable equipment, will be disposed of in appropriate containers.

VIII. Data Recording and Management

Field observations will be recorded on the Well Integrity Assessment Form (Attachment A), and/or in an appropriate Field Notebook or PDA. Well integrity inventory results will be retained in the project file.

IX. Quality Assurance

To verify accurate measurements of well stickup, depth to bottom, depth to groundwater, etc., measurements must be double-checked periodically (e.g., at least one of these measurements per well should be repeated to verify accuracy).

X. References

No references apply to this SOP.

XI. Attachments

A. Well Integrity Assessment Form

WELL INTEGRITY ASSESSMENT FORM

Site Name:______________________________
Well I.D.:______________________________
Date:__________________________________

(For each item, circle the appropriate response or fill in the blank)

Well I.D. Clearly Marked: YES NO
Well Completion: FLUSH MOUNT ABOVE-GRADE STANDPIPE
Lockable Cover: YES NO DAMAGED *(Describe below)*
Lock Present: YES NO ADDED Key Brand/Number:_____________________________
Measuring Point Marked: YES NO ADDED
Well Riser Diameter (inches): ____________________
Well Riser Type: PVC Stainless Steel Other *(Describe)* _____________________

Surface Condition

Cement Intact: YES NO *(Describe below)*
Curb Box/Well Cover Present: YES NO DAMAGED *(Describe below)*
All Bolts Present: YES NO *(Describe below)* NOT APPLICABLE
Ground Surface Slopes
Away from Well YES NO *(Describe below)*

Well Condition

Well Cap: PVC Slip Cap Pressure-fit Cap None
Well Vent: Slot Cut in Riser Vent Hole in Cap None Not Applicable *(Flush Mount Well)*
Reported Well Riser Stickup (feet): _____________*(use negative number if below grade)*
Measured Well Riser Stickup (feet): _____________ *(use negative number if below grade)*

Depth to Water (feet from Top of Well Riser): _________________ *-or-* DRY

Reported Total Depth of Well (feet below grade): ________________
Measured Total Depth of Well (feet below grade): ________________
Well Obstructed: YES NO If yes, list depth in feet from Top of Well Riser: ____________________
Well Bottom: SOFT *(contains sediment)* FIRM *(no sediment)*

Recommendations

Repair Concrete/Surface Completion: YES NO If yes, list date performed:______________________
Re-Survey Well: YES NO If yes, list date performed:______________________
Remove Sediment and Re-Measure Depth: YES NO If yes, list date performed:______________________
Replace Well Cap: YES NO If yes, list date performed:______________________
Replace Bolts: YES NO If yes, list date performed:_______________
Replace Lock: YES NO If yes, list date performed:______________________
Other/Miscellaneous Observations:

____

____

Inspector(s):____

Attachment 3I
Monitoring Well Development

ARCADIS
Infrastructure, environment, facilities

Imagine the result

Monitoring Well Development

Rev. #: 2.2

Rev. Date: March 22, 2010

Approval Signatures

Prepared by: David S. Lipson Date: 03/22/2010

Reviewed by: Michael J Gefell Date: 03/22/2010
(Technical Expert)

I. Scope and Application

Monitoring wells (or piezometers, well points, or micro-wells) will be developed to clear them of fine-grained sediment to enhance the hydraulic connection between the well and the surrounding geologic formation. Development will be accomplished by evacuating well water by either pumping or bailing. Prior to pumping or bailing, the screened interval will be gently surged using a surge block, bailer, or inertia pump with optional surgeblock fitting as appropriate. Accumulated sediment in the bottom of the well (if present) will be removed by bailing with a bottom-loading bailer or via pumping using a submersible or inertia pump with optional surge-block fitting. Wells will also be gently brushed with a weighted brush to assist in removing loose debris, silt or flock attached to the inside of the well riser and/or screen prior to development. Pumping methods will be selected based on site-specific geologic conditions, anticipated well yield, water table depth, and groundwater monitoring objectives, and may include one or more of the following:

- submersible pump
- inertial pump (Waterra™ pump or equivalent)
- bladder pump
- peristaltic pump
- centrifugal pump

When developing a well using the pumping method, the pump (or, with inertial pumps, the tubing) is lowered to the screened portion of the well. During purging, the pump or tubing is moved up and down the screened interval until the well yields relatively clear water.

Submersible pumps have a motor-driven impeller that pushes the groundwater through discharge tubing to the ground surface. Inertial pumps have a check valve at the bottom of stiff tubing which, when operated up and down, lifts water to the ground surface. Bladder pumps have a bottom check valve and a flexible internal bladder that fills from below and is then compressed using pressurized air to force water out the top of the bladder through the discharge tubing to the ground surface. These three types of pumps have a wide range of applicability in terms of well depth and water depth.

Centrifugal and peristaltic pumps use atmospheric pressure to lift water from the well, and therefore can only be practically used where the depth to water is less than 25 feet.

II. Personnel Qualifications

Monitoring well development activities will be performed by persons who have been trained in proper well development procedures under the guidance of an experienced field geologist, engineer, or technician.

III. Equipment List

Materials for monitoring well development using a pump include the following:

- health and safety equipment, as required by the site Health and Safety Plan (HASP):
- cleaning equipment
- photoionization detector (PID) to measure headspace vapors
- pump
- polyethylene pump discharge tubing
- plastic sheeting
- power source (generator or battery)
- field notebook and/or personal digital assistant (PDA)
- graduated pails
- appropriate containers

- monitoring well keys
- water level indicator

Materials for monitoring well development using a bailer include the following:

- personal protective equipment (PPE) as required by the HASP
- cleaning equipment
- PID to measure headspace vapors
- bottom-loading bailer, sand bailer
- polypropylene or nylon rope
- plastic sheeting
- graduated pails
- appropriate containers
- keys to wells
- field notebook and/or PDA
- water level indicator
- weighted brush for well brushing

IV. Cautions

Where surging is performed to assist in removing fine-grained material from the sand pack, surging must be performed in a gentle manner. Excessive suction could promote fine-grained sediment entry into the outside of the sand pack from the formation.

Avoid using development fluids or materials that could impact groundwater or soil quality, or could be incompatible with the subsurface conditions.

In some cases it may be necessary to add potable water to a well to allow surging and development, especially for new monitoring wells installed in low permeability formations. Before adding potable water to a well, the Project Manager (PM) must be notified and the PM shall make the decision regarding the appropriateness and applicability of adding potable water to a well during well development procedures. If potable water is to be added to a well as part of development, the potable water source should be sampled and analyzed for constituents of concern, and the results evaluated by the PM prior to adding the potable water to the well. If potable water is added to a well for development purposes, at the end of development the well will be purged dry to remove the potable water, or if the well no longer goes dry then the well will be purged to remove at least three times the volume of potable water that was added.

V. Health and Safety Considerations

Field activities associated with monitoring well development will be performed in accordance with a site-specific HASP, a copy of which will be present on site during such activities.

VI. Procedure

The procedures for monitoring well development are described below. (Note: Steps 7, 8, and 10 can be performed at the same time using an inertial pump with a surge-block fitting.)

1. Don appropriate PPE (as required by the HASP).

2. Place plastic sheeting around the well.

3. Clean all equipment entering each monitoring well, except for new, disposable materials that have not been previously used.

4. Open the well cover while standing upwind of the well, remove well cap. Insert PID probe approximately 4 to 6 inches into the casing or the well headspace and cover with gloved hand. Record the PID reading in the field notebook. If the well headspace reading is less than 5 PID units, proceed; if the headspace reading is greater than 5 PID units, screen the air within the breathing zone. If the PID reading in the breathing zone is below 5 PID units, proceed. If the PID reading is above 5 PID units, move upwind from well for 5 minutes to allow the volatiles to dissipate. Repeat the breathing zone test. If the reading is still above 5 PID units, don the appropriate respiratory protection in accordance with the requirements of the HASP. Record all PID readings.

5. Obtain an initial measurement of the depth to water and the total well depth from the reference point at the top of the well casing. Record these measurements in the field log book.

6. Prior to redeveloping older wells that may contain solid particulate debris along the inside of the well casing and screen, gently lower and raise a weighted brush along the entire length of the well screen and riser to free and assist in removing loose debris, silt or flock. Perform a minimum of 4 “passes” along the screened and cased intervals of the well below the static water level in the well. Allow the resulting suspended material to settle for a minimum of one day prior to continuing with redevelopment activities.

7. Lower a surge block or bailer into the screened portion of the well. Gently raise and lower the surge block or bailer within the screened interval of the well to force water in and out of the screen slots and sand pack. Continue surging for 15 to 30 minutes.

8. Lower a bottom-loading bailer, submersible pump, or inertia pump tubing with check valve to the bottom of the well and gently bounce the bailer, pump, pump tubing on the bottom of the well to collect/remove accumulated sediment, if any. Remove and empty the bailer, if used. Repeat until the bailed/pumped water is free of excessive sediment and the bottom of the well feels solid. Alternatively, measurement of the well depth with a water level indicator can be used to verify that sediment and/or silt has been removed to the extent practicable, based on a comparison with the well installation log or previous measurement of total well depth.

9. After surging the well and removing excess accumulated sediment from the bottom of the well, re-measure the depth-to-water and the total well depth from the reference point at the top of the well casing. Record these measurements in the field log book.

10. Remove formation water by pumping or bailing. Where pumping is used, measure and record the pre-pumping water level. Operate the pump at a relatively constant rate. Measure the pumping rate using a calibrated container and stop watch, and record the pumping rate in the field log book. Measure and record the water level in the well at least

once every 5 minutes during pumping. Note any relevant observations in terms of water color, visual level of turbidity, sheen, odors, etc. Pump or bail until termination criteria specified in the Field Sampling Plan (FSP) are reached. Record the total volume of water purged from the well.

11. If the well goes dry, stop pumping or bailing and allow well to recover. Resume pumping or bailing when sufficient water has recharged the well.

12. Contain all water in appropriate containers.

13. When complete, secure the lid back on the well.

14. Place disposable materials in plastic bags for appropriate disposal and decontaminate reusable, downhole pump components and/or bailer.

VII. Waste Management

Materials generated during monitoring well installation and development will be placed in appropriate labeled containers and disposed of as described in the Work Plan or Field Sampling Plan.

VIII. Data Recording and Management

Well development activities will be documented in a proper field notebook and/or PDA. Pertinent information will include personnel present on site; times of arrival and departure; significant weather conditions; timing of well development activities; development method(s); observations of purge water color, turbidity, odor, sheen, etc.; purge rate; and water levels before and during pumping.

IX. Quality Assurance

All reused, non-disposable, downhole well development equipment will be cleaned in accordance with the procedures outlined in the Field Equipment Cleaning-Decontamination SOP.

X. References

Not applicable.

ARCADIS

MONITORING WELL DEVELOPMENT LOG

Sampling Personnel:

Job Number:

Weather:

Well ID.

Date:

Time In: **Time Out:**

WELL INFORMATION

check where appropriate

		TIC	TOC	BGS
Well Depth	(feet)			
Water Table Depth	(feet)			

Well Type: Flushmount ☐ Stick-Up ☐

Well Locked: Yes ☐ No ☐

Measuring Point Marked: Yes ☐ No ☐

Well Diameter: 1" ☐ 2" ☐ Other: _____

WELL WATER INFORMATION

Length of Water Column:	(feet)	
Volume of Water in Well:	(gal)	
Pumping Rate of Pump:	(mL/min)	
Pumping Rate of Pump:	(GPM)	
Minutes of Pumping:	(min)	
Total Volume Removed:	(gal)	

Conversion Factors

gallons per feet	1" ID	2" ID	4" ID	6" ID
of water column:	0.041	0.163	0.653	1.469

1 gal = 3.785 L =3785 mL = 0.1337 cubic ft.

Well Brushing Method and Duration:	
Well Screen Surging Method and Duration:	

EVACUATION INFORMATION

Evacuation Method: Bailer ☐ Peristaltic ☐ Grunfos ☐ WaTerra ☐ Other Pump ☐ _______________

Tubing Used: Teflon ☐ Polyethylene ☐

Did well go dry? Yes ☐ No ☐

Water Quality Meter Type: ____________________

Time Parameter	1 Initial	2	3	4	5	6	7	8	9
Volume Purged (ml)									
Depth to Water (ft. TIC)									
Temperature (°C)									
pH									
Conductance (mS/cm)									
Dissolved Oxygen (mg/L)									
Turbidity (NTU)									
ORP (mV)									

Time Parameter	10	11	12	13	14
Volume Purged (ml)					
Depth to Water (ft. TIC)					
Temperature (°C)					
pH					
Conductance (mS/cm)					
Dissolved Oxygen (mg/L)					
Turbidity (NTU)					
ORP (mV)					

MISCELLANEOUS OBSERVATIONS/NOTES

Attachment 3J

Photoionization Detector Air Monitoring and Field Screening

Photoionization Detector Air Monitoring and Field Screening

Rev. #: 1

Rev Date: November 8, 2009

Approval Signatures

Prepared by: (the late) Maureen Geisser Date: July 28, 2003

Christopher C Lutes

Reviewed/revised by: Christopher C. Lutes Date: November 8, 2009
(Technical Expert)

I. Scope and Application

Field screening with a photoionization detector (PID), such as an HNu™, Photovac™, MicroTIP™, or MiniRAE™, is a procedure to measure relative concentrations of volatile organic compounds (VOCs) and other compounds. Characteristics of the PID are presented in Attachment 1 and the compounds a PID can detect are presented in Attachment 2. Field screening will frequently be conducted on the following:

- Work area air to assess exposure to on-site workers of air contaminants via the air pathway;
- Well headspaces as a precautionary measure each time the well cover is opened; and
- Headspace of soil samples to assess the relative concentration of volatile organics in the sample or to select particular intervals for off-site analysis for VOCs.

II. Personnel Qualifications

Personnel performing this method should be familiar with the basic principles of quantiative analytical chemistry (such as calibration) and familiar with the particular operation of the instrument to be used.

III. Equipment List

The following materials, as required, shall be available while performing PID field screening:

- personal protective equipment (PPE), as required by the site Health and Safety Plan (HASP);
- PID and operating manual;
- PID extra battery pack and battery charger;
- calibration canisters for the PID;
- sample jars;
- Q-tips;

- aluminum foil;
- field calibration log (attached); and
- field notebook.

IV. Cautions

PIDs are sensitive to moisture and may not function under high humidity. PIDs cannot be used to indicate oxygen deficiency or combustible gases.

V. Health and Safety Considerations

Since the PIDs cannot detect all of the chemicals that may be present at a sample location, a zero reading on either instrument does not necessarily signify the absence of air contaminants. PIDs cannot be used as an indicator for oxygen deficiency.

VI. Procedure (*Note these procedures were written particular to one specific instrument model, therefore please also refer to your owners manual.Hhowever the general principles – such as always measuring both a zero and span gas after an instrument adjustment/at the beginning of the analytical day, after four hours of testing and again at the end of an analytical day can be applied to all instruments.)*

PID Calibration

PID field instruments will be calibrated and operated to yield “total organic vapor” in parts per million (ppm) (v/v) relative to benzene or isobutylene (or equivalent). Operation, maintenance, and calibration shall be performed in accordance with the manufacturer’s instructions and entered on the PID calibration and maintenance log (Attachment 3).

1. Don PPE, as required by the HASP.
2. Perform a BATTERY CHECK. Turn the FUNCTION switch to the BATTERY CHECK position. Check that the indicator is within or beyond the green battery arc. If battery is low, the battery must be charged before calibration.
3. Allow the instrument to warm up, then calibrate the PID. If equipped, turn the FUNCTION switch to the STANDBY position and rotate the ZERO

POTENTIOMETER until the meter reads zero with the instrument sampling clean air. Wait 15 to 20 seconds to confirm the adjustment. If unstable, readjust. If equipped, check to see that the SPAN POTENTIOMETER is adjusted for the probe being used (e.g., 9.8 for 10.2 electron volts [eV]). Set the FUNCTION switch to the desired ppm range (0-20, 0-200, or 0-2,000). A violet glow from the ultraviolet (UV) source should be visible at the sample inlet of the probe/sensor unit.

4. Listen for the fan operation to verify fan function.

5. Connect one end of the sampling hose to the calibration canister regulator outlet and the other end to the sampling probe of the PID. Crack the regulator valve and take a reading after 5 to 10 seconds. Adjust the span potentiometer to produce the concentration listed on the span gas cylinder. Record appropriate information on a PID Calibration and Maintenance Log (Attachment 3, or equivalent).

6. If so equipped, set the alarm at desired level.

7. Recheck the zero with fresh/clean air

8. Always recheck both zero and span after making any instrment adjustment, after four hours of screenign work and again after sample analysis.

Work Area Air Monitoring

1. Measure and record the background PID reading.

2. Measure and record the breathing space reading.

Well Headspace Screening

1. Measure and record the background PID reading.

2. Unlock and open the well cover while standing upwind of the well.

3. Remove the well cap.

4. Place the PID probe approximately 6 inches above the top of the casing.

5. Record all PID readings and proceed in accordance with the HASP.

Field Screening Procedures

Soil samples will be field screened upon collection with the PID for a relative measure of the total volatile organic concentration. The following steps define the PID field screening procedures.

1. Half-fill two clean glass jars with the sample (if sufficient quantities of soil are available) to be analyzed. Quickly cover each open top with one or two sheets of clean aluminum foil and subsequently apply screw caps to tightly seal the jars. Sixteen-ounce (approximately 500 mL) soil or “mason” type jars are preferred; jars less than 8 ounces (approximately 250 mL) total capacity may not be used.

2. Allow headspace development for at least 10 minutes. Vigorously shake jars for 15 seconds at both the beginning and end of the headspace development period. Where ambient temperatures are below 32°F (0°C), headspace development should be within a heated building.

3. Subsequent to headspace development, remove screw lid to expose the foil seal. Quickly puncture foil seal with instrument sampling probe, to a point about one-half of the headspace depth. Exercise care to avoid contact with water droplets or soil particulates.

4. Following probe insertion through foil seal, record the highest meter response for each sample as the jar headspace concentration. Using the foil seal/probe insertion method, maximum response should occur between 2 and 5 seconds. Erratic meter response may occur at high organic vapor concentrations or conditions of elevated headspace moisture, in which case headspace data should be recorded and erratic meter response noted.

5. The headspace screening data from both jar samples should be recorded and compared; generally, replicate values should be consistent to plus or minus 20%. It should be noted that in some cases (e.g., 6-inch increment soil borings), sufficient sample quantities may not be available to perform duplicate screenings. One screening will be considered sufficient for this case.

6. PID field instruments will be operated and calibrated to yield “total organic vapors” in ppm (v/v) as benzene. PID instruments must be operated with at least a 10.0 eV (+) lamp source. Operation, maintenance, and calibration will be performed in accordance with the manufacturer’s specifications presented in Attachment 12-1. For jar headspace analysis, instrument calibration will be checked/adjusted at least twice per day, at the beginning and end of each day

of use. Calibration will exceed twice per day if conditions and/or manufacturer's specifications dictate.

7. Instrumentation with digital (LED/LCD) displays may not be able to discern maximum headspace response unless equipped with a "maximum hold" feature or strip-chart recorder.

VII. Waste Management

Do not dispose canisters of compressed gas, if there is still compressed gas in the canister. Return the canister to the manufactuer for proper disposal.

VIII. Data Recording and Management

Measurements will be record in the field notebook or boring logs at the time of measurement with notation of date, time, location, depth (if applicable), and item monitored. If a data memory is available, readings will be downloaded from the unit upon access to a computer with software to retrieve the data.

IX. Quality Assurance

After each use, the readout unit should be wiped down with a clean cloth or paper towel.

For a HNu, the UV light source window and ionization chamber should be cleaned once a month in the following manner:

1. With the PID off, disconnect the sensor/probe from the unit.

2. Remove the exhaust screw, grasp the end cap in one hand and the probe shell in the other, and pull apart.

3. Loosen the screws on top of the end cap and separate the end cap and ion chamber from the lamp and lamp housing.

4. Tilt the lamp housing with one hand over the opening so that the lamp slides out into your hand.

5. Clean the lamp with lens paper and HNu cleaning compound (except 11.7 eV). For the 11.7 eV lamp, use a chlorinated organic solvent.

6. Clean the ion chamber using methanol on a Q-tip and then dry gently at 50ºC to 60ºC for 30 minutes.

7. Following cleaning, reassemble by first sliding the lamp back into the lamp housing. Place ion chamber on top of the housing, making sure the contacts are properly aligned.

8. Place the end cap on top of the ion chamber and replace the two screws (tighten the screws only enough to seal the o-ring).

9. Line up the pins on the base of the lamp housing with pins inside the probe shell and slide the housing assembly into the shell.

X. References

Denahan, S.A. et. all *“*Relationships Between Chemical Screening Methodologies for Petroleum Contaminated Soils: Theory and Pracice” *Chapter 5 In Principles and Practices for Petroleum Contaminated Soils,* E.J. Calabrese and P.T. Kostecki Eds., Lewis Publishers 1993.

Fitzgerald, J. “Onsite Analytical Screening of Gasoline Contaminated Media Using a Jar Headspace Procedure” Chapter 4 *in Principles and Practices for Petroleum Contaminated Soils,* E.J. Calabrese and P.T. Kostecki Eds., Lewis Publishers 1993.

ATTACHMENT 1

Characteristics of the Photoionization Detector (PID)

I. Introduction

PIDs are used in the field to detect a variety of compounds in air. PIDs can be used to detect leaks of volatile substances in drums and tanks, to determine the presence of volatile compounds in soil and water, and to make ambient air surveys. If personnel are thoroughly trained to operate the instrument and interpret the data, these PID instruments can be a valuable tool. Its use can help in deciding the level of protection to be worn, assist in determining the implementation of other safety procedures, and in determining subsequent monitoring or sampling locations.

Portable PIDs detect the concentration of organic gases, as well as a few inorganic gases. The basis for detection is the ionization of gaseous species. The incoming gas molecules are subjected to UV radiation, which ionizes molecules that have an ionization potential (IP) less than or equal to that rated for the UV source. Every molecule has a characteristic IP, which is the energy required to remove an electron from the molecule, thus yielding a positively charged ion and the free electron. These ions are attracted to an oppositely charged electrode, causing a current and an electric signal to the LED display. Compounds are measured on a ppm volume basis.

II. HNu PI-101 / MiniRAE or Equivalent PID

The PIDs detect the concentration of organic gases, as well as a few inorganic gases. The basis for detection is the ionization of gaseous species. The incoming gas molecules are subjected to UV radiation, which is energetic enough to ionize many gaseous compounds. Each molecule is transformed into charged ion pairs, creating a current between two electrodes. Every molecule has a characteristic IP, which is the energy required to remove an electron from the molecule, yielding a positively charged ion and the free electron.

Three probes, each containing a different UV light source, are available for use with the PID. Probe energies are typically 9.5, 10.2, and 11.7 eV, respectively. All three probes detect many aromatic and large-molecule hydrocarbons. In addition, the 10.2 eV and 11.7 eV probes detect some smaller organic molecules and some halogenated hydrocarbons. The 10.2 eV probe is the most useful for environmental response work, as it is more durable than the 11.7 eV probe and detects more compounds than the 9.5 eV probe. A listing of molecules and compounds that the HNu can detect is presented in Attachment 2.

The primary PID calibration gas is either benzene or isobutylene. The span potentiometer knob is turned to 9.8 for benzene calibration. A knob setting of zero increases the sensitivity to benzene approximately 10-fold. Its lower detection limit is in the low ppm range. Additionally, response time is rapid; the dot matrix liquid crystal displays 90% of the indicated concentration within 3 seconds.

III. Limitations

The PID instrument can monitor several vapors and gases in air. Many non-volatile liquids, toxic solids, particulates, and other toxic gases and vapors, however, cannot be detected with PIDs (such as methane). Since the PIDs cannot detect all of the chemicals that may be present at a sample location, a zero reading on either instrument does not necessarily signify the absence of air contaminants.

The PID instrument is generally not specific and their response to different compounds is relative to the calibration gases. Instrument readings may be higher or lower than the true concentration. This effect can be observed when monitoring total contaminant concentrations if several different compounds are being detected at once. In addition, the response of these instruments is not linear over the entire detection range. Therefore, care must be taken when interpreting the data. Concentrations should be reported in terms of the calibration gas and probe type.

PIDs are small, portable instruments and may not yield results as accurate as laboratory instruments. PIDs were originally designed for specific industrial applications. They are relatively easy to use and interpret when detecting total concentrations of known contaminants in air, but interpretation becomes more difficult when trying to identify the individual components of a mixture. PIDs cannot be used as an indicator for combustible gases or oxygen deficiency.

ATTACHMENT 2

Molecules and Compounds Detected by a PID

Some Atoms and Simple Molecules

	IP(eV)		**IP(eV)**
H	13.595	I_2	9.28
C	11.264	HF	15.77
N	14.54	HCl	12.74
O	13.614	HBr	11.62
Si	8.149	HI	10.38
S	10.357	SO_2	12.34
F	17.42	CO_2	13.79
Cl	13.01	COS	11.18
Br	11.84	CS_2	10.08
I	10.48	N_2O	12.90
H_2	15.426	NO_2	9.78
N_2	15.580	O_3	12.80
O_2	12.075	H_2O	12.59
CO	14.01	H_2S	10.46
CN	15.13	H_2Se	9.88
NO	9.25	H_2Te	9.14
CH	11.1	HCN	3.91
OH	13.18	C_2N_2	13.8
F_2	15.7	NH_3	10.15
Cl_2	11.48	CH_3	9.840
Br_2	10.55	CH_4	12.98

Paraffins and Cycloparaffins

Molecule	**IP(eV)**
methane	12.98
ethane	11.65
propane	11.07
n-butane	10.63
i-butane	10.57
n-pentane	10.35
i-pentane	10.32
2,2-dimethylpropane	10.35
n-hexane	10.18
2-methlypentane	10.12
3-methlypentane	10.08
2,2-dimethlybutane	10.06
2,3-dimethlybutane	10.02
n-heptane	10.08
2,2,4-trimethlypentane	9.86
cyclopropane	10.06
cyclopentane	10.53
cyclohexane	9.88
methlycyclohexane	9.8

Alkyl Halides

IP(eV)	IP(eV)
HCl	12.74
Cl_2	11.48
CH_4	12.98
methyl chloride	11.28
dichloroemethane	11.35
trichloromethane	11.42
tetrachloromethane	11.47
ethyl chloride	10.98
1,2-dichloroethane	11.12
1-chloropropane	10.82
2-chloropropane	10.78
1,2-dichloropropane	10.87
1,3-dichloropropane	10.85
1-chlorobutane	10.67
2-chlorobutane	10.65
1-chloro-2-methylpropane	10.66
2-chloro-2-methylpropane	10.61
HBr	11.62
Br_2	10.55
methyl bromide	10.53
dibromomethane	10.49
tribromomethane	10.51
CH_2BrCl	10.77
$CHBr_2Cl$	10.59
ethyl bromide	10.29
1,1-dibromoethane	10.19
1-bromo-2-chloroethane	10.63
1-bromopropane	10.18
2-bromopropane	10.075
1,3-dibromopropane	10.07
1-bromobutane	10.13
2-bromobutane	9.98
1-bromo-2-methylpropane	10.09
2-bromo-2-methylpropane	9.89
1-bromopentane	10.10
HI	10.38
I_2	9.28

Alkyl Halides

Molecule	IP(eV)
methyl iodide	9.54
diiodomethane	9.34
ethyl iodide	9.33
1-iodopropane	9.26
2-iodopropane	9.17
1-iodobutane	9.21
2-iodobutane	9.09
1-iodo-2-methylpropane	9.18
2-iodo-2-methylpropane	9.02
1-iodopentane	9.19
F_2	15.7
HF	15.77
$CFCl_3$ (Freon 11)	11.77
CF_2Cl_2 (Freon 12)	12.31
CF_3Cl (Freon 13)	12.91
$CHClF_2$ (Freon 22)	12.45
$CFBR_3$	10.67
CF_2Br_2	11.07
CH_3CF_2Cl (Genetron 101)	11.98
$CFCl_2CF_2Cl$	11.99
CF_3CCl_3 (Freon 113)	11.78
$CFHBrCH_2Cr$	10.75
CF_2BrCH_2Br	10.83
CF_3CH_2I	10.00
n-C_3F_7I	10.36
n-$C_3F_7CH_2Cl$	11.84
n-$C_3F_7CH_2I$	9.96

Aliphatic Alcohol, Ether, Thiol, and Sulfides

Molecule	IP(eV)
H_2O	12.59
methyl alcohol	10.85
ethyl alcohol	10.48
n-propyl alcohol	10.20
i-propyl alcohol	10.16
n-butyl alcohol	10.04
dimethyl ether	10.00
diethyl ether	9.53
n-propyl ether	9.27
i-propyl ether	9.20
H_2S	10.46
methanethiol	9.440
ethanethiol	9.285
1-propanethiol	9.195
1-butanethiol	9.14
dimethyl sulfide	8.685
ethyl methyl sulfide	8.55
diethyl sulfide	8.430
di-n-propyl sulfide	8.30

Aliphatic Aldehydes and Ketones

Molecule	**IP(eV)**
CO_2	13.79
formaldehyde	10.87
acetaldehyde	10.21
propionaldehyde	9.98
n-butyraldehyde	9.86
isobutyraldehyde	9.74
n-valeraldehyde	9.82
isovaleraldehyde	9.71
acrolein	10.10
crotonaldehyde	9.73
benzaldehyde	9.53
acetone	9.69
methyl ethyl ketone	9.53
methyl n-propyl ketone	9.39
methyl i-propyl ketone	9.32
diethyl ketone	9.32
methyl n-butyl ketone	9.34
methyl i-butyl ketone	9.30
3,3-dimethyl butanone	9.17
2-heptanone	9.33
cyclopentanone	9.26
cyclohexanone	9.14
2,3-butanedione	9.23
2,4-pentanedione	8.87

Aliphatic Acids and Esters

Molecule	**IP(eV)**
CO_2	13.79
formic acid	11.05
acetic acid	10.37
propionic acid	10.24
n-butyric acid	10.16
isobutyric acid	10.02
n-valeric acid	10.12
methyl formate	10.815
ethyl formate	10.61
n-propyl formate	10.54
n-butyl formate	10.50
isobutyl formate	10.46
methyl acetate	10.27
ethyl acetate	10.11
n-propyl acetate	10.04
isopropyl acetate	9.99
n-butyl acetate	10.01
isobutyl acetate	9.97
sec-butyl acetate	9.91
methyl propionate	10.15
ethyl propionate	10.00
methyl n-butyrate	10.07
methyl isobutyrate	9.98

Aliphatic Amines and Amides

Molecule	IP(eV)
NH_3	10.15
methyl amine	8.97
ethyl amine	8.86
n-propyl amine	8.78
i-propyl amine	8.72
n-butyl amine	8.71
i-butyl amine	8.70
s-butyl amine	8.70
t-butyl amine	8.64
dimethyl amine	8.24
diethyl amine	8.01
di-n-propyl amine	7.84
di-i-propyl amine	7.73
di-n-butyl amine	7.69
trimethyl amine	7.82
triethyl amine	7.50
tri-n-propyl amine	7.23
formamide	10.25
acetamide	9.77
N-methyl acetamide	8.90
N,N-dimethyl formamide	9.12
N,N-dimethyl acetamide	8.81
N,N-diethyl formamide	8.89
N,N-diethyl acetamide	8.60

Other Aliphatic Molecules with N Atom

Molecule	IP(eV)
nitromethane	11.08
nitroethane	10.88
1-nitropropane	10.81
2-nitropropane	10.71
HCN	13.91
acetonitrile	12.22
propionitrile	11.84
n-butyronitrile	11.67
acrylonitrile	10.91
3-butene-nitrile	10.39
ethyl nitrate	11.22
n-propyl nitrate	
methyl thiocyanate	10.065
ethyl thiocyanate	9.89
methyl isothiocyanate	9.25
ethyl isothiocyanate	9.14

Olefins, Cyclo-ofefins, Acetylenes

Molecule	IP(eV)
ethylene	10.515
propylene	9.73
1-butene	9.58
2-methylpropene	9.23
trans-2-butene	9.13
cis-2-butene	9.13
1-pentene	9.50
2-methyl-1-butene	9.12
3-methyl-1-butene	9.51
3-methyl-2-butene	8.67
1-hexene	9.46
1,3-butadiene	9.07
isoprene	8.845
cyclopentene	9.01
cyclohexene	8.945
4-methylcyclohexene	8.91
4-cinylcylohexene	8.93
cyclo-octatetraene	7.99
acetylene	11.41
propyne	10.36
1-butyne	10.18

Some Derivatives of Olefins

Molecule	IP(eV)
vinyl chloride	9.995
cis-dichloroethylene	9.65
trans-dichloroethylene	9.66
trichloroethylene	9.45
tetrachloroethylene	9.32
vinyl bromide	9.80
1,2-dibromoethylene	9.45
tribromoethylene	9.27
3-chloropropene	10.04
2,3-dichloropropene	9.82
1-bromopropene	9.30
3-bromopropene	9.7
$CF_3CCl{=}CClCF_3$	10.36
$n\text{-}C_5F_{11}CF{=}CF_2$	10.48
acrolein	10.10
crotonaldehyde	9.73
mesityl oxide	9.08
vinyl methyl ether	8.93
allyl alcohol	9.67
vinyl acetate	9.19

Aromatic Compounds

Molecule	IP(eV)
benzene	9.245
toluene	8.82
ethyl benzene	8.76
n-propyl benzene	8.72
i-propyl benzene	8.69
n-butyl benzene	8.69
s-butyl benzene	8.68
t-butyl benzene	8.68
o-xylene	8.56
m-xylene	8.56
p-xylene	8.445
mesitylene	8.40
durene	8.025
styrene	8.47
alpha-methyl styrene	8.35
ethynylbenzene	8.815
naphthalene	8.12
1-methylnapthalene	7.69
2-methylnapthalene	7.955
biphenyl	8.27
phenol	8.50
anisole	8.22
phenetole	8.13
benzaldehyde	9.53
acetophenone	9.27
benzenethiol	8.33
phenyl isocyanate	8.77

Aromatic Compounds

Molecule	IP(eV)
phenyl isothiocyanate	8.520
benzonitrile	9.705
nitrobenzene	9.92
aniline	7.70
fluoro-benzene	9.195
chloro-benzene	9.07
bromo-benzene	8.98
iodo-benzene	8.73
o-dichlorobenzene	9.07
m-dichlorobenzene	9.12
p-dichlorobenzene	8.94
1-chloro-2-fluorobenzene	9.155
1-chloro-3-fluorobenzene	9.21
1-chloro-4-fluorobenzene	8.99
o-fluorotoluene	8.915
m-fluorotoluene	8.915
p-fluorotoluene	8.785
o-chlorotoluene	8.83
m-chlorotoluene	8.83
p-chlorotoluene	8.70
o-bromotoluene	8.79
m-bromotoluene	8.81
p-bromotoluene	8.67
o-iodotoluene	8.62
m-iodotoluene	8.61
p-iodotoluene	8.50
benzotrifluoride	9.68
o-fluorophenol	8.66

Heterocyclic Molecules

Molecule	IP(eV)
furan	8.89
2-methyl furan	8.39
2-furaldehyde	9.21
tetrahydrofuran	9.54
dihydropyran	8.34
tetrahydropyran	9.26
thiophene	8.860
2-chlorothiophene	8.68
2-bromothiophene	8.63
pyrrole	8.20
pyridine	9.32
2-picoline	9.02
3-picoline	9.04
4-picoline	9.04
2,3-lutidine	8.85
2,4-lutidine	8.85
2,6-lutidine	8.85

Miscellaneous Molecules

Molecule	IP(eV)
ethylene oxide	10.565
propylene oxide	10.22
p-dioxane	9.13
dimethoxymethane	10.00
diethoxymethane	9.70
1,1-dimethoxyethane	9.65
propiolactone	9.70
methyl disulfide	8.46
ethyl disulfide	8.27
diethyl sulfite	9.68
thiolacetic acid	10.00
acetyl chloride	11.02
acetyl bromide	10.55
cyclo-$C_6H_{11}CF_3$	10.46
$(n\text{-}C_3F_7)(CH_3)C{=}O$	10.58
trichlorovinylsilane	10.79
$(C_2F_5)_3N$	11.7
isoprene	9.08
phosgene	11.77

Notes:

Reference: HNu Systems, Inc., 1985
IP = Ionization Potential

ATTACHMENT 3

PID CALIBRATION AND MAINTENANCE LOG

Instrument Model Number

Instrument Serial Number

Calibration Gas **ppm**

Date/Time	Initials	Battery Check	Calibration			
			Background Value	True Gas Value	Measured Gas Value	Adjust

COMMENTS:

Attachment 3K
Pump Test Standard Operating Procedures

ARCADIS
Infrastructure, environment, facilities

Imagine the result

PUMPING TEST
STANDARD OPERATING PROCEUDRES

Rev. #: 01

Rev Date: September 2008

Approval Signatures

Goston Lee

Prepared by: ______________________________ Date: September 29, 2008

Goston Lee

Reviewed by: ______________________________ Date: September 29, 2008
(Technical Expert)

I. Test Design

In general conventional hydraulic testing is conducted to provide answers to questions related to water supply problems. Tests are conducted over longer periods of time and provide estimates of hydraulic conductivity values averaged over large aquifer volumes. These tests tend to underestimate the highest hydraulic conductivity values and overestimate the lowest.

When conducting tests for remediation hydrogeology purposes it is important to indentify aquifer heterogeneities which ultimately control the transport of contaminants and reagents distribution within the aquifer. Short-term tests may help identify particular depositional elements and hydraulic conductivity trends and variability associated with facies changes in the aquifer. Data collected from short-term test can then be correlated with detailed hydrostratigraphic information to assist in the development of conceptual site models that describe the transport of contaminants and distribution of reagents.

1. Understand Aquifer Conditions

An aquifer (or permeable zone) pumping test is conducted in order to determine the hydraulic properties (transmissivity, hydraulic conductivity, storage coefficient, leakage, boundaries, anisotropy) of a water-bearing zone or system (including confining beds). Proper design of a pumping test requires a general understanding of the potential hydrologic system prior to the test, so that suitable data are collected to evaluate system parameters. The designer of the test must first develop an appropriate set of assumptions (conceptual model), either taken from previous tests in the immediate area or from well logs and an assessment of the site features that can affect the test (soil or rock types, depth to water, surface- water bodies, existing wells, storm drains). This conceptual model will then help the designer anticipate the necessary design factors such as: number of wells, depth and placement of wells; pumping rate(s); frequency of water-level measurements; and length of pumping. These factors will help the designer determine from the test results the effects of recharge and restrictive boundaries, aquifer geometry, secondary porosity effects (fractures, solution channels), the nature and extent of potentially confining layers, and aquifer interconnections.

2. Estimate Aquifer Parameters

Although the objective of a pumping test is to determine the principal aquifer parameters, the conceptual model requires a prediction of some of these parameters for the design process (i.e., observation well number and spacing requires approximate transmissivity and storage coefficient values). Hydraulic conductivity may be estimated from textural or hydraulic testing of aquifer materials in the laboratory, or from data collected and observations made during drilling or well development (see Driscoll, 1986). Considerable experience is needed to apply these methods for anything but preliminary estimating purposes. Therefore, use as many approaches as possible when making these estimates and remember that they are only estimates. Be ready to adjust preliminary estimates as more information becomes available throughout the process.

For larger tests (and thus larger pumping wells), potential casing storage effects and well (friction and formation) loss may need to be calculated prior to the test. Also, optimum pump size may need to be calculated. These will require an estimate of specific capacity, which is the well discharge rate per unit of drawdown measured at a given time. Specific capacity is typically determined from a step-drawdown test. An added benefit to conducting a step-drawdown test is the graphical results can also be used to calculate transmissivity (but not storage coefficient) in addition to well losses (see Section B.9).

3. Locate the Pumped Well

At many sites, the pumping well location is predetermined because an existing well suits the needs of the test, or the hydraulic properties of a specific location must be measured. If the pumping well location can be selected with relative freedom, the following criteria can be used as a guide for its installation:

a) where the hydrogeology represents the area of interest;
b) proximity to existing wells that could be used as observation wells (see guidelines 5, 6 and 7 below);
c) within the targeted contaminant plume whenever possible;
d) outside the contaminant plume if the system is areally homogeneous (or nearly so) and pumping of contaminated water poses an insurmountable problem;
e) away from groundwater system boundaries (assuming their approximate position is known) when the test purpose is solely to measure aquifer storage and transmission properties;
f) close to groundwater system boundaries (assuming their approximate position is known) when requiring boundary location, orientation (both positive and negative boundaries), or degree of connection (positive boundaries);
g) away from surface features that could obscure the data (for example, surface-water bodies) and away from areas subject to heavy-equipment traffic (i.e., railways and highways) that would put unpredictable stress on the aquifer, unless desiring specific information about the interrelationship of the groundwater system and surface features;
h) away from other producing wells that may not be shut down and may affect test data; and
i) where the site is safely and easily accessible to equipment and personnel.

Although these guidelines generally support test success, strictly adhering to them may produce conflicting test designs. Resolving these design conflicts requires good judgment based on a clear understanding of the test priorities and an appropriate knowledge of the local groundwater system.

4. Design the Pumped Well

a) The casing must accommodate the pump used for the test and allow ample additional space for measuring equipment.

b) The pumped well should be as efficient as possible through sound drilling practices, installation, and construction. A wire-wrapped screen and site-specific filter pack, designed from a sieve analysis, should be used to reduce factors that will mask true aquifer response.

c) If possible, a stilling pipe should be installed in the pumped well for making water- level measurements. The stilling pipe will dampen water-level fluctuations caused by pump vibration, eliminate measurement errors associated with cascading water, and isolate pressure transducers from pressure transients near the pump intake.

d) Generally, the screen in the pumped well should fully penetrate the tested zone to eliminate the complicated data analysis and interpretation required to correct for partial penetration effects (induced vertical flow component in addition to radial flow), with the following two exceptions:

   1) if the screen would form a conduit capable of transmitting chemicals from a contaminated horizon to a clean horizon; or

   2) when attempting to determine an aquifer's vertical anisotropy (ratio of vertical to horizontal hydraulic conductivity). This determination is necessary if remediation well capture zones will not affect the full thickness of the aquifer.

For these two conditions, the pumped well should only penetrate the contaminated portion of the aquifer. In addition, cost considerations may limit full penetration of the tested zone.

e) The pumped well must be fully developed to maximize the pumping rate from wells with limited available drawdown, simplify data interpretation and assure that no additional development occurs during the test.

f) Often, pumping wells are later used as monitoring or recovery wells. Such wells should be designed according to the requirements of the particular application without compromising the aforementioned standards for pumped wells.

5. Determine the Number of Observation Wells

Observation wells help quantify the size, shape, position, and rate of change of the cone of depression formed by pumping, making it possible to determine aquifer parameters. Adding wells increases the amount and accuracy of information acquired, and improves confidence in the data. The number of observation wells selected, however, must balance the information needs with the cost of constructing them.

Without observation wells, only transient analysis (time-drawdown) methods may be used to determine aquifer properties, and only transmissivity and hydraulic conductivity can be determined. A single observation well makes it possible to determine storage coefficient, but data analysis is still restricted to transient methods. Two or more observation wells permit the use of distance-drawdown methods of analysis, greatly improving the accuracy of aquifer parameter estimates. Distance-drawdown analysis is especially important whenever transient analysis methods are apt to produce erroneous results, as often occurs in unconfined aquifers, tight sediments, leaky aquifers, and aquifers with boundaries near the pumped well. Therefore, when possible, use at least two observation wells during a pumping test. Determining parameters such as leakage/delayed yield and anisotropy usually require more than two observation wells.

6. Design the Observation Wells

a) The observation well diameter must be large enough to accommodate instrumentation used to measure water levels and small enough that the volume of water in the well does not cause a time lag in responding to aquifer drawdown changes.

b) Unlike the pumped well, observation wells need not be highly efficient, just open enough to reflect pressure changes that occur in the aquifer. Thus, inexpensive construction materials such as slotted screens may be used (unless they will be used later as monitoring, recovery, or injection wells). Yet, to accurately represent the potentiometric changes that may differ vertically in the aquifer, the well intake must be open to the aquifer from top to bottom. This objective can be achieved with moderate well development. Techniques such as surging and bailing, which provide modestly effective development, can be used.

c) Generally, observation well screens should be fully penetrating to eliminate complications in data interpretation caused by partial penetration. As with the pumping well, the exceptions to this rule are:

    1) Avoid fully penetrating screens where they would create a conduit capable of spreading contamination;

2) Use short screens to assess vertical anisotropy at discrete elevations in the aquifer.

Short screens are appropriate for observation wells installed in aquitards that are being used to assess connectivity, recharge, or delayed yield factors.

As stated above, fully penetrating wells will simplify the data analysis because hydraulic theory for fully penetrating systems is simpler than that for partially penetrating ones. Theory also predicts that, for a confined aquifer, an observation well will show fully penetrating response if either it or the pumped well is fully penetrating. That is, in theory, both need not be fully penetrating --it is sufficient that just one or the other be fully penetrating to observe the simplified fully-penetrating response. In practice, however, it is preferable that both the pumped well and the observation well be fully penetrating, if possible. In aquifers where hydraulic conductivity varies substantially with depth, it is possible that a fully penetrating response would not actually occur unless both the pumped well and observation well were fully penetrating.

7. Situate the Observation Wells

a) Lateral Distribution

When using two observation wells, they should be positioned along a straight line radiating from the well. Accurately assessing horizontal anisotropy or near-well boundaries requires three pairs or sets of observation wells positioned along three different lines emanating from the well. If the principal axis of anisotropy is known, two sets of observation wells will suffice, one along the principal direction of anisotropy and one perpendicular to it. For example, if a fractured rock aquifer is known to be more permeable north-south than east-west, one set of two or more observation wells would be installed on a line north (or south) of the pumped well and another set along a line east (or west) of the pumped well.

In theory, single wells placed on three different lines emanating from the pumped well are sufficient to assess horizontal anisotropy. In practice, however, other heterogeneities can influence drawdown readings enough to bias the calculated anisotropy if just a single well is used along each line. Therefore, it is preferable to use pairs of wells whenever possible.

b) Well Spacing

Observation wells along a particular line from the pumped well should be spaced logarithmically with the distance to each successive observation well approximately double that to the preceding well. For example, three observation wells may be placed at distances of 10 ft, 20 ft, and 40 ft from the pumped well, or 50 ft, 100 ft, and 200 ft from the pumped well.

There are advantages and disadvantages to locating observation wells either near to, or far from, the pumped well. Distance drawdown analysis methods tend to integrate aquifer properties over the area spanned by the observation wells, so distant wells tend to yield aquifer parameters representative of a broad area of aquifer. At great distances, however, wells may exhibit drawdowns so small that they are difficult to measure accurately or analyze confidently. On the other hand, observation wells installed near the pumped well show more substantial drawdown but tend to reveal aquifer properties on a smaller scale. Situating observation wells, therefore, depends on the type of information required. For contamination investigations of small plumes, closely spaced observation wells provide satisfactory data.

The data set will be more reliable if substantial drawdowns can be attained in the observation wells. This is accomplished by maximizing the flow rate and locating the observation wells sufficiently close to the pumped well. As a rule of thumb, the distance from the pumped well to the nearest observation well should not exceed the square root of the expected radius of influence of the pumped well. R can be determined from:

$$R = \sqrt{\frac{0.04Tt}{S}}$$

where

R = radius of influence in ft

T = estimated transmissivity in ft^2/day

t = pumping test duration in days

S = storage coefficient

For example, in tight sediments, if the expected radius of influence is less than 100 ft, at least one observation well should be located within 10ft of the pumped well.

Be aware that the oft-repeated recommendation to locate the nearest observation well one or two aquifer thicknesses from the pumped well is actually a generalization (not entirely correct) for locating partially-penetrating observation wells away from a partially-penetrating pumping well. The actual radial distance for a partially-penetrating observation well must take into account anisotropy, as follows:

$$r = \frac{1.5b}{\sqrt{\frac{K_z}{K_{xy}}}}$$

where

r = radius from pumping well

b = thickness of aquifer

K_z = vertical hydraulic conductivity

K_{xy} = horizontal hydraulic conductivity

In most instances, and especially in unconfined or tight sediments, use closely spaced observation wells and eliminate partial penetration effects by using fully penetrating wells, or compensate for partial penetration effects by determining the anisotropy of the aquifer.

c) Vertical Distribution of Observation Wells

Generally, make sure that observation well screens are located in the pumped aquifer and fully penetrate it. To determine vertical anisotropy, however, screens must only partially penetrate the aquifer. For this determination, install observation wells in pairs at the same location, with one well screened in the pumped interval and the other screened in an unpumped interval of the aquifer to get a three-dimensional view of the pressure reductions caused by pumping.

If pumping is expected to induce leakage across an aquitard and if the leakance must be determined, place one or two piezometers in the aquitard to assess the magnitude of the drawdown, if any, created by the pumped well. Aquitard-monitoring wells should have short screens approximately centered in the aquitard, to keep the screen as far as possible from the top and bottom of the aquitard. Ideally, an aquitard observation well should be drilled at the same location as an observation well completed in the pumped aquifer.

8. Establish the Pumping Test Duration

a) The duration of pumping tests can range from a few hours to a few weeks depending upon the nature of the formation and the type of information required. For example, in highly transmissive confined sediments, if only near-well transmissivity must be known, a 2-hour test might suffice. However, to acquire information about boundaries or leakage, or if sediments are tight or unconfined, a much longer test is required. The preliminary test of the pumped well (Section B.9) will help in planning the test length.

b) For confined aquifers, a test duration of 24 to 48 hours will generally provide the information required.

c) Longer tests are required for unconfined aquifers because the cone of depression expands more slowly and delayed-drainage effects retard the response of the aquifer to pumping. Plan to conduct pumping tests in unconfined aquifers for 3 days or longer.

d) If leakage effects among aquifers must be determined, a longer test is appropriate. For example, under confined conditions, it may be desirable to extend the test to two or three days.

e) Economics may dictate curtailing the length of the pumping test if treating or storing pumped water is expensive. If water disposal is inexpensive, however, it makes sense to extend the test because the cost of the additional pumping and monitoring required is generally nominal.

9. Select the Appropriate Flow Rate and Measurement Device

a) The objective of the pumping test is to stress the aquifer sufficiently to obtain a meaningful, measurable response. Generally, the magnitude of the drawdown response in most observation wells is small. Thus, in most aquifer tests, design the well and pump intake in such a way that a sufficient stress is placed on the aquifer system that can be measured at a distance.

b) Select the pumping rate on the basis of a preliminary test (such as a step-drawdown test, Section B.9) so that the rate can be sustained by the pump for the duration of the test. The rate should not be so large that the water level is drawn down into the screen area, causing cascading effects and entrained air; under no circumstances should the water level be drawn down to the water entry of the pump or tail pipe.

c) Small variations in the discharge rate create large errors in the calculation of aquifer parameters. Therefore, sustaining a constant discharge rate is more important than knowing the exact rate with great accuracy. Accordingly, maintain the flow rate as closely as practical to a constant value, usually within ±1 percent or less. This can be achieved only if the flow rate can be measured precisely and adjusted easily as needed.

d) Always operate the pump against a partially closed valve so that, as drawdown increases during the test, a compensating reduction in back pressure is achieved by gradually opening the valve. The correct valve and flow measurement method are critical to this requirement. Select a valve that can be opened or closed in tiny increments to ease flow-rate control. A ball valve that opens fully or closes fully with a single 90-degree turn of a handle is undesirable because careful adjustments are difficult to achieve. A better choice is a gate valve that requires several 360-degree turns to open or close.

e) Flow measurement devices are typically based on three principles; head-type (orifice, venturi), velocity-type (magnetic, ultrasonic), and displacement-type (rotor, paddlewheel). Measurement devices/methods for a pumping test, in order of preference, are as follows:

   1) Orifice weir with manometer (see Driscoll, pg 537): This is the best method of measuring the flow rate because it is precise, allows instantaneous reading of the flow rate so that adjustments can be made readily, and is relatively “low-tech”. While most orifice weirs accommodate higher flow rates, small-scale versions can be made for flows as small as a few gallons per minute. Such custom-made meters can be calibrated easily in the field with a bucket and stop watch. Installing a totalizing meter in line with and upstream from the orifice weir provides assurance that the total discharge for the test is calculated accurately. After completing the test, total discharge volume is divided by test duration to determine average flow rate.

   2) Instantaneous (ultrasonic) flow meter: non-invasive, can be equipped with a data logger. Some meters may not respond properly when pumping sediment-laden water or two-phase fluids like hydrocarbons and water.

   3) Paddlewheel totalizing meter: shows total volume pumped. When using this type of meter, flow rate must be determined by taking consecutive readings and dividing by the time between them. Accuracy may vary from one meter to another. Also may not respond properly when pumping sediment-laden water or two-phase fluids. Meter inaccuracy at low flow rates can be allayed by installing a flow restrictor (such as manufactured by Clack Corporation) upstream of the meter. The restrictor creates enough back pressure on the pumping unit to minimize flow rate fluctuations.

   4) Bucket, or other container of known volume, and stop watch. For low flows, this procedure is about as accurate as any for determining the flow rate. It also serves as a reliable calibration tool for other flow measurement devices.

Other methods of measuring flow rate involve using various types of weirs, flumes, and open-discharge pipes generally do not provide the precision required for controlling the flow rate during a constant-rate pumping test.

10. Select the Pump

a) The pump used must have sufficient capacity to maintain the required discharge throughout the constant-rate portion of the test and to produce the various flow rates required for the step-drawdown test.

b) The pump should be capable of delivering the planned discharge rate at pressures substantially higher than the apparent nominal pressure required to lift water to the surface and overcome friction losses in the piping system. Pumping against a high head such as 60 to 100 psi tends to reduce discharge rate variations. It also permits operating the pump against a partially closed valve, creating additional head to help minimize flow-rate fluctuations during the test.

c) Submersible or turbine pumps driven by electric motors are ideal for conducting pumping tests because (barring spikes or storms) they run at nearly constant rates, producing generally uniform flow. Turbine pumps driven by gasoline or diesel engines, however, cause greater flow-rate variations because engine output can vary with fuel mixture, and air temperature and pressure.

d) The pump should be equipped with a check valve so that water in the column pipe and discharge pipes doesn't siphon back into the well following pump shut off. This prevents a sudden charge of water from obscuring the early recovery data and making analysis more difficult.

11. Plan for Pumped Water Disposal

a) Discharge pumped water so that pumped aquifer zones are not recharged. To accomplish this, pipe water to nearby storm or sanitary sewers, or lined surface-water bodies. If these options are not available, arrange to spread the discharge water on the ground sufficiently far from the pumping test site so that infiltration will not affect the test results.

b) If the water quality is such that direct discharge is not permitted, treatment may be necessary. Occasionally, water treatment facilities are already available on site. Alternatively, it may be possible to arrange for temporary treatment equipment just for the pumping test. If disposal during the test is not possible, the fluid can be discharged to containers such as frac tanks temporarily. Provisions must be made for the appropriate number and size of containers to handle the volume of water pumped during well development, step-drawdown testing, and constant-rate testing, plus a safety margin.

c) Discharge water must be disposed according to all applicable laws and regu1ations. Contact the governing agencies to determine which restrictions apply.

d) ARCADIS should not be responsible for signing manifests and should not "take possession" of discharged water.

12. Check for Casing Storage

Casing storage effects will render useless the early time/drawdown data from pumping tests. The larger the well diameter and the lower the specific capacity, the longer casing storage effects persist. Data recorded before casing storage effects end (at t_c) cannot be analyzed by any method.

The duration of the casing-storage affected portion of the test can be estimated as follows:

$$t_c = \frac{0.6\left(D^2 - d^2\right)}{Q/s}$$

where

t_c = duration of casing storage effect ('critical time'), in minutes

D = inside diameter of well casing, in inches

d = outside diameter of pump column pipe, in inches

Q = flow rate, in gpm

s = expected drawdown in the pumped well, in ft

Before conducting the test, it is important to estimate t_c. If the value is large, take steps to minimize storage effects if possible. For example, a packer may be installed with the pump column pipe to keep the water standing in the well casing from being removed from the well. If this is done, the packer must be specially designed to permit measurement of the hydraulic pressure in the well just under the packer. Alternatively, it may be possible to install ballast material alongside the column pipe to take up space and reduce the volume of water stored in the casing. For example, a 3.5-inch OD PVC pipe run alongside the column pipe in a low-yielding, 4-inch well, can reduce the duration of casing storage effects by 75 percent.

To demonstrate the significance of casing storage, a 4-inch test well in tight sediments with 1.25-inch column pipe producing 2 gpm with 30 ft of drawdown results in the following calculation:

$$t_c = \frac{0.6\left(4.026^2 - 1.66^2\right)}{2/30}$$

= 121 minutes

Thus, the first two hours of test data from this well cannot be analyzed.

In filter-packed wells, if water in the filter pack can drain quickly into the well (such as in wells that are screened across the water table), the equation for t_c must be modified to account for filter pack storage. The accomplish this, the term

$$D^2 - d^2$$

is replaced by

$$\left(D^2 - d^2\right) + S_y\left({B_d}^2 - {C_d}^2\right)$$

where

B_d = diameter of borehole, in inches

C_d = outside diameter of casing, in inches

S_y = short-term specific yield of filter pack material --approximately 0.1 or 0.15

II. Pretest Activities

a) Unless installed specifically for the test, sound all wells for use in the test to verify well depth. (Do not use water level meters for this purpose, because some meters have probes that leak and trap water when subjected to excessive pressure.) Also, if adequate connection to the aquifer is suspect, conduct a slug test (either ‘in’ or ‘out’ - attempt to change the water level by at least 2 feet) in the observation wells. If the water-level response is too sluggish or no response is apparent, redevelop the well.

b) Label all wells (temporarily, if necessary) for quick and easy identification throughout the test.

c) Unless previously verified, measure the distance of all observation wells from the pumping well to the nearest foot.

2. Select Appropriate Water Level Measuring Devices

a) Pressure Transducers and Data Logger Combination

Transducers connected to electronic data loggers provide rapid water-level measurements with accuracy and ease. Some electronic data loggers (i.e., Hermit) collect and store data from a number of input channels (downhole pressure transducers plus atmospheric pressure) to provide water-level measurements in multiple within several hundred feet radius of the data logger, while others consist of a single logging transducer (i.e., Troll™, Levelogger™). Typical loggers take readings at preprogrammed linear or logarithmic intervals. If desired, data can be transferred to a personal computer for processing.

Small-diameter transducers (typically 0.5 to 0.75 in) are available that cover a range of pressures. Because they yield readings accurate to a percentage of their pressure range (usually about ±0.1 percent of the range in the center of that range, and ±0.2 percent near the limits) transducers that span a wide pressure range have lower absolute accuracies than those that span a narrow range. For example, a typical transducer with a 5 psi range detects water-level changes over a 11.6 ft with an accuracy of ±0.01 ft, whereas, a transducer with a 15 psi range detects changes over a 34.7 ft with an accuracy of ±0.03 ft. Thus, to ensure the greatest accuracy, select the transducer with the pressure range that most closely encompasses the anticipated drawdown or water-level change. Furthermore, confirm transducer water-level measurements throughout a test by manually taking regular water-level readings with a water level meter.

Caution: To prevent transducer malfunction, do not submerge transducers in excess of their operating range.

b) Water Level Meters, Interface Probes

These devices provide quick and easy water-level measurements with reasonable accuracy. They employ a sensor that is lowered into a well on the end of a marked cable (typically imprinted in feet and hundredths of a foot). When the sensor contacts water, a circuit is completed, activating a light, audio signal, ammeter, or digital display in the cable reel or housing. However, because the measurements are manual, the speed of readings cannot match those of a pressure transducer with a data logger. Thus, a water level meter is most useful in taking correlative, manual measurements in wells as a backup and for data checking, as well as measuring wells outside the active observation well network.

When appropriate, one water level meter should be used to take readings in all wells. If more than one meter is used to make site-wide water-level measurements, record the serial numbers and make comparison measurements within a single well to calibrate to a common standard.

c) Wetted Steel Tape

When using a steel tape, attach a weight to the bottom, wipe dry and coat the lower 2 to 3 feet with carpenter's chalk or water-soluble ink from a felt-tip marker, lower the tape into the well until part of the coated section extends below the water level, hold one of the major division (e.g., foot) markings at the predetermined measuring point, and record this reading. After withdrawal, read the wetted line on the coated section to the nearest 0.01 ft. Subtract this reading from the mark held at the measuring point; the difference is the actual depth to water.

A wetted steel tape is accurate and reliable, and is useful to verify and calibrate readings from other instruments. The procedure, however, is more time-consuming than others, limiting its usefulness during the early portion of pumping test when many rapid measurements are required. Furthermore, the approximate depth to water must be known in advance to ensure that part of the chalked section is submerged to produce the wetted line.

3. Verify Measuring Device Accuracy

Test pressure transducers and data logger readings using a bucket or barrel filled with water. Submerge each transducer, accurately measure the water head above the transducer, and compare the measurement to the data-logger reading. Check transducer response to changing heads by raising the transducer a certain distance, observing the change in the datalogger reading, and then measuring the distance with a standard steel tape. Water level meters should be in good working condition and calibrated, ensuring there are no breaks or splices in the cable.

4. Establish a Reference Point for Measuring Water Levels

At each well, establish and clearly mark the position of the selected reference point (often the north side, top of the casing). Determine the elevation of this point, record it, and state how this elevation was determined. This elevation point is important to establish the position of the piezometric surface, so it must be determined accurately.

5. Record Background Water Levels

To establish local trends, measure groundwater levels in all test wells and on-site surface water levels at regular intervals for several days before pumping any of the test wells. Although two days preceding the test may be enough (this meets the standards of some regulators), ideally the period of time should be at least equal to the length of the pumping test (three days to a week is optimum). Unless extreme variations are expected, such as significantly increased stream discharge in response to off-site precipitation, only surface water bodies within the radius of influence of the pumping well need to be monitored. A well outside the radius of influence may provide valuable information about water-level trends if monitored before, during, and after the pumping test. In areas that could be influenced by tidal fluctuations, collect information regarding local tidal variations before, during, and after the test.

If levels in the zones to be monitored during the test might be affected by pumping of other nearby wells, gather information about the discharge rates and operating times of those wells. Also, monitor water levels for a sufficient period before the test to evaluate the influence of nearby wells. Water-level monitoring should be done far enough in advance to allow time to negotiate with well owners and take appropriate action. If possible, arrange to have nearby wells shut down or pumped at a constant rate to ease data interpretation.

6. Record Barometric Pressure

Atmospheric-pressure changes can cause water level changes in confined or semi-confined aquifers, leading to erroneous conclusions about aquifer parameters. To correct for these changes, the barometric efficiency of each appropriate aquifer must be determined. Aquifer barometric efficiency (BE), a ratio of aquifer head change to atmospheric pressure change, can be calculated using:

$$BE = \left(\frac{\Delta h}{\Delta B_p}\right) 100\,\%$$

where

BE = barometric efficiency, in percent

Δh = change in water level resulting from change in atmospheric pressure, in feet

ΔB_p, = change in atmospheric pressure, in feet of water

To measure atmospheric pressure changes, either ensure that the dataloggers being used also measure barometric pressure, or obtain data from a nearby source. Barometric pressure must be recorded throughout the background water-level-measurement period and throughout the test. Ideally, barometric pressure and water-level measurements should be made during a time of significant atmospheric pressure change so their relationship can be more easily correlated.

Logging transducers with vented cables (e.g.: Troll, miniTroll) already account for barometric pressure and no additional adjustment is required.

7. Install a Rain Gauge

Heavy precipitation can cause a significant water-table rise in shallow aquifers. Note that rainfall data from nearby weather stations or airports may not be representative, because precipitation patterns may vary greatly over short distances. Therefore, when testing shallow aquifers, a rain gauge should be installed at the test site and monitored during rainfall. Keep in mind that storm sewers can channel large volumes of water rapidly to shallow aquifers.

8. Set-up: Remaining Equipment Required for Test

a) Keep sensitive electronic equipment away from devices that generate significant magnetic fields. For example, do not place data loggers near electric power generators or electric pump motors. Likewise, radio signals may cause dataloggers or computers to malfunction.

b) Secure data logger and transducer cables at the well head to prevent movement that would affect measurements. Mark a reference point on transducer cables and check regularly to detect slippage.

c) Provide adequate lighting for night readings.

d) Identify all equipment to be used in the test that will affect data. For example, describe (by serial number or otherwise) the pump, any isolation packers, water level meters, data loggers, rain gauges, barometers, flow meters, buckets or volumetric containers, watches, and steel tapes used.

e) Consider having backups for key equipment such as data loggers, generators, water level meters, etc.

9. Perform a Job Safety Analysis

To ensure that everyone is aware of the hazards associated with the work, and that each person knows his/her responsibilities during the preliminary and full-scale test, run through a JSA of the test before the start of pumping.

10. Conduct a Preliminary Pumping (Step-Drawdown) Test

Conduct a short-term preliminary test of the pumping well to estimate the hydraulic properties of the aquifer, estimate the duration of the test, and establish a pumping rate. A step-drawdown test is the most efficient preliminary test to use. If other constraints determine flow rate and the flow rate is sustainable, a step test is unnecessary.

The concept of step-drawdown testing in wells was first developed by Jacob (1947). He proposed that drawdown in a well has two components: formation loss (laminar, proportional to the discharge), and well loss (turbulent, proportional approximately to the square of the discharge). Jacob outlined a multiple-step drawdown test where discharge was increased at specific times, as if pumping of the well was held constant and additional wells were introduced at corresponding increases in pumping rates. Rorbaugh (1953) later noted that Jacob's assumption of second-order turbulent flow did not take into account that turbulence at low rates of discharge is not fully developed. Thus the exponent for turbulent flow should be expressed as an unknown constant. Taking this into consideration, the arithmetically-plotted results of a step-drawdown test can be used to select the discharge rate for a pumping test, determine drawdown for a given pumping rate and optimum pump depth, and even (with some minor calculations) estimate the transmissivity of the formation prior to the test. (This is also a good test for reliability of the flow meter.)

a) Select the pumping rates for the step-drawdown test based on:
   1) production capability estimates made during well development,
   2) prior pumping information,
   3) slug test data (for small wells), or
   4) a brief, preliminary rate test.

Step tests are most commonly run with three steps at 33, 67 and 100 percent or four steps at 25, 50, 75, and 100 percent of the anticipated maximum rate. Sometimes a step is added at 133 percent for a three-step test or 125 percent for a four-step test and the first step is dropped.

b) Conduct the step test, pumping at each level for 30 to 60 minutes. It is important to run the initial step long enough to establish that the effects of well storage have dissipated, with the remaining steps run for the same duration as the initial step. Although standard practice is to allow a recovery period after each step, practical experience shows that these individual recoveries are not necessary.

c) At the end of the step test, mark the setting of the discharge control valve corresponding to the flow rate for the full-scale pumping test. Secure the valve in that position with wire or tape to prevent inadvertent changes.

d) Allow sufficient time after completion for drawdown to return to static level. Although the time may vary, allow at least one day of recovery after the step-drawdown test has been completed before starting the constant-rate test.

11. Synchronize Watches

Just before the constant-rate test, watches and other time-measurement devices (i.e., dataloggers) should be synchronized so that the time of each reading, electronic and manual, can be referenced to the exact minute and hour that pumping started.

III. CONDUCTING THE TEST

1. Record Information

a) Use appropriate data forms

b) Record all required background information on logs before beginning the test

c) Record time as military (24-hour) time.

d) Ensure that everyone taking manual water-level measurements understands the units of measurement on the device or devices they will use.

2. Keep Pertinent Well Construction Details at Hand

To evaluate data plotted during the test, it may be necessary to have access to well construction information, such as the following:

- Lithologic logs;
- Well depths;
- Screen lengths
- Screen type (slotted, wrapped, opening size)
- Filter pack thickness and length
- Pumped well diameter
- Pump characteristics (performance, unit dimensions)
- Pump setting depth
- Topographic maps

3. Start the Test

a) Check all wells to confirm that water is at static level. Record the time since last pumping.

b) Make sure all field personnel are aware of predetermined starting time.

c) Start the pump and timing devices simultaneously. Use both an audible and visible signal to indicate the start of the test, especially if the distance between the pumped well and observation wells is large.

4. Measure Drawdown at Established Times

The widespread use of data loggers with extended memory precludes the older standard of using logarithmic time measurements. However, remember that rapid-frequency readings are needed early in the test in order to observe early effects of pumping and formation storage, plus effects of well construction. Water level measurements should be taken at least every five seconds.

Early time data are of greater importance when conducting pumping tests to identify aquifer heterogeneities and should be collected at short time intervals (< 1 sec) and considered as part of the pumping test analysis. Large data files can be generated and may need to be manipulated with text editors prior to importing data to other software such as Excel.

For manual observation well readings, the following schedule is suggested:

Elapsed Time	Interval Between (minutes) Measurements (minutes)
0-5	1
5-15	2
15-60	5
60-120	10
120-300	30
300-1440	60
1440-end of test	240

Drawdown readings are sometimes difficult to record at the exact time required by the above schedules. If the designated time for a drawdown reading is missed, take a reading anyway and record the actual time. However, try to follow the established schedule as closely as possible to ease data plotting. Use the following table as a guideline for time measurement accuracy.

5. Check the Flow Measuring Device

Unrecorded fluctuations of pumped well discharge rate can make the test data difficult to interpret. Measure and record discharge every 5 minutes during the beginning of the test. When discharge becomes stable, reduce the frequency to hourly checks.

As water levels decline, the discharge rate may decrease, thus requiring adjustment. Whenever adjusting the flow rate, record water levels in the pumped well before and after each adjustment.

6. Monitor Fuel Levels

When using liquid-fuel-driven engines or generators, monitor and refill fuel tanks as needed to prevent premature termination of the test.

7. Plot Data to Evaluate Trends and Catch Aberrations

a) Begin to tabulate and graph the elapsed time, discharge rate, and pumped well drawdown as early as possible in the test, usually after the first hour of testing.

b) Prepare a plot of the log of drawdown (log10s) versus the log of the ratio of time since pumping started to the square of the distance from the pumped well to the observation well (log10t/r2) on arithmetic graph paper and maintain during the test. Compare this data to basic type curves to detect deviations that may be due to discharge variations or other changes in field conditions that need to be documented. A portable computer and printer ease this plotting for tests with many wells.

c) Keep the plots current throughout the test. This information supports informed, intelligent decisions about test progress and may signal anomalies such as equipment malfunctions or unacceptable flow rate variations. Analysis of these plots may suggest that more data is needed to substantiate conclusions about the groundwater system.

8. Collect Groundwater Samples and Measure Field Parameters

Samples of discharge water may provide valuable information about the nature of aquifer water quality as it changes during the pumping test. Depending on the site conditions, samples collected regularly throughout the test may signal proximity to a contaminant source, connection with surface water bodies, or other contributors to water quality change. The number of water samples needed and the frequency and time of their collection depends on both nearness to suspected or known water quality influences and the test budget.

9. Verify Measuring Device Accuracy

Recheck the accuracy of hand-held electronic water-level sounders before starting the recovery portion of the test. During pumping and recovery, check transducer accuracy periodically with reliable manual devices. Every hour or few hours is sufficient for most tests.

10. Measure Water Levels during the Recovery Phase at Established Times

Recovery of water levels following the pumping phase should be measured immediately upon pump shut down and recorded for a period of time equal to the pumping time, or until the water levels have reached 95 percent of the initial, pre-pumping static water level. Use the same drawdown measurement schedule that was used during pumping. A check valve should be used to prevent backflow of water in the riser pipe into the well, which could result in unreliable recovery data.

Recovery phase data may be easier to analyze because no discharge fluctuations occur, and pump-induced turbulence is not a concern in the pumping well. However, note that typically the calculated transmissivity from the pumping phase will be lower than that of the recovery phase due to the added turbulence and vertical flow components during pumping.

11. Record Observation of Pertinent Phenomena

Record any unusual events occurring just before or during the test that may affect test data, such as:

- Weather changes
- Heavy equipment (trains, etc.) passing through area
- Operation times of other wells
- Changes in pumping rate
- Equipment problems, and
- Earthquakes

IV. POST-TEST PROCEDURES

1. Document the “As-Built” Configuration of the Test

Describe the configuration of the test, the observation well locations versus the pumping well, water discharge, outside influences detected during the test, and any modifications to the original plan.

2. Verify Timing Device Agreement and Measuring Device Accuracy

Compare all clocks, watches, and data recorders for agreement and note any discrepancies, identifying the devices and where they were used. Compare manual measurements to datalogger measurements within wells to confirm accuracy of measuring devices.

3. Sound the Pumped Well

Determine if any aquifer material accumulated in the pumped well during the test. Sand or other material accumulating in the well during the test progressively blocks screen areas, reducing the effective aquifer penetration. If the effect of this condition is not taken into account, aquifer parameters calculated from test data will be wrong. Gradually decreasing aquifer penetration in a pumped well significantly complicates test data analysis. The wisest strategy, therefore, is to prevent infilling of screens by sufficient development of the pumped well.

4. Decontaminate All Equipment Contacting Site Groundwater and Soil

Use appropriate decontamination procedures.

5. Monitor Background Information as Long as Possible

If possible, continue to monitor groundwater levels, surface water levels, and barometric pressure data for several days after test completion. This information may reveal trends or relationships undetected before or during the test.

V. SPECIAL CONSIDERATIONS

1. Wells Containing Floating Nonaqueous Phase Liquids

It is best to use pressure transducers to measure water levels in wells containing floating product such as gasoline. Contact with floating product, however, may make transducers and cable unsuitable for future use. Thus, include the cost of replacing transducers (and perhaps cable) when calculating pumping test budgets. Otherwise, protect each transducer and cable assembly by encasing it in plastic tubing or pipe. Be sure that each protected transducer still can respond accurately to any pressure changes.

As an alternative to pressure transducers, make manual measurements (using a interface probe) of both the fuel level and water level individually. Then correct the observed thickness of floating product by its density to arrive at an effective pumping level. Measure product density in the field using a simple density balance (such as drilling fluid balance) or consult an appropriate API table. This manual procedure will work, but takes time and introduces additional measurement and computation errors.

2. Fill Materials

Occasionally, pumping tests are conducted in or adjacent to fill materials. In these circumstances, it is essential that the nature of the fill and possible extremes in heterogeneity be understood and incorporated into the design of the pumping test so that the resulting data set can provide the required information.

3. Karst and Cavernous Aquifers

Flow through the fractures and conduits within a karst aquifer system ranges from conduit to diffuse. Conduit flow describes flow through dissolution channels with velocities commonly high and turbulent. (The presence of conduits typically requires a dual-porosity model for characterization). Diffuse flow, on the other hand, refers to a slow, mostly laminar to slightly turbulent flow through a series of small, discrete pathways that are being enlarged through dissolution. Karst aquifers do not lend themselves to conventional pumping test layout, procedures, and analysis because flow can be dominated by discrete channels. The discrete nature of high-conductivity zones can range several orders of magnitude and thus hydraulic conductivity values vary according to the scale of measurement, from local to regional. Interpretation of pumping tests must take into consideration the portion of the aquifer being tested.

Additional background investigations may need to be conducted before a pumping test is conducted, in order to predict the connectivity of the wells within the test network. This may include borehole and surface geophysics, tracer (natural and introduced) testing, spring flow and water chemistry analysis, slug testing, and lineament analysis.

4. Fractured Aquifers

The challenge to conducting a pumping test within a fractured-rock aquifer is the continuity of fractures can vary significantly within an area and affect its ability to provide water in a consistent manner. Many fractured aquifers also exhibit a preferred permeability direction based on predominant fracture orientations. Recharge may also vary seasonally and cause production problems in low flow periods (low water level and low recharge). During these periods excessive drawdown may occur. Typically, sources completed in bedrock composed of shale, basalt, granite or any consolidated material can have fractured flow concerns.

For these aquifer systems, although a conventional pumping test approach is generally appropriate, more observation wells will be required to determine the anisotropy and to discern both near-well and distant responses. Also, step-drawdown test data provide valuable information in fractured aquifers because flow near the well in fractured aquifers may be mostly turbulent.

VI. REFERENCES

ASTM D4050, *Standard Test Method (Field Procedure) for Withdrawal and Injection Well Tests for Determining Hydraulic Properties of Aquifer Systems*. ASTM 04-08, Soil and Rock.

ASTM D5717, *Standard Guide for Design of Ground-Water Monitoring Systems in Karst and Fractured-Rock Aquifers*. ASTM 04-09, Soil and Rock.

Dawson, Karen J. and Istok, Jonathan D., 1991. *Aquifer Testing --Design and Analysis of Pumping and Slug Tests*. Lewis Publishers, Chelsea, Michigan, 344 p.

Driscoll, Fletcher G., 1986. *Groundwater and Wells, Second Edition*. Johnson Filtration Systems Inc., St. Paul, Minnesota, 1,089 p.

Jacob, C.E., 1947. *Drawdown Test to Determine Effective Radius of Artesian Well*, American Society of Civil Engineers Transaction, Vol 122, No. 2321, pg 1047-1070.

Kruseman, G. P.and de Ridder, N. A., 1990. *Analysis and Evaluation of Pumping Test Data, Second Edition*. International Institute for Land Reclamation and Improvement, Wageningen, The Netherlands, 377 p.

Rorabaugh, M.I., 1953. *Graphical and Theoretical Analysis of Step-Drawdown Test of Artesian Well*. Proceedings of American Society of Civil Engineers, Vol 79, No. 362, 23 p.

Stallman, Robert W., 1971. *Aquifer Test Design, Observation and Data Analysis*. Techniques of Water-Resources Investigations of the United States Geological Survey, Chapter B1, Book 3, 26 p.

Attachment 3L
Specific Capacity Testing and Data Reduction

Specific Capacity Testing and Data Reduction

Rev. #: 2

Rev Date: February 3, 2006

Approval Signatures

Prepared by: Michael J Gefell Date: 2/27/09
Michael Gefell

Reviewed by: J Shi Date: 2/27/09
Jerry Shi (Technical Expert)

I. Scope and Application

Specific-capacity testing is a field method used to estimate the transmissivity of a saturated geologic medium surrounding the screened or open interval of a well. A specific-capacity test involves pumping groundwater from a well at a constant rate and quantifying the pumping rate and magnitude of drawdown inside the tested well after a known duration of pumping. Specific-capacity tests are also referred to as single-well pumping tests or constant-rate tests.

The transmissivity is calculated based on the pumping rate and drawdown measured inside the pumped well. Time-drawdown analysis can be performed with a semilog data plot to estimated transmissivity (Driscoll, 1986). Alternatively, an iterative calculation can be performed based on the pumping duration, the effective radius of the well, and storativity of the formation.

If the thickness of the effective water-bearing zone transmitting groundwater to the well intake is assumed to be approximately equal to the length of the intake, the hydraulic conductivity (K) can be estimated by dividing the transmissivity by the length of the intake.

II. Personnel Qualifications

Specific-capacity tests will be performed by persons who have been trained in the proper usage of pumping and water-level measurement equipment under the guidance of an experienced field geologist, engineer, or technician.

III. Equipment List

The equipment needed for specific-capacity testing includes:

- health and safety equipment, as required in the site Health and Safety Plan (HASP);
- cleaning equipment;
- pump (preferably submersible) capable of pumping at a controlled rate between a fraction of one gallon per minute (gpm) and several gpm, equipped with discharge line;
- power source for the pump;
- calibrated in-line totalizing flow meter or two calibrated buckets;

- stopwatch;
- electronic water-level indicator; and
- field notebook.

IV. Cautions

Wells and piezometers have different water-yielding characteristics as a function of their screen lengths, depth below the water table, and geologic materials in which they are installed. During the first minute of pumping, the water level should be continuously monitored and the pumping rate adjusted to avoid pumping the well dry. Additional cautionary statements pertinent to data reduction are included in Section I. Allowing discharge water to infiltrate next to the well can impact the test results and should be avoided.

V. Health and Safety Considerations

Field activities associated with specific-capacity testing will be performed in accordance with a site-specific HASP, a copy of which will be present on-site during such activities.

VI. Procedures

Pre-Test Set-Up

Prior to installing the pump into the well to be tested, the static water level inside the well is measured to the nearest 0.01 foot relative to a specified datum at the top of the well using the electronic water-level indicator. The water level and the time of measurement are recorded in the field notebook. The water level is measured again several minutes after the initial measurement. This measurement and time are also recorded. This procedure is repeated until two consecutive measurements are identical, indicating approximately static conditions. The static depth-to-water is recorded.

The pump is installed inserted into the well to at least 10 feet below the static water level, or within approximately 1 foot of the bottom of the well if the initial water column in the well is less than 11 feet. The depth of the pump intake below the static water level (indicating the length of the pre-test water column above the pump) is recorded. After the pump is installed inserted (but prior to pumping), the water level in the well is monitored until it has returned to within 0.01 foot of the static water level.

Test Procedures

The specific-capacity test is performed as follows:

1. Hold the water-level probe in the well just above the static water level. If an in-line totalizing flow meter is used, record the pre-test volume measurement in the field notebook. If no in-line flow meter is available, place the end of the discharge line into one of the two calibrated buckets. Record the total volumetric capacity of each bucket.

2. Simultaneously start the pump and stopwatch. Record the start time.

3. Immediately begin monitoring the water level in the well. If the water level inside the test well declines rapidly, quickly reduce the pumping rate to a slower, constant rate. To avoid pumping the well “dry” during the test, the drawdown after one minute of pumping should be less than or equal to 20% of the height of the pre-pumping water column above the pump. All pumping rate adjustments should be completed within 1 or 2 minutes of the start of pumping, after which no adjustment should be made other than minor adjustments that may be necessary to maintain a steady pumping rate.

4. Continue to pump for at least 20 minutes, recording the water level in the well at least once every 3 minutes during pumping. If an in-line flow meter is used, record the volume measurement on the totalizer gauge approximately every 2 minutes during the test. If calibrated buckets are used to measure the pumping rate, record the time at which the bucket reaches the known volumetric capacity of the bucket. Transfer the discharge line to the other (empty) calibrated bucket and record the time when it becomes full. Repeat this procedure for the duration of the test.

5. The specific-capacity test is complete after at least 20 minutes of pumping have elapsed. A longer pumping period is not necessary to estimate transmissivity from the test. However, increasing the length of the test may further increase the reliability of the resulting transmissivity estimate. Immediately before termination of pumping, record the final water-level measurement plus the time of the measurement.

6. Recovery data may be collected following pumping. Such data are highly recommended if the test well is in a location that may be tidally influenced. Also, recovery data provide backup data that may be used to estimate transmissivity. To collect recovery data, measure and record water level data according to the same schedule as used during pumping.

7. Calculate and record the total volume of groundwater removed from the well during the test and the total duration of the test. Divide the total volume (in gallons) by the total pumping duration (in minutes) to calculate and record the average test pumping rate (in gpm).

VII. Waste Management

Water generated during specific capacity testing will be placed in containers, if required per State or local regulations. Containerized waste will be managed and disposed of properly.

VIII. Data Recording and Management

Data from a specific-capacity test are reduced to a transmissivity estimate for the water-bearing formation surrounding the intake of the tested well. The transmissivity may be estimated using a single-well time-drawdown method with multiple drawdown measurements, or else using a specific-capacity procedure with one drawdown measurement. These options are described below.

Time-Drawdown Method

The time-drawdown method of analyzing transmissivity requires graphical data evaluation, but has several advantages. The method does not require an estimate of the formation storativity and the results are not influenced by well efficiency.

Plot the measured drawdown data (measurements in feet on Y-axis) versus the pumping time (minutes, logarithmic scale on X-axis). The semilog data plot typically shows an abrupt initial drawdown at early time, followed by a straight-line trend of data points. Draw a line through the straight-line trend of data points and extend the line through at least one complete log cycle (e.g., 10 to 100 minutes). The data points need not extend through the entire interval of the drawn line. The drawn line is extended to cover at least one complete log cycle for ease in data analysis. Determine the drawdown change (• s) over one log cycle of time for the line drawdown through the straight-line trend in the data points. The value of transmissivity can be solved using the following equation (Driscoll, 1986):

$$T = 264\ Q\ /\ \triangle\ s,$$

where:

T = transmissivity of the water-bearing zone surrounding the intake of the tested well (gallons per day per foot);

Q = pumping rate during the period of the straight-line trend in data points (gpm); and

$\triangle s$ = drawdown change over one log cycle (ft).

Single Drawdown Measurement Method

This method is relatively easy to use, but it requires an estimate of the formation storativity and the results can be influenced by well efficiency. The transmissivity can be estimated using a single drawdown measurement via the following equation (Walton, 1962):

$$\frac{Q}{s} = \frac{T}{\left[264 \log\left(\frac{Tt}{2{,}693 r_w^{\,2} S}\right) - 65.5\right]}$$

Q/s = specific capacity of the well in gpm per foot

Q = average test pumping rate (gpm)

s = drawdown measured inside of tested well after a known duration of pumping (ft)

T = transmissivity of the water-bearing zone surrounding the intake of the tested well (gallons per day per foot)

S = estimated storativity of the aquifer

r_w = effective radius of the well (ft)

t = time between the start of pumping and the time when the drawdown was measured (minutes)

The value of T can be solved iteratively using a specific-capacity test data reduction computer program. If the well screen is surrounded by a sand pack that may be assumed to be substantially more permeable than the formation, the effective radius of the well is taken to be that of the borehole.

The value of S may be estimated without introducing serious error into the results. For confined aquifers, S should be estimated as 0.0001. For unconfined aquifers, the short-term storativity may be comparable to that of a confined aquifer. Only after a protracted pumping duration (several hours or more) does the storativity begin to approximate the aquifer-specific yield of approximately 0.2 to 0.3 (Nwankwor et al., 1984). In the calculation of transmissivity from a specific-capacity test of less than several hours duration, an estimated storativity value of 0.01 can be used.

To obtain an estimate of the K of the water-bearing zone that transmits groundwater to the well, the calculated transmissivity value may be divided by the estimated thickness of the water-bearing zone. In a stratified formation in which the horizontal K may be expected to greatly exceed the vertical K, the thickness of the water-bearing zone may be estimated as the length of the well intake to obtain an estimate of the K immediately surrounding the well intake.

Cautionary Considerations

It should be noted that the above-listed methods are based on the modified non-equilibrium equation. According to Kruseman and de Ridder (1990), these methods are useful provided that:

$$u = \frac{r^2 s}{4Tt}$$

r = effective well radius
S = storativity
T = transmissivity of the test zone (formation interval adjacent to saturated sand pack)
t = the pumping duration

Following data analysis, the value of u should be calculated to confirm that the above condition is satisfied. If u > 0.15, then a different K test method should be employed. These cases are rare when using drawdown data from the pumped well, because the radius is a small number. The S value used in this calculation can be selected on previous site-specific pumping test results using observation well data, or else estimated as described in the previous subsection.

In circumstances when the pumping rate is low (e.g., less than 1 gpm) and the drawdown is high or occurs within the sand pack, the water removed from the well and sand pack storage should be calculated and subtracted from the pumped volume to estimate the volume of water produced by the formation. The volume of water produced by the formation should be divided by the pumping duration to obtain an effective pumping rate for use in calculating T and K.

In situations where the water level in the test well may be influenced by tidal fluctuations, drawdown and recovery data should both be measured and recorded on the same schedule. In these cases, to correct for potential tidal influence, calculate the average magnitude of the drawdown and recovery measured for the same duration during either pumping or drawdown. For example, if the pumping period lasted 30 minutes, calculate the average of the drawdown at 30 minutes and the magnitude of recovery that occurred during the first 30 minutes after shutting off the pump. This average value accounts for the tidal influence assuming that the rate of tidal change was approximately equal during the drawdown and recovery periods, and it should be considered the "effective drawdown" for use in the specific capacity method of Walton (1962). This correction should be useful in many situations, but may not adequately address tidal impacts if the drawdown due to pumping is small compared to the magnitude of the tidal influence. In these cases, it may be necessary to induce more drawdown during the test and/or time the test to coincide with slack tide conditions.

IX. Quality Assurance

QA Quality assurance calculations must be reviewed by a qualified hydrogeologist. Calculations will be provided with backup documentation, such as raw data and graphs of the data.

X. References

Driscoll, F.G., Groundwater and Wells. Johnson Filtration Systems, Inc., St. Paul, Minnesota, 1089 p. 1986.

Kruseman, G.P., and N.A. de Ridder. 1990. *Analysis and Evaluation of Pumping Test Data. International Institute for Land Reclamation and Improvement, Wageningen, The Netherland*s. Second Edition, Publication 47, 377 p. 1990.

Nwankwor, G.I., Cherry, J.A., and R.W. Gillham. 1985. *A Comparative Study of Specific Yield Determinations for a Shallow Sand Aquifer, Ground Water*, Vol. 22, No. 6, pp. 764-772.

Walton, W.C., 1962. *Selected Analytical Methods for Well and Aquifer Evaluation, Illinois State Water Survey* Bulletin 19.

Attachment 3M
Standard Groundwater Sampling for Monitoring Wells

Standard Groundwater Sampling for Monitoring Wells

Rev. #: 1

Rev Date: July 16, 2008

Approval Signatures

Prepared by: Sonya A Cadle Date: 7/16/08

Reviewed by: Date: 7/16/08
(Technical Expert)

I. Scope and Application

This Standard Operating Procedure (SOP) describes the procedures to be used to collect groundwater samples using traditional purging and sampling techniques. For low-flow purging techniques, please refer to the Low Flow Purging SOP. Monitoring wells must be developed after installation at least 1 week prior to groundwater sample collection. Monitoring wells will not be sampled until the well has been developed. During precipitation events, groundwater sampling will be discontinued until precipitation ceases or a cover has been erected over the sampling area and monitoring well.

Both filtered and unfiltered groundwater samples may be collected using this SOP. Filtered samples may be obtained using a 1.0-, 0.45-, or 0.1-micron disposable filter.

II. Personnel Qualifications

ARCADIS personnel directing, supervising, or leading groundwater sample collection activities should have a minimum of 2 years of previous groundwater sampling experience. Field employees with less than 6 months of experience should be accompanied by a supervisor (as described above) to ensure that proper sample collection techniques are employed.

III. Equipment List

The following materials shall be available, as required, during groundwater sampling:

- site plan of monitoring well locations and site Field Sampling Plan (FSP);
- appropriate health and safety equipment, as specified in the site Health and Safety Plan (HASP);
- photoionization detector (PID) or flame ionization detector (FID), as needed, in accordance with the HASP;
- monitoring well construction logs or tables and historical water level information, if available;
- dedicated plastic sheeting or other clean surface to prevent sample contact with the ground;
- if bailers are to be used in sampling:

  - appropriate dedicated bottom-loading, bottom-emptying bailers (i.e., polyvinyl chloride [PVC], Teflon, or stainless steel);
  - polypropylene rope;
- if submersible pumps are to be used in sampling:
  - dedicated tubing and other equipment necessary for purging;
  - generator or battery for operation of pumps, if required;
  - a pump selected in accordance with the FSP or Work Plan (parameter-specific [e.g., submersible, bladder, peristaltic]);
- graduated buckets to measure purge water;
- water-level or oil/water interface probe, in accordance with the FSP or Work Plan;
- conductivity/temperature/pH meter;
- down-hole dissolved oxygen meter, oxidation reduction potential meter, and/or turbidity meter, if specified in the FSP;
- water sample containers appropriate for the analytical method(s) with preservative, as needed (parameter-specific);
- filter, as needed, in accordance with the analytical method and parameter;
- appropriate blanks (trip blank supplied by the laboratory), as specified in the FSP;
- Ziploc-type freezer bags for use as ice containers;
- appropriate transport containers (coolers) with ice and appropriate labeling, packing, and shipping materials;
- appropriate groundwater sampling log (example attached);
- chain-of-custody forms;
- site map with well locations and groundwater contour maps;

- keys to wells and contingent bolt cutters for rusted locks and replacement keyed-alike locks; and

- drums or other containers for purge water, as specified by the site investigation derived waste (IDW) management plan.

IV. Cautions

If heavy precipitation occurs and no cover over the sampling area and monitoring well can be erected, sampling must be discontinued until adequate cover is provided. Rain water could contaminate groundwater samples.

Remember that field logs and some forms are considered to be legal documents. All field logs and forms should therefore be filled out in indelible ink.

It may be necessary to field filter some parameters (e.g., metals) prior to collection, depending on preservation, analytical method, and project quality objectives.

Check monitoring well logs for use of bentonite pellets. Make note of potential use of bentonite pellets on the groundwater sampling log. Coated bentonite pellets have been found to contaminate monitoring wells with elevated levels of acetone.

Store and/or stage empty and full sample containers and coolers out of direct sunlight.

To mitigate potential cross-contamination, groundwater samples are to be collected in a pre-determined order from least impacted to more impacted based on previous analytical data. If no analytical data are available, samples are to be collected in the following order:

1. First sample the upgradient well(s).

2. Next, sample the well located furthest downgradient of the interpreted or known source.

3. The remaining wells should be progressively sampled in order from downgradient to upgradient, such that the wells closest to the interpreted or known source are sampled last.

Be careful not to over-tighten lids with Teflon liners or septa (e.g., 40 mL vials). Over-tightening can impair the integrity of the seal.

V. Health and Safety Considerations

If thunder or lighting is present, discontinue sampling until 30 minutes have passed after the last occurrence of thunder or lighting.

VI. Procedure

The procedures to sample monitoring wells will be as follows:

1. Don safety equipment, as required in the HASP. Depending on site-specific security and safety considerations, this often must be done prior to entering the work area.

2. Review equipment list (Section III above) to confirm that the appropriate equipment has been acquired.

3. Record site and monitoring well identification on the groundwater sampling log, along with date, arrival time, and weather conditions. Also identify the personnel present, equipment utilized, and other relevant data requested on the log.

4. Label all sample containers with indelible ink.

5. Place plastic sheeting adjacent to the well for use as a clean work area, if conditions allow. Otherwise, prevent sampling equipment from contacting the ground or other surface that could compromise sample integrity.

6. Remove lock from well and if rusted or broken, replace with a new brass keyed-alike lock.

7. Unlock and open the well cover while standing upwind of the well. Remove well cap and place on the plastic sheeting.

8. Set the sampling device,meters, and other sampling equipment on the plastic sheeting. If a dedicated sampling device stored in the well is to be used, this may also be set temporarily on the plastic sheeting, for convenience. However, if a dedicated sampling device is stored below the water table, removing it may compromise water-level data, so water level measurements should be taken prior to removing the device.

9. Obtain a water-level depth and bottom-of-well depth using an electric well probe and record on the groundwater sampling log using indelible ink. Clean the probe(s) after each use in accord with the FSP or the equipment

decontamination SOP.

Note: Water levels may be measured at all wells prior to initiating any sampling activities, depending on FSP requirements.

10. Calculate the number of gallons of water in the well using the length of water column (in feet). Record the well volume on the groundwater sampling log using indelible ink.

11. Remove the required purge volume of water from the well (measure purge water volume in measuring buckets). The required purge volume will be three to five well volumes (the water column in the well screen and casing) unless the well runs dry, in which case, the water that comes into the well will be sampled (USEPA, 1996). In any case, the pumping rate will be decreased during sampling to limit the potential for volatilization of organics potentially present in the groundwater.

12. Field parameter measurements will be periodically collected in accord with FSP specifications. The typical time intervals of field parameter measurement are (1) after each well volume removed, and (2) before sampling. If the field parameters are being measured above-ground (rather than with a downhole probe), then the final pre-sampling parameter measurement should be collected at the reduced flow rate to be used during sampling. The physical appearance of the purged water should be noted on the groundwater sampling log. In addition, water level measurements should be collected and recorded to verify that the well purging is in accord with the guidelines set forth in the previous step.

13. Unless otherwise specified by the applicable regulatory agencies, all purge water will be contained. Contained purge water will be managed in accordance with the FSP or Work Plan. If historical concentrations in the well are less than federal or state regulated concentrations appropriate for current land use, *and permission has been granted by the oversight regulatory agency* to dispose of clean purge water on the ground next to the well(s), then purge water will be allowed to infiltrate into the ground surface downgradient from the monitoring well after the well is sampled.

14. After the appropriate purge volume of groundwater in the well has been removed, or if the well has been bailed dry and allowed to recover, obtain the groundwater sample needed for analysis with the dedicated bailer or from the dedicated sampling tubing, pour the groundwater directly from the sampling device into the appropriate container in the order of volatilization sensitivity of

the parameters sampled, and tightly screw on the cap (snug, but not too tight). The suggested order for sample parameter collection, based on volatilization sensitivity, is presented below:

a. volatile organic compounds (VOCs);

b. semi-volatile organic compounds (SVOCs);

c. polychlorinated biphenyls (PCBs)/pesticides;

d. metals; and

e. wet chemistry.

15. When sampling for volatiles, water samples will be collected directly from the bailer or dedicated tubing into 40 mL vials with Teflon-lined septa.

16. For other analytical samples, sample containers for each analyte type should be filled in the order specified by the FSP. If a bailer is used, then the sample for dissolved metals and/or filtered PCBs should either be placed directly from the bailer into a pressure filter apparatus or pumped directly from the bailer with a peristaltic pump, through an in-line filter, into the pre-preserved sample bottle. If dedicated sample tubing is used, then the filter should be installed in-line just prior to filtered sample collection.

17. If sampling for total and filtered metals and/or PCBs, a filtered and unfiltered sample will be collected. Sample filtration for the filtered sample will be performed in the field utilizing a pump prior to preservation. Attach (clamp) a new 1.0-, 0.45-, or 0.1-micron filter to the discharge tubing of the pump (note the filter flow direction). Turn the pump on and allow 100 mL (or manufacturer recommended amount) of fluid through the filter before sample collection. Dispense the filtered liquid directly into the laboratory sample bottles. If bailers are used for purging and sampling, a proper volume of purge water will be placed in a disposable or decontaminated polyethylene container and pumped through the filter and into the sample container using a peristaltic pump.

18. Place the custody seal around the cap and the sampler container, if required. Note the time on the sample label. Secure with packing material and maintain at approximately 4°C on wet ice contained in double Ziploc-type freezer bags during storage in an insulated, durable transport container.

19. Replace the well cap and lock well, or install a new lock if needed.

20. Record the time sampling procedures were completed on the appropriate field logs (using indelible ink).

21. Complete the procedures for chain-of-custody, handling, packing, and shipping. Chain-of-custody forms should be filled out and checked against the labels on the sample containers progressively after each sample is collected.

22. Place all disposable sampling materials (such as plastic sheeting, disposable tubing or bailers, and health and safety equipment) in appropriate containers.

23. If new locks were installed, forward copies of the keys to the client Project Manager (PM) and ARCADIS PM at the end of the sampling activities.

VII. Waste Management

Purge water will be managed as specified in the FSP or Work Plan, and according to state and/or federal requirements. Personal protective equipment (PPE) and decontaminated fluids will be contained separately and staged at the sampling location. Containers must be labeled at the time of collection. Labels will include date, location(s), site name, city, state, and description of matrix contained (e.g., soil, groundwater, PPE). General guidelines for IDW management are set forth in a separate IDW management SOP.

VIII. Data Recording and Management

Initial field logs and chain-of-custody records will be transmitted to the ARCADIS PM at the end of each day unless otherwise directed by the PM. The groundwater team leader retains copies of the groundwater sampling logs. All field data should be recorded in indelible ink.

IX. Quality Assurance

Field-derived quality assurance blanks will be collected as specified in the FSP, depending on the project quality objectives. Typically, field rinse blanks will be collected when non-dedicated equipment is used during groundwater sampling. Field rinse blanks will be used to confirm that decontamination procedures are sufficient and samples are representative of site conditions. Trip blanks for VOCs, which aid in the detection of contaminates from other media, sources, or the container itself, will be kept with the coolers and the sample containers throughout the sampling activities.

X. References

USEPA. 1986. RCRA Groundwater Monitoring Technical Enforcement Guidance Document (September 1986).

USEPA. 1991. Handbook Groundwater, Volume ii Methodology, Office of Research and Development, Washington, DC. USEPN62S, /6-90/016b (July, 1991).

U.S. Geological Survey (USGS). 1977. National Handbook of Recommended Methods for Water-Data Acquisition: USGS Office of Water Data Coordination. Reston, Virginia.

Attachment 3N

Surface and Subsurface Soil Sampling Using Manual Methods

ARCADIS
Infrastructure, environment, facilities

Imagine the result

Surface and Subsurface Soil Sampling Using Manual Methods

Rev. #: 1

Rev Date: March 6, 2009

Approval Signatures

Prepared by: Michael J Gefell Date: 3/6/09

Reviewed by: Todd P. Date: 3/6/09
(Technical Expert)

I. Scope and Application

This document describes procedures for surface and subsurface soil sampling using hand tools.

II. Personnel Qualifications

ARCADIS personnel directing, supervising, or leading soil sampling activities should have a minimum of 2 years of previous environmental soil sampling experience. ARCADIS personnel providing assistance to soil sample collection and associated activities should have a minimum of 6 months of related experience or an advanced degree in environmental sciences.

III. Equipment List

The following materials will be available, as required, during soil sampling activities:

- personal protective equipment (PPE), as specified by the site Health and Safety Plan (HASP);
- stainless steel bowls;
- stainless steel spoons;
- stainless steel spades;
- stainless steel hand augers;
- indelible ink pens;
- engineer’s ruler or survey rod;
- sealable plastic bags (e.g., Ziploc®);
- equipment decontamination materials
- sample bottles and preservatives appropriate for the parameters to be sampled for laboratory analysis, if any;
- transport container with ice (if sampling for laboratory analysis);
- appropriate sample containers and forms; and

- field notebook and/or personal digital assistant (PDA).

Documentation forms and notebooks to have on hand include: soil sample log forms, chain-of-custody forms, sample labels and seals, field logbook/PDA.

IV. Cautions / Hazards

Task specific Job Safety Analysis (JSAs) must be developed to identify site hazards associated with the investigation and reviewed by all field crew members prior to the start of work. Safe Performance Self-Assessment (SPSA) to be performed by employees before performing a new task. Underground utilities will be cleared per the ARCADIS Utility Location Policy and Procedure.

V. Health and Safety Considerations

Soil sample collection will be performed in accordance with a site-specific Health and Safety Plan (HASP) and task specific JSA forms, copies of which will be present on site during such activities.

VI. Procedure

Soil samples may be collected at intervals from the ground surface to various depths. Sample locations will be identified using stakes, flagging, or other appropriate means, and will be noted in a field logbook, PDA, and/or soil sampling logs. Sample points will be located by surveying, use of a global positioning system (GPS), and/or measurements from other surveyed site features.

1. Equipment that will come in contact with the soil sample should be cleaned in accordance with the appropriate equipment decontamination SOP(s), or else new, disposable equipment should be used. Collect equipment blanks in accordance with the project Quality Assurance Project Plan (QAPP).

2. Clear the ground surface of brush, root mat, grass, leaves, or other debris.

3. Use a spade, spoon, scoop, or hand auger to collect a sample of the required depth interval.

4. Use an engineer's ruler to verify that the sample is collected to the correct depth and record the top and bottom depths from the ground surface.

5. To collect samples below the surface interval, remove the surface interval first; then collect the deeper interval. To prevent the hole from collapsing, it may be

necessary to remove a wider section from the surface or use cut polyvinyl chloride (PVC) tubing or pipe to maintain the opening.

6. Collect samples for volatile organic compounds (VOCs) as discrete samples using Encore® samplers or cut syringes (see Extraction/Preservation of Soil/Sediment Samples for VOCs SOP).

7. Homogenize samples for other analyses across the required interval or mix them with other discrete grab samples to form a composite sample (see Compositing or Homogenizing Samples SOP).

8. Place sample in clean sample container; label with sample identification number, date, and time of collection; and place on ice (if obtained for laboratory analysis). Prepare samples for packaging and shipping to the laboratory in accordance with the Chain-of-Custody Handling, Packing, and Shipping SOP.

9. Backfill sample holes to grade with native material or with clean builder's sand or other suitable material.

VII. Waste Management

Waste soils will be managed as specified in the FSP or Work Plan, and according to state and /or federal requirements. Personal Protective Equipment (PPE) and decontamination fluids will be contained separately and staged at the project site for appropriate disposal. Waste containers must be a sealed and labeled at the time of generation. Labels will indicate date, sample locations, site name, city, state, and description of the matrix (e.g., soil, PPE).

VIII. Data Recording and Management

Field documentation such as log book entries and chain-of –custody records will be transmitted to the ARCADIS PM or Task Manager each day unless otherwise directed. The field team leader will retain all site documentation while in the field and add to project files when the field mobilization is complete.

IX. Quality Assurance

Quality assurance samples (rinse blanks, duplicates, and MS/MSDs) will be collected at the frequency specified in the FSP and/or QAPP and depending on the project quality objectives. Reusable soil sampling equipment will be cleaned prior to use following equipment cleaning SOP. Field rinse blanks will be used to confirm that decontamination procedures are sufficient and samples are representative of site conditions. Any deviations from the SOP will be discussed with the project manager prior to changing any field procedures.

Attachment 3O
Test Pit Excavation (Non-Entry)

ARCADIS
Infrastructure, environment, facilities

Imagine the result

Test Pit Excavation (NON-ENTRY)

Rev. #: 2

Rev Date: May 28, 2008

Approval Signatures

Prepared by: Andrew Kanik Date: 5/28/2008

Reviewed by: Michael J Gefell Date: 5/28/2008
(Technical Expert)

I. Scope and Application

This SOP outlines policies and procedures for the advancement of test-pits using rubber-tire or track-mounted backhoes. For all work activities conducted by ARCADIS involving test pits or other excavations, ARCADIS staff will refer to and comply with ARCADIS HS Procedure No. ARC HSCS005, Excavation and Trenching. Test pits will be excavated using a decontaminated, rubber-tired backhoe or track-hoe as appropriate. Test pits may be performed based on the need to identify subsurface structures, facilitate the collection of soil samples and provide larger-scale subsurface characterization than allowed using soil borings. Personnel should stand upwind of the excavation area to the extent possible. Continuous air monitoring may be conducted as indicated in the site Health and Safety Plan (HASP). Excavating will be conducted at the selected locations that have been cleared for utilities until significant source materials, groundwater, or bedrock is encountered, or the purpose of the test pit has been met, or the physical limits of the backhoe have been reached. Test pit materials will be visually observed and described with respect to depth. Samples may be collected for laboratory or geotechnical analyses. Photographs of the test pits and excavated materials should be taken for future reference.

II. Personnel Qualifications

ARCADIS personnel overseeing, directing, or supervising the sampling portion of the test pit activities will have a minimum of 6 months of previous related experience under the supervision of an experienced (2 years) oversight person and at a minimum a 4-year degree (Bachelors) in environmental sciences, engineering, hydrogeology, or geology, and have completed health and safety training as required by OSHA Regulation 29 CFR 1910.120 (HAZWOPER). Personnel will also have completed any client-specific training as may be required. If the test pit is excavated by ARCADIS personnel, a competent person as defined by ARC HSCS005 will be on-site at all times.

If the test pit is excavated by a subcontractor, the subcontractor will provide the competent person per OSHA 1926.32(f). The excavation subcontractor will maintain all appropriate licenses and/or certifications as required by the State and Municipality. The equipment operator and any assistants working on site will, prior to beginning work, have completed all health and safety and other training as may be required by ARCADIS and the client.

III. Equipment List

The following equipment will be available, as required, during test pitting:

- rubber-tired (or track-mounted) backhoe in good working order;
- flame ionization detector (FID) and/or photoionization detector (PID), and/or other colorimetric;
- sample containers and forms;
- daily field log and/or field notebook;
- supplies and equipment to comply with site- and client-specific health and safety procedures;
- stainless steel shovel, scoop, hand auger, or trowel;
- digital camera;
- polyethylene sheeting; and
- ground stakes.

IV. Cautions

Water used for decontamination of excavation equipment will be of a quality acceptable for project objectives. Testing of water supply should be considered.

Work may be conducted on or in proximity to steep terrain. Site-specific health and safety issues will be thoroughly reviewed by all site personnel prior to beginning work.

V. Health and Safety Considerations

A site-specific Health and Safety Plan (HASP) meeting client requirements will be prepared along with Job Safety Analyses (JSAs) that outline the H&S hazards and controls for conducting the test pit activities. Project staff will review and be familiar with these plans and JSAs prior to work. These documents will detail the excavation safety requirements per ARC HSCS005. In addition, underground and above ground utilities will be located and cleared per ARCADIS H&S Procedure ARC HSFS019 – Utility Location.

VI. Procedures

Where necessary to characterize soil conditions, soil samples will be collected from the backhoe bucket, either directly or with a decontaminated stainless steel scoop or trowel.

Samples should be homogenized, if appropriate.

Material removed from the test pits during excavation will be placed on polyethylene sheeting. Visually clean soils will be segregated from soils that may contain source materials. Soils meeting field screening or laboratory analytical criteria may be placed back into the excavation. Soils not meeting screening or laboratory analytical criteria will be managed on site as described in the *Waste Management* section below. For sites that cannot be fully secured, clean fill will be available to backfill excavations immediately upon completion of test pits. To facilitate surveying, the location of the test pits will be marked with stakes after they have been backfilled. Stakes should be placed at the ends of the test pit and at any significant bend or corner, as appropriate.

VII. Waste Management

All water generated during decontamination procedures will be collected and contained onsite in 55-gallon drums or a temporary frac-tank pending laboratory analysis and appropriate disposal.

Personal protective equipment (such as gloves, disposable clothing, and other disposable equipment) resulting from personnel cleaning procedures and soil sampling/handling activities will be placed in plastic bags. These bags will be transferred into appropriately labeled 55-gallon drums for appropriate disposal.

Depending on volume generated, soil materials will be placed in sealed 55-gallon steel drums or stockpiled on site (placed on and covered by plastic sheeting). The material will be analyzed to determine the appropriate disposal method.

VIII. Data Recording and Management

The supervising geologist/engineer/scientist will be responsible for documenting activities using a daily field log to record all relevant information in a clear and concise format. As an alternative, a bound field notebook may be used at the discretion of field personnel to document field activities. Where appropriate, photographs will be taken to supplement written notes. The record of test pitting will include:

- start and finish dates of excavating;

- name and location of project;
- project number, client, and site location;
- sample number and depths;
- depth to water;
- observations of soil type/characteristics and lithology;
- documentation of any elevated organic vapor emissions;
- names of Contractor’s personnel, inspectors, or other people onsite; and
- weather conditions.

IX. Quality Assurance

Equipment will be cleaned prior to use onsite. At the discretion of the ARCADIS Project Manager or field geologist/engineer/scientist, equipment may be decontaminated between each test pit location, and prior to leaving the site. All equipment and associated tools that may have come in contact with contaminated soils and/or waste materials will be cleaned with high-pressure steam cleaning equipment using a potable water source. More detailed equipment cleaning procedures are provided in the HASP.

X. References

United States Department of Labor. 1989. Occupational Safety & Health Administration (OSHA), Title 29 Code of Federal Regulations (CFR)Part 1926.651 Subpart P Excavations, .54 Federal Register (FR) 45959, October 31, 1989 and 59 FR 40730, Aug. 9, 1994.

ARCADIS HS Procedure No. ARC HSCS005, Excavation and Trenching, 12 May 2008.

ARCADIS H&S Procedure ARC HSFS019 – Utility Location, 22 February 2008

Attachment 3P
Water-Level and NAPL Thickness Measurements Procedures

ARCADIS
Infrastructure, environment, facilities

Imagine the result

Water-Level and NAPL Thickness Measurement Procedures

Rev. #: 0

Rev Date: February 27, 2009

Approval Signatures

Prepared by: Andrew Korik Date: 2/27/09
Andrew Korik

Reviewed by: Michael J Gefell Date: 2/27/09
Michael Gefell (Technical Expert)

I. Scope and Application

Monitoring well water levels and thickness of non-aqueous phase liquids (NAPLs) will be determined, as appropriate, to develop groundwater elevation contour maps and to assess the presence or absence of NAPL in wells. This SOP applies to light and/or dense NAPLs (LNAPLs and DNAPLs, respectively). In addition, because this SOP describes water-level measurement from surveyed measurement points, this SOP can be followed, to obtain surface water level measurements from surveyed measurement points.

Fluid levels will be measured using an electric water-level probe and/or NAPL-water interface probe from established reference points. Reference points are surveyed, and are established at the highest point at the top of well riser, and will be based on mean sea level, or local/onsite datum. The Operating and Maintenance (O&M) Instruction Manual for the electric water level probe and/or and interface probe should be reviewed prior to commencing work for safe and accurate operation.

II. Personnel Qualifications

Individuals conducting fluid level measurements will have been trained in the proper use of the instruments, including their use for measuring fluid levels and the bottom depth of wells. In addition, ARCADIS field sampling personnel will have current health and safety training including 40-hour HAZWOPER training, site supervisor training, site-specific training, first aid, and CPR, as needed. In addition, ARCADIS field sampling personnel will be versed in the relevant SOPs and posses the required skills and experience necessary to successfully complete the desired field work. ARCADIS field personnel will also be compliant with client-specific training requirements, such as (but not limited to) LPS or other behavior-based training, and short-service employee restrictions.

III. Equipment List

The following materials, as required, shall be available during fluid level measurements.

- photoionization detector (PID)
- appropriate health and safety equipment, as specified in the site Health and Safety Plan (HASP)

- laboratory-type soap (Alconox or equivalent), methanol/hexane rinse, potable water, distilled water, and/or other equipment that may be needed for decontamination purposes
- electronic NAPL-water interface probe
- electronic water-level meter
- 6-foot engineer's rule
- portable containers
- plastic sheeting
- field logbook and/or personal digital assistant (PDA)
- indelible ink pen
- digital camera (optional, if allowed by site policy)

IV. Cautions

Electronic water-level probes and NAPL-water interface probes can sometimes produce false-positive readings. For example, if the inside surface of the well has condensation above the water level, then an electronic water-level probe may produce a signal by contacting the side of the well rather than the true water level in the well. In addition, NAPL-water interface probes can sometimes indicate false positive signals when contacting a sediment layer on the bottom of a well. In contrast, a NAPL-water interface probe may produce a false-negative (no signal) if a floating layer of non-aqueous phase liquid (NAPL) is too thin, such as a film or sheen. To produce reliable data, the electronic water level probe and/or interface probe should be raised and lowered several times at the approximate depth where the instrument produces a tone indicating a fluid interface to verify consistent, repeatable results. In addition, a bottom-loading bailer should periodically be used to check for the presence of NAPLs rather than relying solely on the NAPL-water interface probe.

The graduated tape or cable with depth markings is designed to indicate the depth of the electronic sensor that detects the fluid interface, but not the depth of the bottom of the instrument. When using these devices to measure the total well depth, the additional length of the instrument below the electronic sensor must be added to the apparent well depth reading, as observed on the tape or cable of the instrument, to obtain the true total depth of the well. If the depth markings on the tape or cable are worn or otherwise difficult to read, extra care must be taken in obtaining the depth readings.

V. Health and Safety Considerations

The HASP will be followed, as appropriate, to ensure the safety of field personnel. Access to wells may expose field personnel to hazardous materials such as contaminated groundwater or NAPL. Other potential hazards include stinging insects that may inhabit well heads, other biologic hazards, and potentially the use of sharp cutting tools (scissors, knife). Appropriate personal protective equipment (PPE) will be worn during these activities. Field personnel will thoroughly review client-specific health and safety requirements, which may preclude the use of fixed/folding-blade knives.

VI. Procedure

Calibration Procedures

If there is any uncertainty regarding the accuracy of the tape or cable associated with the electronic water-level probe or NAPL-water interface probe, it should be checked versus a standard length prior to use to assess if the tape or cable above the meter has been correctly calibrated by the manufacturer, and to identify evidence of tape or cable stretching, etc.

1. Measure the lengths between markers on the cable with a 6-foot engineer’s rule or a fiberglass engineer’s tape. The tape or cable associated with the electronic water-level probe or NAPL-water interface probe should be checked for the length corresponding to the deepest total well depth to be monitored during the data collection event.

2. If the length designations on the tape or cable associated with the electronic water-level probe or NAPL-water interface probe are found to be incorrect, the probe will not be used until it is repaired by the manufacturer.

3. Record verification of this calibration process in field logbook or PDA.

Measurement Procedures

The detailed procedure for obtaining fluid level depth measurements is as follows. Field notes on logs will be treated as secured documentation and indelible ink will be used. As a general rule, the order of measuring should proceed from the least to most contaminated monitoring wells, based on available data.

1. Identify site and well number in field logbook using indelible ink, along with date, time, personnel, and weather conditions.

2. Field personnel will avoid activities that may introduce contamination into monitoring wells. Activities such as dispensing gasoline into vehicles or generators should be accomplished well in advance of obtaining field measurements.

3. Don PPE as required by the HASP..

4. Clean the NAPL/water interface probe and cable in accordance with the appropriate cleaning procedures. Down-hole instrumentation should be cleaned prior to obtaining readings at the first monitoring well and upon completion of readings at each well.

5. Clean the NAPL/water level interface probe and cable with a soapy (Alconox) water rinse followed by a solvent rinse (if appropriate based on site-specific constituents of concern) an analyte-free water rinse Contain rinse water in a portable container that will be transferred to an on-site container.

6. Put clean plastic sheeting on the ground next to the well.

7. Unlock and open the well cover while standing upwind from the well. Place the well cap on the plastic sheeting.

8. Locate a measuring reference point on the well casing. If one is not found, initiate a reference point at the highest discernable point on the inner casing (or outer if an inner casing is not present) by notching with a hacksaw, or using an indelible marker. All down-hole measurements will be taken from the reference point established at each well on the inner casing (on the outer only if an inner casing is not present).

9. Measure to the nearest hundredth of a foot and record the height of the inner and outer casings (from reference point, as appropriate) to ground level.

10. Record the inside diameter of the well casing in the field log.

11. If an electronic water level probe is used to measure the water level, lower the probe until it emits a signal (tone and or light) indicating the top of the water surface. Gently raise and lower the instrument through this interface to confirm its depth. Measure and record the depth of the water surface, and the total well depth, to the nearest hundredth of a foot from the reference point at the top of

the well. Lower the probe to the bottom of the well to obtain a total depth measurement.

12. If a NAPL/water interface probe is being used to measure the depth and thickness of NAPL, lower the instrument until it emits a signal (tone and or light) indicating whether LNAPL is present. Continue to lower the NAPL/water level interface probe until it indicates the top of water. Lower the probe to the bottom of the well to obtain a total depth measurement. Note also of the depth indicating the bottom of water and top of DNAPL layer, if any, based on the signal emitted by the interface probe. At each fluid interface, gently raise and lower the instrument through each the interface to confirm its depth. Measure to the nearest hundredth of a foot and record the depth of each fluid interface, and the total well depth, from the reference point.

13. Clean the NAPL/water interface probe and cable in accordance with the appropriate cleaning procedures.

14. If using a bailer to confirm the presence/absence of NAPL, the bailer should either have been previously dedicated to the well, or be a new previously unused bailer.

15. Compare the depth of the well to previous records, and note any discrepancy.

16. Lock the well when all activities are completed.

VII. Waste Management

Decontamination fluids, PPE, and other disposable equipment will be properly stored on site in labeled containers and disposed of properly. Be certain that waste containers are properly labeled and documented in the field log book. Review appropriate waste management SOPs, which may be state- or client-specific.

VIII. Data Recording and Management

Fluid level measurement data will be recorded legibly on “write-in-the-rain” field notebook in indelible pen and/or a PDA. Field situations such as apparent well damage or suspected tampering, or other observations of conditions that may result in compromised data collection will be photographically documented where practicable.

IX. Quality Assurance

As described in the detailed procedure, the electronic water-level meter and/or NAPL-water interface probe will be calibrated prior to use versus an engineer's rule to ensure accurate length demarcations on the tape or cable. Fluid interface measurements will be verified by gently raising and lowering the instrument through each interface to confirm repeatable results.

X. References

No literature references are required for this SOP.

ARCADIS

Appendix B

Health and Safety Plan

ARCADIS
Infrastructure, environment, buildings

Imagine the result

Chevron Environmental Management Company

Environmental Health and Safety Plan (E-HASP)

Chevron Facility # 6518040
Former Gulf Petroleum Terminal
Oceanside, NY

June 2010

ARCADIS

Barb Schnurr

Barbara Schnurr
Designated H&S Plan Writer

Greg Mason
Designated H&S Plan Reviewer

William McCune
Project Manager

Environmental Health and Safety Plan (E-HASP)

Chevron Facility #6518040
Former Gulf Petroleum Terminal
Oceanside, NY

Prepared for:
Chevron Environmental Management Company

Prepared by:
ARCADIS U.S., Inc.
1 International Boulevard
Suite 406
Mahwah
New Jersey 07495
Tel 201.684.1410
Fax 201.684.1420

Our Ref.:
B0047517.0003.00001

Date:
June 2010

1. Introduction **1**

2. Purpose **2**

3. Components of the HASP **3**

4. Project Site History and Requirements **4**

4.1 Site Background 4

4.2 Site Description 4

4.3 List of Project Tasks and Scope of Work 4

5. ARCADIS Organization and Responsibilities **6**

5.1 Project Manager/Task Manager 6

5.2 Other Project Team Responsibilities 6

6. Hazard Control **8**

6.1 Project Hazard Analysis Worksheet 8

6.2 Chevron Hazard ID Tool 9

6.3 Job Loss Analyses (JLAs), H&S Procedures and PPE 12

6.4 Field Health & Safety Handbook 13

7. Hazard Communication (HazCom) **17**

7.1 Chemical Hazards 17

8. Tailgate Meetings **20**

9. Personal Exposure Monitoring and Respiratory Protection **21**

9.1 Respirator Cartridge Change Schedule 23

10. Medical Surveillance **24**

11. General Site Access and Control **25**

12. Emergency Action Plan (EAP) **26**

13. Client-Specific Health and Safety Requirements **27**

14. Department of Transportation (DOT) Dangerous Goods Shipping Requirements **28**

14.1 Materials of Trade (MOT) 28

14.2 Department of Transportation 28

15. Loss Prevention System^{TM} (LPS^{TM}) and Loss Prevention Observations (LPOs) **30**

16. Subcontractors 31

17. Project Personnel HASP Certification 32

Tables

Table 1. Master Chemical and Storage List 17

Table 2A. Chemical Hazard Information 17

Table 2B. Historical Maximum Contaminant Levels at Site 19

Table 3. Exposure Monitoring Requirements 21

Figures

Figure 1. HARC- Risk Assessment Matrix (H&S Procedure ARC HSMS002) 8

Appendices

A HASP Addendum Pages and Log Table

B Project Hazard Analysis Worksheet

C JLAs and Procedures

D PPE Equipment List

E Forms and MSDSs

<u>Forms</u>:

- i. Daily Safety Meeting Form / Permit-to-Work and High-Risk Permit-to-Work Forms
- ii. Air Monitoring Log
- iii. Employee Signature Form
- iv. Subcontractor Acknowledgement: Receipt of HASP Signature Form
- v. Visitor Acknowledgement and Acceptance of HASP Signature Form
- vi. Hazardous Materials Transportation Form
- vii. Hazardous Materials Shipment Form
- viii. Site-Specific HASP Worker and Visitors Tests
- ix. Management of Change Form
- x. Management of Change Tracking Log
- xi. Underground / Overhead Utilities Checklist, Chevron Guidance Document and Borehole Clearance Form
- xii. Daily / Periodic Excavation Inspection Form
- xiii. Standard Work Request Template

F Emergency Action Plan and Guidance

G Chevron-Specific Requirements

H Journey Management Plan

I Investigative-Derived Waste SOP

Chevron Project Manager HASP Acceptance

Chevron Environmental Management Company (CEMC) has reviewed this HASP and has accepted that it meets the objectives of the Operational Excellence (OE) Guidelines.

Peter L KASBOHM
Printed Name

Peter Kasbohm
Signature

6-16-2010
Date

1. Introduction

All work on this project will be carried out in compliance with ARCADIS' Health and Safety policies and procedures, and the Occupational Safety and Health Administration's Hazardous Waste Operations and Emergency Response regulation 29 CFR 1910.120. The design of this health and safety plan (HASP) conforms to the requirements of the ARC HSFS010 (HASP H&S Procedure). Specific health and safety information for the project is contained in this HASP. All personnel working on hazardous operations or in the area of hazardous operations shall read and be familiar with this HASP before doing any work. All project personnel shall sign the certification page acknowledging that they have read and understand this HASP.

Changes in the scope of the project or introduction of new hazards to the project shall require revision of the HASP by the HASP writer and reviewer, and approval by the Project Manager. The HASP Addendum Form and log table are included as Appendix A.

Appendix G of this E-HASP describes Chevron-specific policies, procedures and/or recommendations that must be followed in addition to requirements detailed in the site E-HASP. When ARCADIS and Chevron policies or procedures overlap, personnel are expected to adhere to the most stringent version. It is not the intent of this E-HASP to provide specific recommendations when policies conflict or overlap; therefore, personnel are expected to be familiar with policy variations in order to make informed decisions regarding compliance with company and client policies and procedures.

2. Purpose

The purpose of this HASP is to ensure a comprehensive safety program is in place at every Chevron project site. The plan serves to identify and communicate health, environmental and safety hazards that may be present at the job site and provides information to assist the workers in mitigating the risks associated with these hazards.

3. Components of the HASP

The following documents or booklets are referenced in this HASP and must accompany this HASP at all times:

1. Employee Field Health & Safety Handbook

2. LPS Handbook

3. Work Request

4. Project Site History and Requirements

4.1 Site Background

The former Chevron/Gulf Petroleum Terminal is located at 1 Industrial Place, Oceanside, Town of Hempstead, Nassau County, New York hereafter referred to as “the site”. The site is listed under the Brownfield Cleanup Program (BCP) Site No. C130165 and New York State Department of Environmental Conservation (NYSDEC) Spill No. 92-03883 and consists of a 7.223-acre property. The site is currently unoccupied and all former buildings have been demolished. The site is bound to the south by Barnum’s Channel, to the east by the Long Island Railroad, to the north by the former Exxon Petroleum Terminal, and to the west by Hampton Road. A junk yard and office buildings are located west of Hampton Road fronting Hog Island Channel. Daly Boulevard is located north of the former Exxon Petroleum Terminal and another petroleum terminal (Sprague) is located north of Daly Boulevard.

4.2 Site Description

Site Type (Check as many as applicable):

	Active		Secure	X	Industrial		Landfill		Service station
X	Inactive		Unsecured		Commercial		Well field		Water work
			Uncontrolled		Residential		Railroad	X	Undeveloped
Other specify:									

As shown on Figure 1, the site is located on a peninsula with Barnum’s Channel located to the south and Hog Island Channel located to the west. An unnamed channel is located east of the peninsula. The site topography is approximately 5 feet above sea level.

4.3 List of Project Tasks and Scope of Work

The RI scope of work consists of the following:

1. Monitoring well integrity inspection.
2. Monitoring well installation
3. Excavation
4. Soil borings.
5. Pump test

6. Dye trace

7. Groundwater monitoring and sampling

5. ARCADIS Organization and Responsibilities

5.1 Project Manager/Task Manager

In planning and preparation of this project, the project manager and/or task manager has completed the project-specific H&S Stewardship Checklist & Project Hazard Analysis Worksheet. The project Hazard Analysis Worksheet was completed using the Hazard Analysis Risk Control (HARC) ranking process (ARCADIS H&S Procedure ARC HSMS002) (see Section 4 of this HASP). Additional responsibilities of the project manager and task manager are as follows:

- Review all applicable H&S Procedures, and ensure that project activities conform to all requirements.
- Obtain client-specific health and safety information and communicate with the client on health and safety issues.
- Communicate with the Site Safety Officer (SSO) on health and safety issues.
- Allocate resources for correction of identified unsafe work conditions.
- Ensure ARCADIS site workers have all training necessary for the project.
- Report all injuries, illnesses and near-misses to the Client H&S Resource or Project H&S Manager (PHSM), lead incident investigations, and ensure that any recommendations made are implemented.

5.2 Other Project Team Responsibilities

Additional personnel designated to carry out H&S job functions for the project, and their responsibilities are listed below. The same person may fill more than one role:

ARCADIS Project Team	Responsibility and Tasks
To Be Determined	**SSO** • Reviews and works in accordance with the components of this HASP. • Ensures that this HASP is available to and reviewed by all site personnel including subcontractors. • Ensures that necessary site-specific training is performed (both initial and “tailgate” safety briefings. • Ensures site visitors have been informed of the hazards related to ARCADIS work, and have signed the Site Visitors Log. • Ensures that work is performed in a safe manner and has authority to stop work when necessary to protect workers and/or the public. • Coordinates activities during emergency situations.

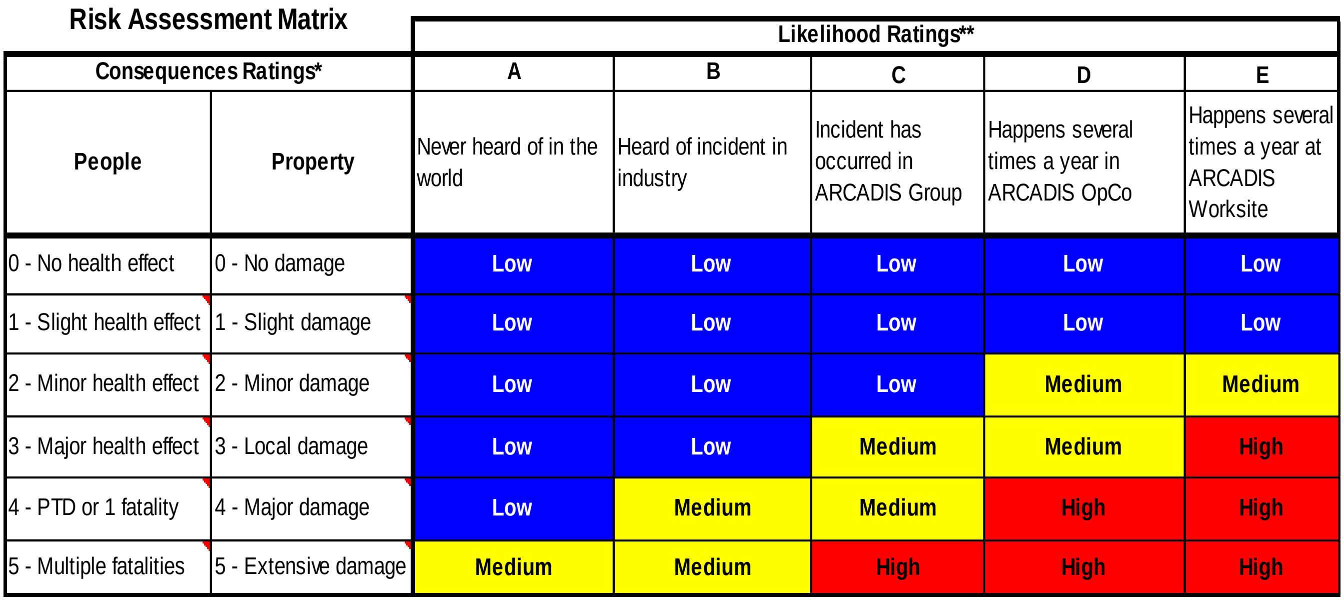
ARCADIS Project Team	Responsibility and Tasks
	• Ensures that all necessary permits and safety information provided by the client is disseminated to other site personnel and is maintained in an organized manner. • Communicates with the PM, Client H&S Resource and/or the PHSM on health and safety issues. • Reports all injuries, illnesses and near-misses to the PM, Client H&S Resource and PHSM. • Ensures that necessary safety equipment is maintained and used at the site. • Contacts a health and safety professional for assistance in establishing the respiratory cartridge change schedule as required.
To Be Determined	**Site Workers** • Reads and works in accordance with the components of this HASP. • Reports all unsafe working conditions to the SSO. • Reports all injuries, no matter how minor, to the SSO. • Works in a safe manner. • Signs the HASP acceptance log in Appendix E.
Chris Merrifield	**Project Health and Safety Manager (PHSM)** The PHSM oversees all aspects of the site safety program, and prepares site-specific health and safety guidance documents or addenda to this plan. The PHSM does not report to the Project Manager, and is separately accountable to the ARCADIS project team for site health and safety. The PHSM acts as the sole contact to regulatory agencies on matters of safety and health. Other responsibilities include: • Overall authority for health and safety compliance and HASP conformance for the project. • General health and safety program administration. • Conducts project health and safety audits as warranted. • Determines the level of personal protection required. • Updates equipment or procedures based on information obtained during site operations. • Establishes air-monitoring parameters based on expected contaminants. • Assists in injury, illness and near-miss investigations and follow-up.
Greg Mason	**Client Health and Safety Resource** The designated Client H&S Resource is responsible for : • Assisting the SSO in issues as they arise. • Performing site audits and assessments. • Assisting with near-miss/incident investigations. • Serves as the liaison with corporate during H&S regulatory issues as they may arise.

6. Hazard Control

6.1 Project Hazard Analysis Worksheet

Preparation of the Project Hazard Analysis Worksheet in Appendix B is critical in determining the hazards associated with the scope(s) of work at this project site. The project team should identify and document the general tasks to be completed at the project site (i.e., groundwater monitoring and sampling, soil sampling, monitoring well installation, excavation, remediation system operation and maintenance, etc.) on the “Hazard Analysis 1” tab of the worksheet. Using the HARC – Risk Assessment Matrix (Figure 4) as a guide, the project development team should evaluate the hazards associated with each scope of work, document controls for each hazard and combine them on the “Hazard Analysis 2” tab of the worksheet.

Figure 4. HARC- Risk Assessment Matrix (H&S Procedure ARC HSMS002)



Risk Assessment Matrix

Consequences Ratings*		Likelihood Ratings**				
		A	B	C	D	E
People	Property	Never heard of in the world	Heard of incident in industry	Incident has occurred in ARCADIS Group	Happens several times a year in ARCADIS OpCo	Happens several times a year at ARCADIS Worksite
0 - No health effect	0 - No damage	Low	Low	Low	Low	Low
1 - Slight health effect	1 - Slight damage	Low	Low	Low	Low	Low
2 - Minor health effect	2 - Minor damage	Low	Low	Low	Medium	Medium
3 - Major health effect	3 - Local damage	Low	Low	Medium	Medium	High
4 - PTD or 1 fatality	4 - Major damage	Low	Medium	Medium	High	High
5 - Multiple fatalities	5 - Extensive damage	Medium	Medium	High	High	High

The Hazard Analysis Worksheet is provided in **Appendix B** of this HASP.

The Risk Assessment Matrix can also be used in the field to evaluate and control risks as they arise. When practiced in the context of H&S Procedure ARC HSMS002, hazard control becomes an ongoing exercise that supports a safer working environment through continuous planning and preparation.

6.2 Chevron Hazard ID Tool

The Chevron Hazard ID Tool is designed as an additional hazard recognition tool for the control of 10 types of hazardous energy on the job. The objectives in using this tool are:

- provide you with a method to identify energy sources in your environment.
- enable you to identify potential hazards associated with energy sources.
- enhance your hazard assessment skills through the integration of this tool.

What is this Hazard Identification Tool?

- A visual aid that helps you focus on hazard identification
- A tool that helps you identify hazards based on energy source identification
- A simple method to help you complete daily activities and tasks safely and reliably
- A tool which easily integrates with existing hazard assessment methodologies such as Job Loss Analysis, the TRACK tool and philosophy, the STOP WORK authority principle, etc.

Each energy source is briefly described below.

Gravity

The force caused by the attraction of all other masses to the mass of the earth.

Examples: falling object, collapsing roof, and a body tripping or falling.

Motion

The change in position of objects or substances.

Examples: vehicle, vessel or equipment movement; flowing water; wind; and body positioning: lifting, straining, or bending.

Mechanical

The energy of the components of a mechanical system, i.e. rotation, vibration, motion, etc. within otherwise stationary piece of equipment/machinery.

Examples: rotating equipment, compressed springs, drive belts, conveyors, and motors.

Electrical

The presence and flow of an electric charge.

Examples: power lines, transformers, static charges, lightning, energized equipment, wiring, and batteries.

Pressure

Energy applied by a liquid or gas which has been compressed or is under a vacuum.

Examples: pressure piping, compressed cylinders, control lines, vessels, tanks, hoses, and pneumatic and hydraulic equipment.

Temperature

The measurement of differences in the thermal energy of objects or the environment, which the human body senses as either heat or cold.

Examples: open flame; ignition sources; hot or cold surfaces, liquids or gases; steam; friction; and general environmental and weather conditions.

Chemical

The energy present in chemicals that inherently, or through reaction, has the potential to create a physical or health hazards to people, environment or equipment.

Examples: flammable vapors, reactive hazards, carcinogens or other toxic compounds, corrosives, pyrophorics, combustibles, oxygen-deficient atmospheres, welding fumes, and dusts.

Biological

Living organisms that can present a hazard.

Examples: animals, bacteria, viruses, insects, blood-borne pathogens, improperly handled food, and contaminated water.

Radiation

The energy emitted from radioactive elements or sources and naturally occurring radioactive materials (NORM).

Examples: lighting issues, welding arcs, solar rays, microwaves, lasers, X-Rays, and NORM scale.

Sound

Sound is produced when a force causes an object or substance to vibrate—the energy is transferred through the substance in waves.

Examples: equipment noise, impact noise, vibration, high-pressure release, and the impact of noise to communication.

The hierarchy of controls are:

1. Remove energy source

2. Prevent release

3. Protect from release

4. Stop Work Authority

6.3 Job Loss Analyses (JLAs), H&S Procedures and PPE

A JLA has been completed for each safety critical task. The list of JLAs is available from the Chevron One Team Incident Prevention System (OTIPs) database are included in Appendix C. Hazards identified on the Project Hazard Analysis Worksheet are addressed in the JLAs as well as control methods to protect employees and property from hazards. The JLA also lists the type of personal protective equipment (PPE) required for the completion of the project. A detailed list of PPE for the project is located in Appendix D.

Note: These JLAs, called “seed” JLAs by Chevron, are to serve as the basis of understanding and controlling the hazards expected to be encountered on the project site. These seed JLAs must be obtained from the OTIPs database, printed out, and taken into the field. It must be understood by all field personnel that these seed JLAs were developed based on the hazard analysis process in the office and may not include all hazard information that becomes present on site. Therefore, it is important that field personnel, using the TRACK concept and LPSA process, evaluate any hazards and control measures not identified in the seed JLA.

It is expected that seed JLAs be edited in the field and discussed with personnel in daily tailgate meetings (see Section 8.0 – Tailgate Meetings). These slightly altered seed JLAs do not require tracking on the HASP addendum forms. However, if there is a significant change in a seed JLA due to a lessons learned, addition or deleted of job step, change in equipment, etc. then the ARCADIS PM, PHSM and individual(s) with experience with the task must be consulted prior to proceeding with the work activity. This may require the modification of the seed JLA in the OTIPS database. The newly numbered JLA should be identified in the HASP.

ARCADIS H&S Procedures applicable to this project are listed below. These procedures should be reviewed by the project manager, task manager and site personnel. The Client H&S Resource should be contacted with any questions concerning the procedures.

- ARC HSFS019 – Utility Location
- ARC HSCS005 – Excavation and Trenching
- ARCHSGE001 – Tailgate Health and Safety Meetings
- ARCHSGE015 – Personal Protection Equipment
- ARCDOT302 – Private Roadway Work Zone Safety

6.4 Field Health & Safety Handbook

The Field H&S Handbook is an ARCADIS document containing information about topic-specific health and safety requirements for the field. This handbook contains relevant general topics and is used as part of the overall HASP process. To aid in the consistency of the HASP process the handbook will be used as an informational source in conjunction with this HASP. The following handbook sections are minimally required reading for this project:

II. Health and Safety Administration

- A. ARCADIS Responsibilities
- B. Employee Responsibilities
- C. Visitors
- D. Communications
- E. HARC Process (ARCHSMS002)
- F. Incident Investigation (ARCHSMS010)
- G. Near-miss Reporting
- H. Stop Work Authority
- I. Short-service Employee Program
- J. Workplace Violence and Harassment
- K. Drug and Alcohol Awareness
- L. Emergency Action Plan
- M. WorkCare
- N. Subcontractors

O. Regulatory Inspections (ARCHSG013)

P. Recordkeeping and Postings

III. General Field Health & Safety Requirements

A. Daily Safety Meetings/Tailgates

B. Department of Transportation – HAZMAT Transportation/Dangerous Goods (ARCHSGED02)

C. First-aid/CPR

D. Blood-borne Pathogens (ARCHSIH005)

E. General Health and Safety Rules and Safe Work Permits

F. General Housekeeping, Personal Hygiene and Field Sanitation

G. Site Security, Work Zone and Decontamination for HAZWOPER Sites

H. Personal Safety and Other Unique Site Conditions

I. Severe Weather

J. Fire Prevention

K. Hazard Communication

L. Noise

M. Heat and Cold Stress

N. Biological Hazards

P. Medical Surveillance (ARCHSGE010)

Q. Field Office General Health and Safety Requirements

R. Personal Protective Equipment (ARCHSGE015)

T. Vehicle Safety Inspection

U. Driving

X. Boating Operations Safety

AA. Electrical Safety (ARCHSFS006)

CC. Hand and Power Tools

EE. Ergonomics

FF. Site Storage and Hazardous Chemicals, Gases and Solvents

GG. HAZWOPER and HAZMAT Response (ARCHSSF012)

II. Drum and Other Material Handling

KK. Signs, Signals and Barricades

LL. Traffic Control (ARCHSFS017)

MM. Utility Location (ARCHSF019)

NN. Backing Safety

IV. General Field Construction Health and Safety Best Management Practices

A. Excavation/Trenching (ARCHSCS005)

B. Heavy Equipment (ARCHSCS006)

C. Temporary Working Surfaces

D. Steel Erection (ARCHSCS010)

E. Rollover and Overhead Protection

F. Permit to Work

G. Specific Work Areas, Hazards and Activities

H. Roadway

I. Water Operations Work

J. Industrial Hygiene and Monitoring Equipment

7. Hazard Communication (HazCom)

All project required chemicals must be handled in accordance with OSHA 29 CFR 1910.1200, ARCADIS-HazCom Procedure (ARC HSGE007), and the requirements outlined in the Field H&S Handbook. Table 1 lists all chemicals that will be brought, used, and/or stored on the site by ARCADIS or its subcontractors. Material Safety Data Sheets (MSDS) for chemicals brought on site are included in Appendix E.

Table 1. Master Chemical and Storage List

Chemical Name	Estimated Quantity	Chemical Storage Location
Hydrochloric Acid (HCl)	<100mL	Laboratory preserved sample bottles
Nitric Acid (HNO_3)	<100mL	Laboratory preserved sample bottles
Sodium Hydroxide (NaOH)	<100mL	Laboratory preserved sample bottles
Sulfuric Acid (H_2SO_4)	<100mL	Laboratory preserved sample bottles
Alconox / Liquinox	As needed	Field Vehicle
Marking Paint	As needed	Field Vehicle
Fluourecin	1 gal	Field Vehicle

7.1 Chemical Hazards

Air monitoring will be conducted as outlined in this HASP to collect exposure data for chemicals of concern (COC) or for chemicals brought onsite for use. Table 2 lists the properties of chemicals that will be encountered at the site.

Table 2A. Chemical Hazard Information

Chemical Name	IP[1] (eV)	Odor Threshold (ppm)	Routes of Entry/ Exposure Symptoms	8-hr TWA[2] (ppm)	IDLH[3] (NIOSH) (ppm)	STEL[4] (ppm)	Source TLV/PEL
Benzene	9.24	1.5	Inhalation, Skin Absorption, Ingestion, Skin/Eye Contact. Irritation to the eyes, skin, nose, respiratory system; dizziness; headache, nausea, staggered gait; anorexia, lassitude (weakness, exhaustion); dermatitis; bone marrow depression; [potential occupational carcinogen]	0.5	500	2.5	TLV

Chemical Name	IP[1] (eV)	Odor Threshold (ppm)	Routes of Entry/ Exposure Symptoms	8-hr TWA[2] (ppm)	IDLH[3] (NIOSH) (ppm)	STEL[4] (ppm)	Source TLV/PEL
1,2 dichloroethane	11.05	6-40	Depressed cns, nausea, vomiting, dermatitis, irritated eyes, corneal opacity, liver, kidney and cardiovascular damage. Carcinogenic	50 10 1	Ca (50ppm)	C 100; 200* 2 ppm *5-min peak in any 3 hrs	PEL TLV REL
Cis-1,2 dichloroethene	9.65	NA	Irritated eyes, nausea, vomiting, and epigastric distress. Symptoms of exposure-related narcosis including drowsiness, tremor, incoordination, dizziness, and weakness; defatting of skin/dermatitis	200	1000	None listed	PEL TLV REL
MTBE	ND	ND	Irritated eyes; central nervous system effects; lightheadedness, dizziness, weakness; nausea; headache; muscular disturbances; skin irritant; damage to eye tissue	40	NA	NA	PEL TLV REL
Trichloroethene	9.45	0.5-167 ppm	Irritation eyes, skin; headache, visual disturbance, lassitude (weakness, exhaustion), dizziness, tremor, drowsiness, nausea, vomiting; dermatitis; cardiac arrhythmias, paresthesia; liver injury; [potential occupational carcinogen]	100 ppm 10 ppm 25 ppm (10-hr)	Ca (1,000 ppm)	C200 ppm; 300 ppm* 25 ppm C2 ppm (60 min) *5min peak in	PEL TLV REL
Vinyl chloride	9.99	10-20 ppm	Lassitude (weakness, exhaustion); abdominal pain, gastrointestinal bleeding; enlarged liver; pallor or cyanosis of extremities; liquid: frostbite; [potential occupational carcinogen]	1 ppm	None listed	5 ppm* *avg not exceeding any 15 min	None listed

Notes:

[1] IP-ionization potential in eV-electronvolts
[2] TWA-time-weighted average
[3] IDLH-immediately dangerous to life and health
[4] STEL-short-term exposure limit (either 10 or 15 minutes)

See Section 8 for information on air monitoring requirements.

Table 2B. Historical Maximum Contaminant Levels at Site

Date	Chemical Name	Location	Type	Concentration
1/2007	Benzene	MW-34D	Groundwater	69.9 ppb
1/2007	1,2 dichloroethane	MW-32D	Groundwater	2.1 ppb
1/2007	Cis-1,2 dichloroethene	MW-24VD	Groundwater	7.8 ppb
1/2007	MTBE	MW-36D	Groundwater	176 ppb
3/2006	Trichloroethene	MW-25D2	Groundwater	800,000 ppb

8. Tailgate Meetings

Tailgate safety briefings will be conducted at least daily at the beginning of the work day, or as tasks/hazards change. Each tailgate safety briefing will be documented on the form included in Appendix E.

9. Personal Exposure Monitoring and Respiratory Protection

Personal and area exposure monitoring will be documented on the Real Time Exposure Monitoring Data Form provided in Appendix E. All monitoring equipment will be maintained and calibrated in accordance with manufacturer's recommendations. All pertinent monitoring data will be logged on the form and maintained on site for the duration of project activities. Calibration of all monitoring equipment will be conducted daily and logged on the same form.

Table 3 lists exposure monitoring requirements and associated action levels for site exposure hazards (e.g. chemical, noise, radiation, etc). Action levels have been developed for exposure monitoring with real-time air monitoring instruments as specified in the table. Air monitoring data will determine the required respiratory protection levels at the Site during scheduled intrusive activities. The action levels are based on sustained readings indicated by the instrument(s). Air monitoring will be performed and recorded at up to 30-minute intervals.

If elevated concentrations are indicated, the monitoring frequency will be increased, as appropriate. If sustained measurements are observed during this time, the following actions will be instituted, and the Project Manager and Project Health and Safety Manager will be notified. For purposes of this HASP, sustained readings are defined as the average airborne concentration maintained for a period of one (1) minute.

Table 3. Exposure Monitoring Requirements

TASK 1 – Is exposure monitoring required for the completion of this project? ☐ YES ☒ NO If yes, complete the following:				
Exposure Hazard	**Monitoring Equipment**	**Monitoring Frequency**	**Action Level**	**Required Action**
NA	NA	NA	NA	NA
TASKS 2, 3, 4 – Is exposure monitoring required for the completion of this project? ☒ YES ☐ NO If yes, complete the following:				
Exposure Hazard	**Monitoring Equipment**	**Monitoring Frequency**	**Action Level**	**Required Action**
Total Hydrocarbons	Photo Ionization Detector (PID) 10.6 eV lamp or greater	Continuous in breathing zone/ Work zone perimeter	≤0.5 ppm	Normal operations
			> 0.5 ppm	Begin Monitoring for benzene with colorimetric tubes
			> 0.5 ppm, ≤20 ppm	Normal operations
			> 20 ppm	Upgrade to level C PPE
			> 200 ppm	Stop work and investigate cause of reading; contact SSO/PM

Exposure Hazard	Monitoring Equipment	Monitoring Frequency	Action Level	Required Action
Benzene	Colorometric tube	As dictated by PID readings	≤0.5 ppm > 0.5 ppm, ≤1.0 ppm > 20.0 ppm	Normal operations Upgrade to level C PPE Stop work and investigate cause of reading; contact SSO/PM
TASKS 2, 3, 4 – Is exposure monitoring required for the completion of this project? ☒ YES ☐ NO If yes, complete the following:				
Exposure Hazard	**Exposure Hazard**	**Exposure Hazard**	**Exposure Hazard**	**Exposure Hazard**
Particulates	PDR 1000 Data RAM	Continuous in breathing zone/ Work zone perimeter	0.5 mg/m^3 (sustained for 5 minutes)	Institute dust control measures. If wetting techniques fail to keep below action level, Stop Work. Contact SSO/PM. If exceedences continue reassess hazards/ implement engineering controls.
Oxygen	Multi-gas meter	Continuous in breathing zone/ prior to and during excavation entry	< 19.5% > 19.5% to <23.5% > 23.5%	Stop work, evacuate confined spaces/ work area, investigate cause of reading, and ventilate area Normal operations Stop work, evacuate confined spaces/work area, investigate cause of reading, and ventilate area Stop work, evacuate confined spaces/ work area, investigate cause of reading, and ventilate area
Carbon Monoxide	Multi-gas meter	Continuous in breathing zone/ prior to and during excavation entry	0 ppm to <20 ppm > 20 ppm	Normal operations Stop work, evacuate confined spaces/ work area, investigate cause of reading, and ventilate area
TASKS 2, 3, 4 – Is exposure monitoring required for the completion of this project? ☒ YES ☐ NO If yes, complete the following:				
Exposure Hazard	**Exposure Hazard**	**Exposure Hazard**	**Exposure Hazard**	**Exposure Hazard**
Hydrogen Sulfide	Multi-gas meter	Continuous in breathing zone/ prior to and during excavation entry	0 ppm to < 5 ppm > 5 ppm	Normal operations Stop work, evacuate confined spaces/ work area, investigate cause of reading, and ventilate area
Flammable Vapors (LEL)	Multi-gas meter	Continuous in breathing zone/ prior to and during excavation entry	< 10% LEL > 10% LEL	Normal operations Stop work, ventilate area, investigate source of vapors

9.1 Respirator Cartridge Change Schedule

Respirators will be stored in clean containers (i.e., self-sealing bag) when not in use. If respirators are required to be worn based on the action levels established above, respirator cartridges will be replaced in accordance with the following change-out schedule.

Type of Cartridge	Cartridge Change-out Schedule
Particulate (i.e., High Efficiency Particulate Air)	At least weekly or whenever the employee detects an increase in breathing resistance. This will occur as the filter becomes loaded with particulate matter.
Sorbent (i.e., organic vapor)	At the end of each day's use or sooner, if the respirator manufacturer change-out schedule software program dictates otherwise. The Project H&S Manager or the client H&s resource must be consulted regarding gas/vapor cartridge change-out schedule. This will be determined per the ARCADIS Respiratory Protection procedure – ARC HSGE017.

Personnel who wear APRs must be trained in their use, must have successfully passed a qualitative respiratory fit test in accordance with 29 CFR 1910.134 within the last 12 months, and must have medical clearance for APR use.

With the exception of protection against particulates*, if the action plan outlined above calls for an upgrade to an air-purifying respirator (for protection against organic vapors and other gaseous chemicals), the following will apply:

- The respirator cartridge will be equipped with an end-of-service-life indicator (ESLI) certified by NIOSH for the contaminant.

- If there is no ESLI appropriate for a contaminant, the project will implement a change schedule for cartridges to ensure that they are changed before the end of their service life.

**Note – A Cartridge Change Schedule is not necessary for cartridges used in the protection against particulates provided that the cartridges are changed out when there is a perceived resistance in breathing experienced by the user.*

10. Medical Surveillance

Medical surveillance requirements for the project are provided on the Project Manager/Task Manager H&S Stewardship Checklist & Project Hazard Analysis Worksheet (Appendix B). All medical surveillance requirements as indicated on the worksheet must be completed and site personnel medically cleared before being permitted on the project site.

11. General Site Access and Control

The SSO will coordinate access and control security at the work site. As the work dictates, the SSO will establish a work area perimeter. The size of the perimeter will be based on the daily task activities and will be discussed with all project personnel during the tailgate meeting and then documented on the tailgate meeting form. Control zones for Level C or above will be demarcated by either visual or physical devices and will be monitored for effectiveness by the SSO.

Only authorized personnel will be allowed beyond the perimeter. Other site workers and visitors to the site should be kept out of the work site. If visitors need access to the site, the SSO will escort the visitor at all times. All visitors will log in and out with the SSO. The visitor log sheet is included in Appendix E.

12. Emergency Action Plan (EAP)

In the event that an injury, over-exposure or spill has occurred, an EAP will be implemented. Appendix F provides the EAP and notifications for the project. All employees working on this project must be shown the location and proper use of all emergency equipment prior to beginning work on the project. The Response Guidance is a Chevron-specific requirement that must be followed in the event of an emergency situation (fire, spill, earthquake, etc.). Drills should be performed at the project site periodically. Drills should be documented on the General Permit to Work form.

13. Client-Specific Health and Safety Requirements

ARCADIS project personnel must comply with the client’s specific H&S requirements at all times. Client-specific H&S requirements are provided in Appendix G.

14. Department of Transportation (DOT) Dangerous Goods Shipping Requirements

ARCADIS has policies in place for transporting small quantities of hazardous materials and for offering for shipping via ground or air. These policies are designed to meet the applicable requirements. As such, only ARCADIS staff that have been trained in the proper methods to prepare and ship hazardous materials are authorized to do so. Tasks associated with the packaging, labeling, marking, and preparation of hazardous materials for shipping or transport must have all appropriate and applicable training.

14.1 Materials of Trade (MOT)

DOT allows for a small amount of hazardous materials that are used in or an inherent part of our work to be transported in company vehicles. This includes things like gasoline, paint, small compressed gas cylinders, calibration gas, etc. To transport these:

- Staff will complete Materials of Trade training.
- Vehicles used in transportation to and from off-site work locations will be in conformance with ARCADIS vehicle safety procedures.

Hazardous materials will be transported as described above as a result of the activities covered in this HASP. Site personnel who transport materials mentioned above will complete the Hazardous Materials Transportation Form included in Appendix E.

14.2 Department of Transportation

Staff who collect, prepare, package, mark, label, complete shipping declarations, offer shipments to a transporter, directly transport or are engaged in other activities associated with the transportation of Hazardous Materials (referred to as Dangerous Goods in Canada and by the International Air Transport Association [IATA]) will have appropriate and applicable training. DOT requires all individuals who participate in hazmat shipping including activities such as completing the paperwork (but not signing it), filling a container with a hazardous material (including filling a drum with drill cuttings or purge water), marking, labeling, and packaging the hazardous material, etc., have awareness level training on the DOT requirements. DOT requires additional job function training for those who conduct specific activities including:

- Staff who have to sign shipping papers or manifests, are listed as the 24-hour emergency contacts on shipping and have the responsibility for identifying, classifying, packaging, marking, and labeling HazMat packages, and/or are directing or overseeing others who do these tasks will become certified through the completion of additional training.

- The above training allows the offering employee to ship only by ground. If the shipment is to be offered for air transport, additional training is required.

Shipments as described above will be made as a result of the activities covered in this HASP. Site personnel shipping hazardous materials will complete the Hazardous Materials Shipment Form included in Appendix E.

15. Loss Prevention System^{TM} (LPS^{TM}) and Loss Prevention Observations (LPOs)

As part of any project, no matter how simple or complex, LPOs should be conducted when practical and when able to integrate into normal business activities. LPOs should be scheduled based on the risk of the tasks being performed, and should be conducted for different tasks and at different times. Completion of LPOs should be documented on the tailgate meeting form. See Section 1.2.2.2 of the Chevron-Specific HASP Requirements in Appendix G for frequency requirements and guidance on LPOs on Chevron projects.

16. Subcontractors

A copy of this HASP is to be provided to all subcontractors prior to the start of work so that the subcontractor is informed of the hazards at the site. While the ARCADIS HASP will be the minimum health and safety requirements for the work completed by ARCADIS and its subcontractors, each subcontractor, in coordination with ARCADIS health and safety personnel, is expected to perform its operations in accordance with its own HASP, policies and procedures unique to the subcontractor's work to ensure that hazards associated with the performance of the work activities are properly controlled. Copies of any required safety documentation for a subcontractor's work activities will be provided to ARCADIS for review prior to the start of on-site activities.

In the event that the subcontractor's procedures/requirements conflict with requirements specified in this HASP, the more stringent guidance will be adopted after discussion and agreement between the subcontractor and ARCADIS project health and safety personnel. Hazards not listed in this HASP, but known to the subcontractor or known to be associated with the subcontractor's services, must be identified and addressed to the ARCADIS project or task manager and SSO prior to beginning work operations.

If the subcontractor prefers to adopt this HASP, the **"Subcontractor Acknowledgement Memo" must be signed and dated by the subcontractor's management and placed in the project file.** Once the signed memo is received by the project manager, an electronic version of our HASP can be submitted to the subcontractor to use as their own. Subcontractors working at the site will need to have this plan with them, and will also need to sign the Subcontractor HASP receipt signature page of the ARCADIS HASP (Appendix E). Subcontractors are responsible for the H&S of their employees at all times, and have the authority to halt work if unsafe conditions arise.

The Project/Task Manager and SSO (or authorized representative) has the authority to halt the subcontractor's operations and to remove the subcontractor or subcontractor's employee(s) from the site for failure to comply with established health and safety procedures or for operating in an unsafe manner.

17. Project Personnel HASP Certification

All site project personnel will sign the certification signature page provided in Appendix E of this HASP.

ARCADIS

Appendix A

HASP Addendum Pages and Log Table

Addendum Page

This form should be completed for new tasks associated with the project. The project manager and/or task manager should revise the Project Hazard Analysis Worksheet with the new task information and attach to this addendum sheet. JLAs should be developed for any new tasks and attached as well.

Review the addendum with all site staff, including subcontractors, during the daily tailgate briefing, and complete the tailgate briefing form as required. Attach a copy of the addendum to all copies of the HASP including the site copy, and log in the Addendum Log Table A-1 on the next page.

Addendum Number: ____________________ Project Number: __________________________

Date of Changed Conditions: ____________________ Date of Addendum: __________________________

Description of Change that Results in Modifications to HASP:

Signed: ___________________________________
Project Manager

Signed: ___________________________________
Site Safety Officer

Signed: ___________________________________
H&S Plan Writer

Signed: ___________________________________
H&S Plan Reviewer

Addendum Log Table

Addendums are to be added to every copy of the HASP, and logged on Table A-1 to verify that all copies of the HASP are current:

Table A-1 Addendum Log Table

Addendum Number	Date of Addendum	Reason for Addendum	Person Completing Addendum
1			
2			
3			
4			
5			
6			
7			
8			
9			
10			

Chevron Project Manager Modifications to HASP Acceptance

Chevron Environmental Management Company (CEMC) has reviewed these HASP modifications and has accepted that they meet the objectives of the Operational Excellence (OE) Guidelines.

____
Printed Name

____ ____________________
Signature Date

Make additional copies of this form as necessary.

Appendix B

Project Hazard Analysis Worksheet

ARCADIS US Project Manager and Task Manager/Principal-In-Charge H&S Stewardship Checklist

Project Hazard Analysis Page

Project Name:	**RI/FS at Former Gulf Terminal, Oceanside, NY**		**Project Number:**	**B0047517.0003.00001**	
Client:	**Chevron EMC**		**Principal-In Charge:**	**John Vogeley**	
Project / Task Manager:	**William McCune**	**Completed By:**	**William McCune**	**Date:**	**19-Jun-10**

ARCADIS Project Hazard Analysis Worksheet

TRACK

Recognize and Assess the Hazards for the Project

For each potential hazard, determine the worst case conditions for the entire project and all of the tasks and assess them using High (H), Medium (M), Low (L). Use the drop down list in each "Assess" cell. If a hazard is not expected on the site, leave the "Assess" box blank.

	Recognize the Hazards	**Assess**	**Recognize the Hazards**	**Assess**	**List Types of other Physical Hazards Below**
Physical Hazards:	Heat		Holes/Pits	**Medium**	
	Cold		Ionizing Radiation	**Low**	
	Noise	**Low**	Non-ionizing Radiation	**Low**	
	Walking/Working surfaces (includes slip/trip/fall & floor/wall openings)	**Medium**	Electricity	**Low**	
	Visible Dust		Poor lighting	**Low**	
	LASER		Severe Weather	**Low**	
	Other:		Overhead Hazards	**Low**	
	Other:		None: Mark with an "X"		

Control the Hazard: (Briefly describe how the identified hazards will be controlled)

Chemical Hazards:	Flammable/ Combustible		Corrosive		**List the Names of the Major Chemicals Below**
	Compressed gas		Toxic	**Medium**	BTEX
	Explosive		Highly toxic		TCE
	Organic peroxide		Irritant		DCE
	Oxidizer		Sensitizer		
	Water reactive		Carcinogen	**Medium**	
	Unstable reactive		Mutagen		
	Dust/Fumes/ Particulates		None: Mark with an "X"		

Control the Hazard: (Briefly describe how the identified hazards will be controlled)

Use of appropriate care, monitoring and PPE will be used to mitigate potential for exposure to chemical hazards.

Environmental/ Equipment Hazards:	Heavy machinery	**Medium**	Cranes/Hoists/Rigging		**List Types of Other Environmental / Equipment Hazards Below**
	Trenching/excavation	**Medium**	Ladders		
	Docks – marine operations		Scaffolding		
	Construction activities		Manlifts		
	Diving operations		Welding		
	Drilling	**Medium**	Gas cylinders		
	Forklifts		Roadway work		
	Water operations work		Railroad work		
	Heights (fall protection)		Mining work		
	Overhead/ Underground utilities	**Medium**	Energized / Pressurized equip (LO/TO)		
	Confined spaces		Drums and containers	**Low**	
	Power tools		Other		
	Other		None: Mark with an "X"		

Control the Hazard: (Briefly describe how the identified hazards will be controlled)

Use of well marked exclusion areas and spotters during test pit excavation will be use to mitigate heavy equipment/excavation hazards.

Appropriate drum handling equipment and attention to pich points will serve to mitigate hazardss of drum handling.

Biological Hazards	Animal/Human fluids or blood		Contaminated Needles		**List Types of Other Biological Hazards Below**
	Animal/Human tissue(s)		Live Bacterial Cultures		
	Poisonous/irritating plants		Insects/rodents/snakes		
	Other:		None: Mark with an "X"	x	

Control the Hazard: (Briefly describe how the identified hazards will be controlled)

Although not anticipated, reasonable care will be be used to identify and mitigate biological hazards

Ergonomic Hazards	Repetitive motion		Limited movement		**List Types of Other Ergonomic Hazards Below**
	Awkward position		Forceful exertions		
	Heavy lifting		Vibration		
	Frequent lifting		Other:		
	Other:		None: Mark with an "X"	x	

Control the Hazard: (Briefly describe how the identified hazards will be controlled)

Although not anticipated, reasonable care will be be used to identify and mitigate ergonomic hazards

Personal Safety/Security	Personal safety		Employees working early/late		**List Types of Other Personal Safety / Security Hazards Below**
	Security issue		Potentially dangerous wildlife		
	Project site in isolated area		Guard or stray dogs in area		
	Employees working alone		No/limited cell phone service		
	Fatigue		Other:		
	Other		None: Mark with an "X"	x	

Control the Hazard: (Briefly describe how the identified hazards will be controlled)

Although not anticipated, reasonable care will be be used to identify and mitigate personal safety hazards

Driving Safety	Driving early/late		City driving		**List Types of Other Driving Hazards Below**
	Driving long trips		Pulling a trailer		
	Driving off-road		ATV driving:		
	Bad weather driving		Other		
	Other		None: Mark with an "X"	x	

Control the Hazard: (Briefly describe how the identified hazards will be controlled)

Although not anticipated, reasonable care will be be used to identify and mitigate driving related hazards.

Training Required	40 hour HAZWOPER		Bloodborne pathogens		**List Types of Other Training Required Here**
	24 hour HAZWOPER		Confined space		
	HAZWOPER site supervisor		Lockout/tagout		
	OSHA 30 hour Construction		Electrical Safety		
	OSHA 10 hour Construction		Fire Extinguishers		
	PPE		Fall Protection		
	Respiratory protection		Noise exposure		
	Chemical hygiene		Forklifts		
	Hazard communication		Asbestos		
	Hazardous waste		Lead		
	First-aid/CPR		Cadmium		
	DOT/IATA hazmat transportation		SPCC		
	MSHA		Radiation safety		
	Diving		Client specific		
	FRA		None: Mark with an "X"		
Medical Screening	Medical Surveillance Exam (HAZWOPER)		Other hazardous substance		**List Types of other Medical Screening Here**
	Pulmonary Function Test if wearing respirator and employee not part of HAZWOPER		Audiometric test if noise is a hazard and employee not part of HAZWOPER		
	Client required drug and/or alcohol testing		Blood and/or urine screening		
	Hepatitis B Immunization (or declination on file)		None: Mark with an "X"	x	

Keep Safety First In All Things

Appendix C

JLAs and Procedures

ARCADIS

Table C-1 JLAs List Table

OTIPs JLA #	Date	JLA Type	Work Type

Job Loss Analysis

General

Client Name	CHEVRON ENV MANAGEMENT
JSA ID	2224
Job Name	Environmental-Sample cooler handling
Task Description	Sample Cooler Handling
Project Number	B00475170003
Project Name	FACILITY NO. 6518040 - 2010 RI/FFS WORK
PIC Name	VOGELEY, JOHN
Project Manager	MCCUNE, WILLIAM
Status Name	(3) Completed
Cretaion Date	4/20/2010 8:30:20 AM

User Roles

Role	Employee	Due Date	Completed	Approve	Supervisor	Active Employee
Created By	Beier, Kathryn	5/11/2010	4/20/2010		Messinger, John	True

Job Steps

Job Step	Job Step Description		Potential Hazard	Critical Action	HSP Reference
1	Transfer field samples to sample packing area	1	Lifting heavy coolers may result in muscle strain especially to lower back.	Use proper lifting techniques and keep back straight. Use buddy system for large coolers, Use mechanical aids like hand trucks if readily available to move coolers. Do not over fill coolers with full sample containers for temporary movement to the sample prep area. Ensure an adequate supply of sample coolers are in field	
		2	Hazards to hands from broken glass caused by over tightening lids or improper placement in cooler	Inspect all bottles and bottle caps for cracks/leaks before and after filling container. Do not over tighten sample lids. Clean up any broken bottles immediately, avoid contact with sample preservatives. Wear leather gloves when handling broken glass.	
		3	Exposure to chemicals (acid preservatives or site contaminants) on the exterior of sample bottles after filling.	Wear protective gloves for acid preservatives and safety glasses with side shields during all sample container handling activities (before and after filling), Once filled follow project specific HASP PPE requirements for skin and eye protection.	
		4	Samples containing hazardous materials may violate DOT/IATA HazMat shipping regulations	All persons filling a sample bottle or preparing a cooler for shipment must have complete ARCADIS DOT HazMat shipping training. Compare the samples collected to the materials described in the Shipping Determination for the Project and ensure consistent. Re perform all Shipping determinations if free product is collected and not anticipated during planning.	

2	Sample cooler selection	1	Sample coolers with defective handles, lid hinges, lid hasps cracked or otherwise damaged may result in injury (cuts to hands, crushing of feet if handle breaks etc)	Only use coolers that are new or in like new condition, No rope handled coolers unless part of the manufacturer's handle design.	ARCADIS Shipping Guide US-001
		2	Selection of excessively large coolers introduces lifting hazards once the cooler is filled.	Select coolers and instruct lab to only provide coolers of a size appropriate for the material being shipped. For ordinary sample shipping sample coolers should be 48 quart capacity or smaller to reduce lifting hazards.	ARCADIS Shipping Guide US-001
3	Pack Samples	1	Pinch points and abrasions to hands from cooler lid closing unexpectedly	Beware that lid could slam shut; block/brace if needed; be wary of packing in strong winds. New coolers may be more prone to self closing, tilt cooler back slightly to facilitate keeping lid open.	
		2	Awkward body positions and contact stress to legs and knees when preparing coolers on irregular or hard ground surfaces.	Plan cooler prep activities. Situate cooler where neutral body positions can be maintained if practical, like truck tailgate. Avoid cooler prep on gravel rough surfaces unless knees and legs protected during kneeling.	
		3	Frostbite or potential for oxygen deficiency when packing with dry ice. Contact cold stress to fingers handling blue ice or wet ice	Dry ice temperature is -109.30F. Wear thermal protective gloves. DO NOT TOUCH with bare skin! Dry ice sublimates at room temp and could create oxygen deficiency in closed environment. Maintain adequate ventilation! Do not keep dry ice in cab of truck. Wear gloves when handling blue ice or gaging wet ice. Dry Ice is DOT regulated for air shipping, follow procedures in Shipping Determination.	
4	Sealing, labeling and Marking Cooler	1	Cuts to hands and forearms from strapping tape placement or removing old tape and labels	Do not use a fixed, open-blade knife to remove old tags/labels, USE SCISSORS or other safety style cutting device. Only use devices designed for cutting. Do not hurry through task.	
		2	Lifting and awkward body position hazards from taping heavy coolers, dropping coolers on feet during taping.	Do not hurry through the taping tasks, ensure samples in cooler are evenly distributed in cooler to reduce potential for overhanging cooler falling off edge of tailgate/table when taping.	
		3	Improper labeling and marking may result in violation of DOT/IATA Hama shipping regulations delaying shipment or resulting in regulatory penalty	Do not deviate from ARCADIS Shipping Guide or Shipping Determination marking or labeling requirements.	
5	Offering sample cooler to a carrer or lab couriour for shipment.	1	Lifting heavy coolers may result in muscle strain especially to lower back.	See lifting hazard controls above.	
		2	Carrier refusal to accept cooler may cause shipping delay and/or result in violation of DOT HazMat shipping regulations.	Promptly report all rejected and refused shipments to the ARCADIS DOT Program Manager. Do Not re-offer shipment if carrier requires additional labels markings or paperwork inconsistent with your training or Shipping Determination without contacting the ARCADIS DOT Compliance Manager.	

Personal Protective Equipment

Type	Personal Protective Equipment	Description	Required
Eye Protection	safety glasses		Required
Hand Protection	chemical resistant gloves (specify type)	nitrile	Required
Hand Protection	work gloves (specify type)	leather	Required

Supplies

Type	Supply	Description	Required
Miscellaneous	Other	Scissors	Required

Job Loss Analysis

General

Client Name	CHEVRON ENV MANAGEMENT
JSA ID	2222
Job Name	Environmental-Drilling, soil sampling, well installation
Task Description	Drilling with drill rig
Project Number	B00475170003
Project Name	FACILITY NO. 6518040 - 2010 RI/FFS WORK
PIC Name	VOGELEY, JOHN
Project Manager	MCCUNE, WILLIAM
Status Name	(3) Completed
Cretaion Date	4/20/2010 8:21:39 AM

User Roles

Role	Employee	Due Date	Completed	Approve	Supervisor	Active Employee
Created By	Beier, Kathryn	5/11/2010	4/20/2010		Messinger, John	True

Job Steps

Job Step	Job Step Description		Potential Hazard	Critical Action	HSP Reference
1	Set up necessary traffic and public access controls	1	Struck by vehicle due to improper traffic controls	Use a buddy system for placing site control cones and/or signage. Position vehicle so that you are protected from moving traffic. Wear Class II traffic vest	
2	Utility Clearance	1	Potential to encounter underground or aboveground utilities while drilling	Complete utility clearance in accordance with the ARCADIS H&S procedure	ARCADIS H&S Procedure ARCHSFS019
3	General drill rig operation	1	Excessive noise is generated by rig operation.	When the engine is used at high RPMs or soil samples are being collected, use hearing protection.	
		2	During drill rig operation, surfaces will become hot and cause burns if touched, and COCs in the soils more readily vaporize generating airborne contaminates.	Due to friction and lack of a drilling fluid, heat will be produced during this method. Mainly drill augers. Be careful handling split spoons. Wear proper work gloves. When soils and parts become heated, the COC could volatilize. Air monitoring should always be performed in accordance with the HASP.	
		3	Moving parts of the drilling rig can pull you in causing injury. Pinch points on the rig and auger connections can cause pinching or crushing of body parts.	Stay at least 5 feet away from moving parts of the drill rig. Know where the kill switch is, and have the drillers test it to verify that it is working. Do not wear loose clothing, and tie long hair back. Avoid wearing jewelry while drilling. Cone off the work area to keep general public away from the drilling rig	
		4	Dust and debris can cause eye injury and soil cuttings and/or water could contain COCs.	Wear safety glasses and stay as far away from actual drilling. W operation as practicable. Wear appropriate gloves to protect from COCs.	

3	General drill rig operation	5	Drilling equipment laying on the ground (i.e. augers, split spoons, decon equipment, coolers, etc), create a tripping hazard. Water from decon buckets generate mud and cause a slipping hazard.	Keep equipment and trash picked up, and store away from the primary work area.	
		6	The raised derrick can strike overhead utilities, tree limbs or other elevated items	Never move the rig with the derrick up. Ensure there is proper clearance to raise the derrick, and that you are far enough away from overhead power lines. See the Utility Location H&S policy and procedure for guidance.	
4	Mudd rotary drilling	1	The raised derrick can strike overhead utilities, tree limbs or other elevated items	Never move the rig with the derrick up. Ensure there is proper clearance to raise the derrick, and that you are far enough away from overhead power lines. See the Utility Location H&S policy and procedure for guidance.	
		2	This technology uses fluid, which collects with sediments in large basin. Fluid can splash out and cause slipping/mud hazard. Liquid mixture can splash into your eyes.	Wear rubber boots if needed, and keep clear of muddy/wet area as much as practicable. If area becomes excessively muddy, consider mud spikes or covering the area with a material that improves traction. Wear safety glasses.	
5	Hollow stem auger drilling	1	All hazards in step 3 apply. Additionally,The raised derrick can strike overhead utilities, tree limbs or other elevated items	Never move the rig with the derrick up. Ensure there is proper clearance to raise the derrick, and that you are far enough away from overhead power lines. See the Utility Location H&S policy and procedure for guidance.	
6	Air rotary drilling	1	this drilling method works with high air pressure and can generate flying debris that can strike your body or get debris in your eyes.	When the drill rig is being driven into media, it will produce flying debris. The flaps behind the drill rig should stay closed whenever possible to reduce the risk of flying debris. Safety glasses and hard hat should always be worn when the drill rig is operating. When penetrating asphalt protect surrounding cars that may be present to avoid debris damage to paint or winshields.	
		2	The raised derrick can strike overhead utilities, tree limbs or other elevated items	Never move the rig with the derrick up. Ensure there is proper clearance to raise the derrick, and that you are far enough away from overhead power lines. See the Utility Location H&S policy and procedure for guidance.	
		3	When drilling through bedrock prior to groundwater dust can be produced from pulverization. Inhalation of dusts/powder can occur	Supplemental water should be used to manage dust creation and/or dust masks if necessary.	
7	Reverse rotary drilling	1	This method will use fresh water to pump out drill cuttings through the center of the casing. Water/sediment mixture is generated and could cause contact with impacted soils or groundwater	Ensure the pit construction can hold the amount of cuttings that are anticipated. Air monitoring should also be used of pit area	

7	Reverse rotary drilling	2	Fire hydrants are often used for water source. Hydrants deliver water at high pressure. Pressurized water can cause flying parts/debris and excessive slipping hazards.	Water usage from fire hydrants should be cleared with local muncipalities prior to use. Only persons that know how to use the hydrant should be performing this task. Ensure all connections are tight, and hose line is not run over to cut by traffic. Any leaks from the hydrant should be reported immediately.	
		3	Settling pit construction can cause tripping hazard from excavated soils, and plastic sheeting can cause slipping.	cone off the area to keep the general public/visitors away from the settling pit. Ensure proper sloping of excavation.	
		4	The raised derrick can strike overhead utilities, tree limbs or other elevated items	Never move the rig with the derrick up. Ensure there is proper clearance to raise the derrick, and that you are far enough away from overhead power lines. See the Utility Location H&S policy and procedure for guidance.	
8	Rotosonic drilling	1	Fire hydrants are often used for water source. Hydrants deliver water at high pressure. Pressurized water can cause flying parts/debris and excessive slipping hazards.	Water usage from fire hydrants should be cleared with local muncipalities prior to use. Only persons that know how to use the hydrant should be performing this task. Ensure all connections are tight, and hose line is not run over to cut by traffic. Any leaks from the hydrant should be reported immediately	
		2	This method requires a lot of clearance. The drill head can turn 90 degrees to attach to the next drill flight or casing. This usually requires a large support truck to park directly behind the rig. As the drill head raises the new casing flight is angled down at the same time until it can be turned completely vertical.	Ensure sufficient overhead clearance.	
		3	Heavy lifting of cores can cause muscle strain.	Always use 2 people to move core containers. Use caution moving core samples to layout area. Plan layout area to ensure adequate aisle space between core runs for logging. Keep back straight and use job rotation.	
		4	The rotosonic drill head can move very quickly up and down while working on a borehole. Moving parts can strike someone or catch body parts	The operator and helper must communicate and stay clear of the path of the drill head. The drill utilizes two large hydrualic clamps to continuously hold casings while load/unloading previous casings. Do not wear loose clothing.	
9	Direct push drilling	1	The drill rods will be handled by workers most of the time rather than the rig doing it, therefore pinch points can cause lacerations and crushing of fingers/body parts.	Keep a minimum of 5 feet away from drill rig operation and moving parts.	
		2	The direct push rigs are uaually meant to fit in spaces where larger rig can't. Tight spaces can pin workers.	Do not put yourself between the rig and a fixed object. Use Spotters or a tape measure to ensure clearances in tight areas. Pre-plan equipment movement from one location to the next.	

9	Direct push drilling	3	some direct push equipment is controlled by wireless devices. These controls can fail and equipment can strike workers or cause damage to property.	The drill rig should be used in a large open area to test wireless controls prior to moving to boring locations. The operator of the rig will test the kill switch with wireless remote prior to use. Operator will stay in range of rig while moving so that wireless signal will not be too weak and cause errors to the controls.	
		4	Sampling sleeves must be cut to obtain access to soil. Cutting can cause lacerations.	Preferably let the driller cut the sleeves open. Many drillers have holders for the sleeve to allow for stability when cutting. If we cut the sleeves, use a hook blade, change blade regularly, and cut away from the body.	
10	Rock Coring	1	flying debris can hit workers or cause debris to get in eyes.	Rock chips or overburden may become airborne from drilling method. Wear safety glasses and hard hat and remain at a safe distance from back of drill rig.	
		2	Heavy lifting of cores can cause muscle strain.	Always use 2 people to move core containers. Use caution moving core samples to layout area. Plan layout area to ensure adequate aisle space between core runs for logging. Keep back straight and use job rotation.	
11	Sample collection and processing	1	Injuries can result from pinch points on sampling equipment, and from breakage of sample containers.	Care should be taken when opening sampling equipment. Look at empty containers before picking them up, and do not over-tighten container caps. Use dividers to store containers in the cooler so they do not break.	Sample cooler handling JLA
		2	lifting heavy coolers can cause back injuries	Use two people to move heavy coolers. Use proper lifting techniques.	Sample cooler handling JLA
12	Monitoring well installation	1	Same hazards as in Step 3 with general drill rig operation	See step 3	
		2	monitoring well construction materials can clutter the work area causing tripping hazards.	Well construction materials should be picked up during the well installation process.	
		3	Heavy lifting can cause muscle strains, and cutting open bags can cause lacerations.	Well construction materials are usually 50 lbs or greater. Team lift or use drill rig to hoist bags. Always use work gloves while cutting open bags.	
		4	Well pack material (i.e. sand, grout, bentonite) can become airborne and get in your eyes.	Wear safety glasses for protection from airborne sand and dust.	
		5	Cutting the top of the well to size can cause jagged/sharp edges on the top of the well casing.	Wear gloves when working with the top of the well casing, and file any sharp jagged edges that resulted from cutting to size.	
13	Soil cutting and purge water management	1	Moving full drums can cause back injury, or pinching/crushing injury.	Preferably have the drilling contractor move full drums with their equipment. If this is not practicable, use lift assist devices such as drum dollys, lift gates, etc. Employ proper lifting techniques, and perfrom TRACK to identify pinch/crush points. Wear leather work gloves, and clear all walking and work areas of debris prior to moving a drum.	Drum handling JLA

Personal Protective Equipment

Type	Personal Protective Equipment	Description	Required
Eye Protection	safety glasses		Required
Foot Protection	steel-toe boots		Required
Hand Protection	chemical resistant gloves (specify type)		Required
Hand Protection	work gloves (specify type)	leather	Required
Head Protection	hard hat		Required
Hearing Protection	ear plugs		Required
Miscellaneous PPE	traffic vest--Class II or III		Required
Respiratory Protection	dust mask		Recommended

Supplies

Type	Supply	Description	Required
Communication Devices	mobile phone		Required
Decontamination	Decon supplies (specify type)		Required
Miscellaneous	fire extinguisher		Required
Miscellaneous	first aid kit		Required
Personal	eye wash (specify type)	bottle	Required
Traffic Control	traffic cones		Required

Job Loss Analysis

General

Client Name	CHEVRON ENV MANAGEMENT
JSA ID	2225
Job Name	General Industry-Driving - passenger vehicles
Task Description	Driving to/from/onsite
Project Number	B00475170003
Project Name	FACILITY NO. 6518040 - 2010 RI/FFS WORK
PIC Name	VOGELEY, JOHN
Project Manager	MCCUNE, WILLIAM
Status Name	(3) Completed
Cretaion Date	4/20/2010 8:33:00 AM

User Roles

Role	Employee	Due Date	Completed	Approve	Supervisor	Active Employee
Created By	Beier, Kathryn	5/11/2010	4/20/2010		Messinger, John	True

Job Steps

Job Step	Job Step Description		Potential Hazard	Critical Action	HSP Reference
1	Preforming Pre-trip inspections	1	Cuts scrapes to hands and fingers checking engine fluids	Use TRACK to plan inspection activity in the engine compartment. Wear protective gloves if reaching in poorly illuminated areas of the engine.	
		2	Pinch crush hazards to hands and fingers checking engine fluids or closing doors.	Identify and keep hands fingers away from pinch hazards from doors and vehicle hood or tailgate (if present).	
		3	Awkward body positions checking tires, spare tire, undercarriage, or engine compartment.	Maintain neutral body positions and avoid awkward reaches under the vehicle or in engine compartment.	
		4	Failure to inspect vehicle emergency equipment may result in extensive vehicle damage or delay treatment in the event of injury	Conduct equipment inspections by visibly inspecting fire extinguisher and first aid kit for cleanliness, in date items/tags, readiness for use.	
2	Vehicle loading and unloading	1	Object placement obstructing rear, side or blindspot view	Avoid placing objects in a manner that obstructs your view, brake equipment down to a smaller more manageable size to keep low profile in vehicle. If hanging clothes in vehicle, place in manner that does not obstruct blind spots.	
		2	Unsecure objects causing pedal, steering or gear shift obstruction or injury during vehicle operation.	Secure all loads in vehicle (both in the bed of trucks and in passenger cabin) to prevent unanticipated movement or shifting that could injure driver, passenger, or affect safe operation of vehicle.	
		3	Obstuction of vehicle safety equipment caused by object placment in vehicle.	Keep emergency equipment clear and unobstructed to ensure ready availablity.	

3	Vehicle operation	1	Failure to use Smith System "5-Keys" increases risk of accident and injruy.	Use Smith System "5-Keys", maintain space cushion around vehicle, maintain 4 second rule and add (second for each addtional hazard (wet roads, snow, etc). Brake gradual, keep eyes moving, check mirrors every 6-8 seconds, use turn signals, focus on relavent objects, use early lane positioning when approaching turns.	
		2	Injury or death from failure to wear seatbelt	Always wear seatbelts even if driving short distances off of a public roadway.	
		3	Cell phone use increases risk of accidnt and injury	Avoid using cell phones in any capacity when operating a vehicle, check client for cell use on project sites and follow requirements. Follow all local laws.	
		4	Use of radar detectors encourages speeding resulting in increased risk for accident or injury	Use of radar detectors and similar devices is prohibited.	
4	Routine maintenance	1	Pinch crush hazards to hands and fingers replacing engine fluids or closing doors/hood.	Inspect and indetify pinch and crush hazards and keep hands/fingers clear when closing hood, tailgates, or doors.	
		2	Burn hazards to hand form checking/replacing fluids in engine compartment	When practical allow engine to cool prior to servicing or adding fluids. Use protective gloves.	
		3	Vehicle damage from improper fuse replacement	Never replace a fuse with a higher amperage than the one being replaced. Only replace fuses of type being replaced.	
		4	Failing to use Wright Express for vehicles equipped with fuel card impairs maintenace tracking that could affect vehicle safety	If vehicle is assigned a Wright Express Card, use the card so accurate maintenance tracking can be performed by LeasePlan.	

Supplies

Type	Supply	Description	Required
Communication Devices	mobile phone		Required
Miscellaneous	fire extinguisher		Required
Miscellaneous	first aid kit		Required
Traffic Control	Other	Roadway emergency kit	Required

Job Loss Analysis

General

Client Name	CHEVRON ENV MANAGEMENT
JSA ID	2223
Job Name	Environmental-Groundwater Sampling and free product recovery
Task Description	Groundwater Sampling and Free product recovery
Project Number	B00475170003
Project Name	FACILITY NO. 6518040 - 2010 RI/FFS WORK
PIC Name	VOGELEY, JOHN
Project Manager	MCCUNE, WILLIAM
Status Name	(3) Completed
Cretaion Date	4/20/2010 8:26:13 AM

User Roles

Role	Employee	Due Date	Completed	Approve	Supervisor	Active Employee
Created By	Beier, Kathryn	5/11/2010	4/20/2010		Messinger, John	True

Job Steps

Job Step	Job Step Description		Potential Hazard	Critical Action	HSP Reference
1	Stage at pre determined sampling location and set up work zone and sampling equipment	1	personnel could be hit by vehicluar traffic.	Set-up cones and establish work area. Position vehicle so that field crew is protected from site traffic. Unload as close to work area as safely possible.	
		2	Sampling equipment, tools and monitoring well covers can cause tripping hazard	Keep equipment picked up and use TRACK to assess and changes	
2	Open wells to equilibrate and gauge wells	1	When squatting down, personnel can be difficult to see by vehicular traffic.	Wear Class II traffic vest if wells are located proximal to vehicular traffic. Use tall cones and the buddy system if practicable.	
		2	pinchpoints on well vault can pinch or lacerate fingers	Use correct tools to open well vault/cap. Wear leather gloves when removing well vault lids, and chemical protective gloves while guaging. Wear proper PPE including safety boots, knee pads and safety glasses.	
		3	Lifting sampling equipment can cause muscle strain	Unload as close to work area as safely possible; use proper lifting and reaching techniques and body positioning; don't carry more than you can handle, and get help moving heavy or awkward objects.	
		4	Pressure can build up inside well causing cap to release under pressure	Keep head away from well cap when removing. If pressure relief valves are on well use prior to opening well	
3	Begin Purging Well and Collecting Parameter Measurements	1	Electrical shock can occur when connecting/disconnecting pump from the battery.	Make sure equipment is turned off when connecting/disconnecting. Wear leather gloves. Use GFCIs when using powered tools and pumps. Do not use in the rain or run electrical cords through wet areas.	

3	Begin Purging Well and Collecting Parameter Measurements	2	purge water can spill or leak from equipment	Stop purging activities immediately, stop leakage and block any drainage grate with sorbent pads. Call PM to notify them of any reportable spill.	
		3	Water spilling on the ground can cause muddy/slippery conditions	Be careful walking in work area when using plastic around well to protect from spillage	
		4	lacerations can occur when cutting materials such as plastic tubing	When cutting tubing, use tubing cutter. No open fixed blades should ever be used. When possible wear work gloves, leather type.	
		5	purge water can splash into eyes	Pour water slowly into buckets/drums to minimize splashing. Wear safety glasses	
4	Collect GW or Free Product Sample	1	Working with bailer rope can cause rope burns on hands.	Slowly raise and lower the rope or string for the bailer. Wear appropriate gloves for the task.	
		2	sample containers could break or leak preservative	Discard any broken sampleware or glass properly. Do not overtighten sample containers. Wear chemical protective gloves	
5	Recovery of Free Product from well	1	exposure to free product	Additional chemical protection may be necessary based on the type of product. Additionally, safety goggles, a faceshield, or respiratory protection may be required. Verify in the HASP.	
6	Staging of Well Purge water and/or Free Product	1	Muscle strains can occur when moving purge water or drums	If using buckets, do not fill buckets up to the top. Always keep lid on buckets when traveling or moving them to another location. Only half fill buckets so when dumping the buckets weigh less. See drum handling JLA for movement of drums.	Drum handling JLA

Personal Protective Equipment

Type	Personal Protective Equipment	Description	Required
Eye Protection	safety glasses		Required
Foot Protection	steel-toe boots		Required
Hand Protection	chemical resistant gloves (specify type)		Required
Hand Protection	work gloves (specify type)	leather	Required
Head Protection	hard hat		Required

Supplies

Type	Supply	Description	Required
Communication Devices	mobile phone		Required
Decontamination	Decon supplies (specify type)		Required
Miscellaneous	fire extinguisher		Required
Miscellaneous	first aid kit		Required
Personal	eye wash (specify type)	bottle	Required
Traffic Control	traffic cones		Required

Job Loss Analysis

General

Client Name	CHEVRON ENV MANAGEMENT
JSA ID	2226
Job Name	General Industry-Roadway work
Task Description	Working on a Private Roadway or Parking Area
Project Number	B00475170003
Project Name	FACILITY NO. 6518040 - 2010 RI/FFS WORK
PIC Name	VOGELEY, JOHN
Project Manager	MCCUNE, WILLIAM
Status Name	(3) Completed
Cretaion Date	4/20/2010 8:35:33 AM

User Roles

Role	Employee	Due Date	Completed	Approve	Supervisor	Active Employee
Created By	Beier, Kathryn	5/11/2010	4/20/2010		Messinger, John	True

Job Steps

Job Step	Job Step Description		Potential Hazard	Critical Action	HSP Reference
1	Deployment and removal of traffic control devices	1	Lifting hazards and awkward body positions from moving warning signs and control devices	Avoid excessive force pushing or pulling devices from vehicle; use the buddy system for heavier items; lift with legs and not back; avoid lifting and twisting motions.	ARCADIS H&S Handbook section III LL
		2	Struck by vehicle during placement	Wear high visibility clothing and Class II (minimum) traffic vest. Choose lime green color to avoid motorist confusion with traffic barrels. Always face oncoming traffic, use spotter if performing work that keeps focus off traffic. Ensure vehicle equipped with light bars and/or other warning devices and ensure they are activated, including vehicle flashers.	ARCADIS H&S Handbook section III LL
		3	Increased risk of injury (ergonomic from reacted moving or impact from increased vehicle exposure) from poor traffic control planning and implementation	Develop traffic control plan consistent with Manual of Uniform Traffic Control Devices, ensure lane closure tapers are computed properly, place devices in a manner that offers protection as other devices are deployed, place early warning devices first to warn drivers of pending work zone.	ARCADIS H&S Handbook section III LL
2	Flagger activities	1	Struck by vehicle while performing activity	Always face oncoming traffic, wear high visibility clothing described in step 1 above. Flaggers to be properly trained in proper flagging technique, if using paddles, ensure correct paddle warning displayed.	Certain states require flagger training: www.flagger.com
		2	Fatigue form standing in one position for extended periods of time.	Use job rotation when practical, shift weight form one leg to the other periodically, wear comfortable boots.	Certain states require flagger training: www.flagger.com

2	Flagger activities	3	Dehydration, heat stress (summer months), cold stress (winter months), sunburn, windburn	Ensure drinking water is in immediate vicinity of the flagger, check with flagger periodically to evaluate signs of heat or cold stress, avoid caffeine or sugary drinks during hot or cold weather, schedule work for worker to eat at regular intervals, wear sun block	Certain states require flagger training: www.flagger.com
		4	Struck by debris off roadway from passing vehicles	Be aware of hazard and be vigilant for debris, wear eye protection at all times.	Certain states require flagger training: www.flagger.com
3	Working in work zone	1	Struck by vehicle while performing work	Always stay behind protective barriers or channeling devices, never park vehicle that exposes workers to on coming traffic outside of barriers and channeling devices. Wear clothing and PPE described in step one above. Park vehicles within work zone to act as barriers to oncoming traffic when possible.	
		2	Struck by equipment in work area	Establish eye contact with all equipment operators when entering equipment operating radius, wear high visibility clothing and PPE as described in step one above. Park project vehicle away form active work are but still in work zone barriers or channeling devices.	
		3	Slips, trips and falls on wet or uneven surfaces in road right of way.	Wear proper footwear with good tread and ankle support. Plan route when walking on sloped surfaces, when walking along roadway stay as far off roadway as possible to avoid falling into traffic if tripping.	

Personal Protective Equipment

Type	Personal Protective Equipment	Description	Required
Eye Protection	safety glasses		Required
Foot Protection	steel-toe boots		Required
Hand Protection	work gloves (specify type)	leather	Required
Head Protection	hard hat		Required
Miscellaneous PPE	traffic vest--Class II or III		Required

Supplies

Type	Supply	Description	Required
Communication Devices	mobile phone		Required
Communication Devices	walkie talkie	if using flaggers	Required
Miscellaneous	fire extinguisher		Required
Miscellaneous	first aid kit		Required
Traffic Control	traffic cones		Required

1. POLICY

It is ARCADIS US policy to be proactive in the identification, assessment and control of health and safety hazards and associated risks. To those means, any work involving trenching and excavation that is under the control or direction of ARCADIS or an ARCADIS subcontractor will be accomplished following, at a minimum, this procedure.

It is ARCADIS' policy that ARCADIS staff will not enter excavations and trenches unless it is absolutely necessary. If there are no suitable alternatives and it becomes necessary to enter excavations or trenches, this procedure, at a minimum will be strictly followed.

It is also the policy of ARCADIS to ensure an OSHA-defined Excavation Competent Person is on-site for all excavation work under ARCADIS contractual control. The competent person will be provided by the entity on site responsible for performing the excavation work unless otherwise required by the client. Thus, if an ARCADIS subcontractor is conducting the excavation work, that subcontractor will provide the competent person. If ARCADIS is self-performing the excavation services, then ARCADIS will provide a competent person whether a specialized subcontractor or authorized employee.

2. PURPOSE AND SCOPE

2.1 Purpose

To effectively control or eliminate the hazards presented by working near or entry into excavations or trenches, this procedure sets forth the accepted practice for and establishes the requirements for workplace safety near excavations and trenches and employee and subcontractor entry into such.

2.2 Scope

This procedure along with associated checklists and the Utility Location procedure (ARC HSFS019) apply to all employees of ARCADIS-US. Only trained and authorized personnel are permitted to work near or enter excavations and trenches, perform rescue services, or act as the excavation competent person.

3. DEFINITIONS

Exhibit 1 includes relevant definitions to this procedure including that for competent person qualifications.

4. RESPONSIBILITIES

4.1 Corporate H&S with Division and Practice Experts

On an annual basis, review and update, as necessary, this procedure. In addition, review cancelled checklists periodically to ensure conformance to this procedure. Provide the excavation competent person and qualified person training and retraining, or recommend qualified training provider. Provide technical assistance regarding excavation and trench protocol, atmospheric testing equipment, PPE, hazard assessment and research information on unusual hazards. Audit project-specific excavation sites for compliance with this procedure.

4.2 Principal in Charge (PIC), Project Manager (PM), and Task Manager (TM)

PIC, PM and TMs are responsible to:

- Verify that all excavation and trench protocols are properly identified and addressed within the project work plan, project health & safety plan, and/or other project-related documents.
- Verify that their divisional or project team employees have received the proper training provided by Corporate Health & Safety or qualified training source prior to conducting excavation/trenching entry activities.
- Verify that any ARCADIS employee acting as the Excavation Competent person has been authorized and trained to do so as noted in Exhibit 1
- Verify that the proper entry equipment, including personal protective equipment (PPE), atmospheric testing equipment and safety equipment, is available for use by their divisional employees.
- Verify that copies of the completed checklists are available for Corporate Health and Safety review and retained with the project files

4.3 Health and Safety Plan Writers and Reviewers

Utilize this procedure as guidance to ensure the appropriate identification, assessment and control of excavation and trenching hazards for documentation in project HASPs

4.4 Entry/Work Supervisors (also see Training and Duties of Entry Supervisor)

- Work in direct coordination with and under the direction of the project excavation competent person
- Interface with the client representative to identify hazards associated with the client's excavation and trenching and/or work permit programs.
- Review existing soil sampling (if any) data or other pertinent hazard characterization information recorded by the client.
- Investigate the client's excavation/trenching protocol, to verify that any identified hazards and previous experience with earthwork at the site is properly communicated.
- Coordinate entry operations with the client's employees when both client and ARCADIS employees will be working in or near an excavation/trench.

- Coordinate necessary rescue assistance with either the client's in-house rescue team and/or the offsite rescue assistance specified by the client. The offsite rescue assistance specified by the client must have applicable rescue experience and be within a reasonable response distance.

- Verify that the client takes the necessary precautions in notifying their employees that our employees will be installing an excavation or trench.

- Review the lockout/tagout and isolation measures implemented by the client as necessary based on proximity of utilities or other energy sources in the area of the excavation/trench

- Immediately report any unusual or unforeseen excavation or trenching hazards to Corporate Health and Safety prior to authorizing entry

- Verify that all tests and precautionary measures identified on the Daily/Periodic Inspection Checklist located in Exhibit 1 and the ARCADIS Utility Location Policy and Procedure ARC HSFS019 has been performed prior to authorizing subsurface work or entry into an excavation or trench

- Offer all entrants an opportunity to review the applicable control measures and testing results and an opportunity to request a reevaluation as necessary

- Issue, authorize, and have the Utility Clearance and Daily/Periodic Inspection forms readily available for review

- Verify that copies of the completed clearance forms and checklists are properly disseminated to Corporate Health and Safety and retained with the project files, as specified in Section 8.0 – Records.

4.5 Entrants

- Qualified Employee Entrants must have training and instruction in their duties and responsibilities regarding the following:

- Recognize the hazards which may be faced during entry, as well as the signs and symptoms of exposure to the hazard(s).

- Maintain visual contact and/or verbal communications with the attendant at all times.

- Use the PPE, air monitoring and testing equipment that has been provided or have access to the information.

- Maintain an awareness of all required hazard controls and consult with the Competent Person as necessary

- Obey evacuation orders given by the Attendant, automatic alarm activation, or when self-perceived.

4.6 Competent Person

Meet all of the requirements specified for the Qualified Employee Entrants plus adequate training and experience for their duties and responsibilities to complete the following tasks:

- Anticipation, identification and control of excavation and trenching hazards, as well as the signs and symptoms of exposure to the hazard(s), and the Authority to implement all corrective actions including Stopping Work.
- Implement the ARCADIS Utility Clearance Policy and Procedure and complete the Daily/Periodic Excavation Inspection Checklist
- Verify adequate training and experience of all Entrants prior to entry

4.7 Attendants

- An attendant must be stationed outside the excavation and be available to monitor operations above and below ground. The attendant may have no other duties besides those listed in this section.
- All attendants must have training and instruction in their duties and responsibilities regarding excavation/trenching entry. The following are assigned duties.
- Maintain an accurate count of all entrants in the excavation
- Monitor activities both inside and outside the excavation/trench to verify the continued safety of entrants
- Maintain visual contact or verbal communication with all entrants
- Order evacuation of the excavation/trench if an uncontrolled hazard develops, either within or outside the space, or upon observing a behavioral effect of hazard exposure among entrants
- Keep unauthorized persons away from the excavation area
- Participate in non-entry rescue as appropriate
- Summon rescue and other emergency services
- Attendants must maintain current certification in basic first aid and cardiopulmonary resuscitation (CPR).

4.8 All ARCADIS Employees

Use the TRACK process described below regularly and frequently. In addition, employees read and understand all documented hazard identification and risk assessments conducted using the HARC process and documented in HASPs, JSAs, and other written plans that are associated with their work. ARCADIS employees will:

- Recognize the hazards of trenches and excavations
- Understand and follow the methods for working near trenches and excavations
- Notify the PIC, PM, TM or entry/work supervisor if they have not received appropriate training
- Participate in entry operations only if trained and authorized to do so
- Never enter an excavation/trench without completion of the required Utility Location Procedure, Daily/Periodic Inspection Checklist and have an authorized attendant
- Never attempt entry rescue within a excavation unless trained in entry rescue with appropriate equipment available
- If unexpected conditions arise during entry, immediately notify other entrants, evacuate the space and inform the designated Competent Person

5. PROCEDURE

5.1 General Safety Requirements for all Excavations

- All surface obstructions must be moved or supported so as to protect employees and equipment.
- Prior to excavation, all underground installations (water, electric, telephone, gas, etc.) must be located and documented in accordance with ARCADIS Utility Clearance Policy and Procedure ARC HSFS019.
- When excavating in areas near underground installations, proper precautions must be taken to determine the exact location of the installations and to adequately protect and support them. While an excavation is open, underground installations shall be protected, supported or removed as necessary to protect employees.
- Structural ramps that are used solely by employees as a means of access or egress from excavations shall be designed by a competent person.
- Structural ramps used for access or egress of equipment shall be designed by a competent person qualified in structural design, and shall be constructed in accordance with the design.
- Ladders used for access and egress from the excavation must extend at least 36” (3 feet) above the landing surface.

- If personnel are working in a location exposed to vehicular traffic they must be provided with and be required to wear reflective safety vests. Adequate, signs, barriers or other equivalent traffic controls must be used to protect employees.

- Personnel are not permitted to be beneath elevated loads handled by equipment or be in excavations when heavy equipment is digging in or near the excavation.

- Mobile equipment located near open excavations must be adequately protected from falling or rolling into excavations by the use of barricades or warning devices.

- All excavations over 4 feet in depth must be tested for hazardous atmospheres whenever personnel are required to enter and a potential exists for the existence of hazardous contaminants or oxygen deficiency. Excavations less than 4 feet in depth must be evaluated by the competent person and at the competent person's discretion be tested for hazardous atmospheres whenever personnel are required to enter and a potential exists for the existence of hazardous contaminants or oxygen deficiency.

- Means of rescue including a lifeline and body harness must be used by personnel entering excavations with a potential for air hazards. A standby person must be stationed outside the excavation to tend the lifeline(s).

- Water must not be allowed to accumulate in open excavations where employees are working. When necessary, means such as diverting natural drainage around the excavation or actively pumping water must be used to prevent or control water accumulation.

- All structures adjacent to an open excavation must be supported, or a registered professional engineer (PE) must determine that the structure will not be affected by the excavation activities.

- Excavated materials (spoil) must be placed no closer than 2 feet from the edge of an open excavation, and otherwise retained to prevent loose material from falling into the excavation.

- Protection such as guardrails, barricades or covers must be in place to protect personnel from possible falls into open excavations, pits, wells and shafts.

- Work tasks will be designed to limit the number of personnel required to enter any excavation. All tasks that can be completed remotely from outside the excavation (such as soil sampling) will be conducted in such a manner.

- Personnel will not be allowed to enter any excavation unless adequate protective systems and procedures are utilized to prevent accidents and injury.

- All excavations over four feet in depth shall be provided with a stairway, ladder, ramp, or other safe means of egress so as to require no more than 25 feet of lateral travel. As deemed necessary by the competent person, excavations less than 4 feet in depth will be provided with a stairway, ladder, ramp, or other safe means of egress so as to require no more than 25 feet of lateral travel.

5.2 Excavations Requiring Protective Systems

This section defines excavations that require protective systems.

- All excavations into which employees will enter, regardless of depth, where the potential for cave-in exists.

- Any excavation over 4 feet in depth into which employees will enter that is not entirely in stable rock as defined in this procedure.

- Any excavation near a structure, (e.g. foundations, piers, footers, walls, sidewalks, tanks, roadways, etc.), as required by the registered professional engineer reviewing the stability of the excavation and the structure.

- All excavations over 20 feet in depth must be designed by a registered professional engineer regardless of whether personnel will enter it or not.

- All excavations with adjacent structures which are located a distance less than 6 times the depth of the excavation away shall be reviewed by a registered professional engineer to determine if the stability of the structure will be affected by the excavation.

- Support systems for an adjacent structure must be designed by a registered professional engineer.

5.3 Selection and Use of Protective Systems

5.3.1 Shoring or Shielding

- If shoring or shielding is selected as the protective system for an excavation, soil classification in accordance with 1926 Subpart P Appendix A (see Section 9 of this procedure) is required.

- One of the following options must be utilized for all excavations which will be shored or shielded.

    - Timber shoring as specified in 1926 Subpart P Appendix C must be utilized

    - Hydraulic shoring, trench jacks, air shores, or shields as required in 1926.652 (c)(2) must be utilized following the system manufacturer’s data

- A system which follows other tabulated data (approved by a registered professional engineer) must be utilized

- The excavation must be designed by a registered professional engineer

5.3.2 Sloping

- If sloping is selected as the protective system for an excavation, the excavation sides must be sloped at a maximum of 34 degrees (1.5 Horizontal: 1 Vertical), unless the procedure listed above is followed.

- Soil classification in accordance with Section 10 of this procedure) is required for all excavations with sides which will be sloped greater than 34^{o} (1.5 Horizontal: 1 Vertical). If it will be sloped greater than 34^{o}, the one of the following options must be utilized:

    - Option 1 - assume Type C and slope 1.5/1 - probably the most common and preferred method for us

    - Option 2 - classify soil according to the standard and use Type A/B sloping requirements

    - Option 3 – use other tabulated data with PE approval

    - Option 4 – PE approval of sloping/benching design

5.4 Atmospheric Testing for Entry

Any excavation over 4 feet in depth with a potential for hazardous contaminants or oxygen deficiency must be tested for hazardous atmospheres prior to and during activities involving entry. After atmospheric testing, if the area is found to be oxygen deficient or a hazardous atmosphere exists or could exist a confined space permit must be obtained if the area will be entered.

The site designated "competent person" will document initial and periodic air monitoring results for all activities requiring entry into the excavation. All atmospheric testing of excavations must be conducted in the following sequence and meet the following air quality criteria.

- Oxygen content must be 19.5 to 23.5%

- Combustible gas or vapor must not exceed 10% of its lower explosive limit (LEL)

- Toxic air contaminant levels must not exceed 50% of the PEL or TLV for the specific contaminant whichever is lower

- Carbon monoxide must not exceed 10 ppm for a 5 minute average or ceiling value of 25 ppm

- Hydrogen sulfide must not exceed 0.5 ppm

5.5 Location of Underground/Overhead Utilities

- The competent person and the project manager shall both verify that local underground facilities location/protection agencies are notified within the required time frame prior to the initiation of excavation activities and meet all requirements in the ARCADIS Utility Location Policy and Procedure ARC HSFS019.

- Prior to initiation of excavation or trenching operations the competent person shall verify that all utilities have been located.

5.6 Daily/Periodic Inspections

- Prior to initiation of daily excavation or trenching operations the competent person shall complete a daily inspection of the excavation.

- During excavation or trenching operations the competent person shall complete a periodic inspection after any event (e.g., thunderstorm, vibration, excessive drying) that may affect excavation stability.

- The competent person shall complete the daily/periodic inspection checklist (A copy of the checklist is attached to this Policy as Exhibit A– Subcontractors must complete an equivalent inspection form) is completed for each inspection of excavation and trenching activities.

5.7 Soil Classification for Selection of Protective Systems

5.7.1 Soil Classification

This section describes a method of classifying soil and rock deposits based on site and environmental conditions, and on the structure and composition of the earth deposits. This section contains definitions, sets forth requirements, and describes acceptable visual and manual tests for use in classifying soils.

This section applies when a sloping, benching or shoring system is utilized as a method of protection for employees from cave-ins.

5.7.2 Soil Classification Definitions

5.7.2.1 Types/Classes of Soil

Type/Class A Soils are cohesive soils with an unconfined, compressive strength of 1.5 ton per square foot (tsf) (144kPa) or greater. Examples of cohesive soils are: Clay, silty clay, sandy clay, clay loam and in some cases, silty clay loam and sandy clay loam. Cemented soils such as caliche and hardpan are also considered Type A. However, no soil is Type A if the following apply.

- The soil is fissured
- The soil is subject to vibration from heavy traffic, pile driving, or similar effects
- The soil has been previously disturbed
- The soil is part of a sloped, layered system where the layers dip into the excavation on a slope of four horizontal to one vertical (4 Horizontal:1 Vertical) or greater
- The material is subject to other factors that would require it to be classified as a less stable material

5.7.2.1.1 Type Class B Soils

- Cohesive soils with an unconfined compressive strength greater than 0.5 tsf (48 kPa) but less than 1.5 tsf (144 kPa)
- Granular cohesionless soils including angular gravel (similar to crushed rock), silt, silt loam, sandy loam and, in some cases, silty clay loam and sandy clay loam
- Previously disturbed soils except those which would otherwise be classed as Type C soil
- Soil that meets the unconfined compressive strength or cementation requirements for Type A, but is fissured or subject to vibration
- Dry rock that is not stable
- Material that is part of a sloped, layered system where the layers dip into the excavation on a slope less steep than four horizontal to one vertical (4 Horizontal:1 Vertical), but only if the material would otherwise be classified as Type B

5.7.2.1.2 Type/Class C Soils

- Cohesive soil with an unconfined compressive strength of 0.5 tsf (48 kPa) or less
- Granular soils including gravel, sand, and loamy sand
- Submerged soil or soil from which water is freely seeping

- Submerged rock that is not stable
- Material in a sloped, layered system where the layers dip into the excavation or a slope of four horizontal to one vertical (4 Horizontal:1 Vertical) or steeper

5.7.2.2 Methods for Classifying Soils

Each soil and rock deposit shall be classified by a competent person as Stable Rock, Type A, Type B, or Type C in accordance with the definitions set forth in this section. The classification of the deposits shall be made based on the results of at least one visual and at least one manual analysis conducted by a competent person using tests described below, or in other recognized methods of soil classification and testing such as those adopted by the American Society for Testing Materials, or the U.S. Department of Agriculture textural classification system.

The visual and manual analyses, such as those noted as being acceptable in this section, shall be designed and conducted to provide sufficient quantitative and qualitative information as may be necessary to identify properly the properties, factors, and conditions affecting the classification of the deposits. Visual analysis is conducted to determine qualitative information regarding the excavation site in general, the soil adjacent to the excavation, the soil forming the sides of the open excavation, and the soil taken as samples from excavated material.

Observe the following:

- Samples of soil that are excavated and soil in the sides of the excavation. Estimate the range of particle sizes and the relative amounts of the particle sizes. Soil that is primarily composed of fine grained material is cohesive material. Soil composed primarily of coarse grained sand or gravel is granular material.
- Soil as it is excavated. Soil that remains in clumps when excavated is cohesive. Soil that breaks up easily and does not stay in clumps is granular.
- The side of the open excavation and the surface area adjacent to the excavation. Crack like openings such as tension cracks could indicate fissured material. If chunks of soil spall off a vertical side, the soil could be fissured. Small spalls are evidence of moving ground and are indications of potentially hazardous situations.
- The area adjacent to the excavation and the excavation itself for evidence of existing utility and other underground structures, and to identify previously disturbed soil.

- The open side of the excavation to identify layered systems. Examine layered systems to identify if the layers slope toward the excavation. Estimate the degree of slope of the layers.

- The area adjacent to the excavation and the sides of the opened excavation for evidence of surface water, water seeping from the sides of the excavation, or the location of the level of the water table.

- The area adjacent to the excavation and the area within the excavation for sources of vibration that may affect the stability of the excavation face.

Manual analysis of soil samples is conducted to determine quantitative as well as qualitative properties of soil and to provide more information in order to classify soil properly.

5.7.2.3 Classifications

A. Plasticity. Mold a moist or wet sample of soil into a ball and attempt to roll it into threads as thin as 1/8 inch in diameter. Cohesive material can be successfully rolled into threads without crumbling. For example, if at least a two inch (50 mm) length of 1/8 inch thread can be held on one end without tearing, the soil is cohesive.

B. Dry strength. If the soil is dry and crumbles on its own or with moderate pressure into individual grains or fine powder, it is granular (any combination of gravel, sand, or silt). If the soil is dry and falls into clumps which break up into smaller clumps, but the smaller clumps can only be broken up with difficulty, it may be clay in any combination with gravel, sand or silt. If the dry soil breaks into clumps which do not break up into small clumps and which can only be broken with difficulty, and there is no visual indication the soil is fissured, the soil may be considered unfissured.

C. Thumb penetration. The thumb penetration test can be used to estimate the unconfined compressive strength of cohesive soils. Type A soils with an unconfined compressive strength of 1.5 tsf can be readily indented by the thumb; however, they can be penetrated by the thumb only with very great effort. Type C soils with an unconfined compressive strength of 0.5 tsf can be easily penetrated several inches by the thumb, and can be molded by light finger pressure. This test should be conducted on an undisturbed soil sample, such as a large clump of spoil, as soon as practicable after excavation to keep to a minimum the effects of exposure to drying influences. If the excavation is later exposed to wetting influences (rain, flooding), the classification of the soil must be changed accordingly.

D. Other strength tests. Estimates of unconfined compressive strength of soils can also be obtained by use of a pocket penetrometer or by using a hand operated shearvane.

E. Drying test. The basic purpose of the drying test is to differentiate between cohesive material with fissures, unfissured cohesive material, and granular material. The procedure for the drying test involves drying a sample of soil that is approximately one inch thick (2.54 cm) and six inches (15.24 cm) in diameter until it is thoroughly dry:

1. If the sample develops cracks as it dries, significant fissures are indicated.

2. Samples that dry without cracking are to be broken by hand. If considerable force is necessary to break a sample, the soil has significant cohesive material content. The soil can be classified as an unfissured cohesive material and the unconfined compressive strength should be determined by using the thumb penetration or other test.

5.7.2.4 If a sample breaks easily by hand, it is either a fissured cohesive material or a granular material. To distinguish between the two, pulverize the dried clumps of the sample by hand or by stepping on them. If the clumps do not pulverize easily, the material is cohesive with fissures. If they pulverize easily into very small fragments, the material is granular.

5.7.2.5 Layered system

A layered system shall be classified in accordance with its weakest layer. Each layer may be classified individually where a more stable layer lies under a less stable layer.

5.7.2.6 Reclassifying Soils

A layered system shall be classified in accordance with its weakest layer. Each layer may be classified individually where a more stable layer lies under a less stable layer.

In most instances the ARCADIS designated Excavation/Trenching Competent person will assume Type C soil, unless they have conclusive data to validate Type A or B.

5.7.2.7 Excavation Construction Based on Soil Type

The Maximum allowable slope means the steepest incline of an excavation face that is acceptable for the most favorable site conditions as protection against cave-ins, and is expressed as the ratio of horizontal distance to vertical rise (H:V). Short-term exposure means a period of time less than or equal to 24 hours that an excavation is open. Soil and rock deposits must be classified in accordance with Appendix A to Subpart P of Part 1926. The maximum allowable slope for a soil or rock deposit must be determined from the table provided below. The actual slope must not be steeper than the maximum allowable slope. The actual slope must be less steep than the maximum allowable slope, when there are signs of distress. If that situation occurs, the slope must be cut back to an actual slope which is at least horizontal to one vertical (1/2H:1V) less steep than the maximum allowable slope. When surcharge loads from stored material or equipment, operating equipment, or traffic are present, a competent person must determine the degree to which the actual slope must be reduced below the maximum allowable slope, and must assure that such reduction is achieved. Surcharge loads from adjacent structures must be evaluated in accordance with 1926.651(I). Configurations of sloping and benching systems must be in accordance with 29 CFR 1926 Subpart P, Appendix B.

EXCAVATION SLOPE TABLE 2
29 CFR 1926 SUBPART P APPENDIX B
MAXIMUM ALLOWABLE SLOPES

Soil or Rock Type	Maximum Allowable Slopes (H:V)[1] for Excavations Less Than 20 Feet Deep[2]
Stable Rock	Vertical (90 degrees)
Type A[3]	¾:1 (53 degrees)
Type B	1:1 (45 degrees)
Type C	1:½ (34 degrees)

1. Numbers shown in parentheses next to maximum allowable slopes are angles expressed in degrees from the horizontal. Angles have been rounded off.
2. Sloping or benching for excavations greater than 20 feet deep must be designed by a registered professional engineer.
3. A short-term maximum allowable slope of 1/2H:1V (63 degrees) is allowed in excavations in Type A soil that are 12 feet (3.67 m) or less in depth. Short-term maximum allowable slopes for excavations greater than 12 feet (3.67 m) in depth must be 3/4H:1V (53 degrees).

6. TRAINING

6.1 Project - Specific Training

All staff working on a site where trenching and excavation activities are being conducted by ARCADIS or its subcontractors will be provided with site orientation on excavation projects shall include a discussion of the following:

- Site excavation hazards and procedures
- Requirements for conducting activities remotely whenever possible
- Client requirements and procedures for excavation activities

- This Procedure

Daily Safety Meetings on projects involving excavation activities shall include a discussion of:

- Site excavation hazards and procedures
- Requirements for conducting activities remotely whenever possible
- Client requirements and procedures for excavation activities
- This Excavation and Trenching Procedure, as appropriate

6.2 Additional Training

Besides site orientation training, additional training will be provided as follows based on the employee's activities:

- All employees who work in the area of potential excavation/trenching sites will receive awareness level training as provided and/or approved by ARCADIS Corporate H&S in order to recognize and to understand the hazards.
- Entrants, Attendants, and Entrant Supervisors will receive additional training as approved by Corporate H&S. This training will be classroom in nature and cover the details of trenching and excavation hazards and controls
- Qualified Competent Persons will be provided training as follows:

  In order to be assigned duties as a competent person with respect to excavation and trenching, in addition to the criteria noted in Exhibit 1, personnel must complete an ARCADIS approved training course or an equivalent course approved by Corporate Health and Safety including but not limited to the following topics:

  - Introduction to trenches and excavations
    - Definition of trenches and excavations
    - General requirements of OSHA 29 CFR 1926 Subpart P
  - Responsibilities and requirements of a competent person
    - Necessary authority
    - When other/outside resources may be necessary
  - Hazard Identification and Assessment
    - Cave-In Hazards including nearby structures
    - Underground utilities

  - Confined Space
  - Hazardous atmospheres
  - Water accumulation
  - Vehicular traffic and falling loads
- Hazard controls
  - Soil analysis and testing (visual and manual
  - Protective systems
    - Shoring
    - Sloping
    - Shielding
    - Benching
  - Personal protective equipment
  - Utility location
  - Atmospheric testing
  - Water drainage and pumping
  - Site housekeeping and management
    - Spoils
    - Traffic control
    - Overhead hazard protection
  - Communications
    - Verbal
    - Signaling
  - Access and egress
- Emergency Procedures
  - Warning signs of cave-in
  - Evacuation procedures

    - Rescue

- Inspections
    - Checklists
    - Potential deficiencies

All training provided must be reviewed and approved by Corporate Health & Safety and will be managed through ARCHIMEDES.

Documentation of training certification received by attendance at any training course including externally provided training courses will be kept by the employee with copies provided to ARCHIMEDES.

7. REFERENCES

7.1 ARCADIS Health and Safety Procedure ARC HSFS010– Health and Safety Planning

7.2 ARCADIS Health and Safety Procedure ARC HSFS004 – Control of Hazardous Energy (Lockout/Tagout)

7.3 ARCADIS Utility Clearance Policy and Procedure ARC HSF019

7.4 ARCADIS Confined Space Policy and Procedure ARC HSF003

7.5 OSHA 29 CFR Part 1926 Subpart P - Excavations

8. RECORDS

8.1 Training records will be kept by the individual employee with copies of such certificates kept by ARCHIMEDES. Training dates and times will be kept by ARCHIMEDES.

8.2 Completed clearance forms and checklists will be kept in the project files with copies available for Corporate H&S review.

8.3 Copies of all HASPs that document excavation trenching procedures will be kept in the project files.

9. APPROVALS AND HISTORY OF CHANGE

Approved By: Michael Thomas, CIH, CPEA

Michael A Thomas

History of Change

Revision Date	Revision Number	Reason for change
12 May 2008	01	Original document
13 June 2008	02	Modified Section 5.1 – 4th bullet related to structural ramps. Modified Section 5.2 to designate a 6x factor for structural integrity of structures near the excavation. Revised Exhibit 1 to modify the definition of a Competent person
9 January 2009	03	Cleaned up definitions, deleted training requirements from Section 5.0 and moved them to Section 6.0, modified purpose statement

Exhibit 1 – Definitions

Attendant is a trained qualified individual stationed outside the excavation whose duty is to monitor authorized entrants inside the excavation or trench and have a means of communication with the designated rescue services.

Benching/Benching system means a method of protecting employees from cave-ins by excavating the sides of an excavation to form one or a series of horizontal levels or steps, usually with vertical or near-vertical surfaces between levels.

Cave-in means the separation of a mass of soil or rock material from the side of an excavation, or the loss of soil from under a trench shield or support system, and its sudden movement into the excavation, either by falling or sliding, in sufficient quantity so that it could entrap, bury or otherwise injure and immobilize a person.

Competent person means one who, through education, training, and/or experience, is capable of identifying existing and predictable hazards or working conditions which are unsanitary, hazardous, or dangerous to employees and who has authorization to take prompt corrective measures to eliminate them. All ARCADIS employee's, must meet the following minimum requirements to be considered a Competent Person:

- Be nominated to the appropriate Division H&S Director by their supervisor or project manager to be considered as a competent person. The nomination will include the submittal of various documentation that describes why the person should be nominated and to provide evidence that they have met the criteria listed below.
- Be jointly approved by the appropriate Division H&S Director and the appropriate Practice/Client H&S Manager or resource.
- Attend ARCADIS Competent Person training or an equivalent course approved by Corporate Health and Safety
- Have a minimum of 1 year of supervised field experience and approval from their supervisor to fill the role of competent person
- If on an Environmental project where HAZWOPER training is required by ARCADIS, completed a 40 Hour HAZWOPER and HAZWOPER Supervisor training course and be current on their annual 8 Hour refresher
- Attended a 10 or 30 Hour OSHA Construction Safety Course or have equivalent training to that provided by the 10 or 30 hour course
- If a hazardous atmosphere is present, or there is limited entry or exit and the excavation or trench must be entered as a confined space, the person must also be Confined Space trained and authorized as per the ARCADIS Confined Space procedure ARC HSFS003

Excavation means any man-made cut, cavity, trench, or depression in an earth surface formed by earth removal into which a person can bodily enter. **Entry** constitutes the act by which an employee proceeds into an excavation or trench. Consideration of hazards, especially cave-ins and fall protection must still be considered and accounted for when equipment or personnel are near an excavation or trench, even if personnel will not be entering.

Entrants are employee's who are trained and authorized to enter a trench or excavation. Entrants must have attended a Qualified Excavation Training course offered or approved by Corporate Health and Safety.

Failure means the breakage, displacement, or permanent deformation of a structural member or connection so as to reduce its structural integrity and its supportive capabilities.

Hazardous Atmosphere is an atmosphere which exposes employees to a risk of death, incapacitation, injury, or acute illness from one or more of the following:

- An atmospheric concentration of any substance in excess of 50% of its established permissible exposure limit (PEL); or its assigned threshold limit value (TLV) or other value listed on the Material Safety Data Sheet (MSDS) for the chemical constituent, whichever is lower.
- A flammable gas, vapor, or mist in excess of 10% of its lower explosive limit (LEL).
- An airborne combustible dust at a concentration that obscures vision at a distance of 5 feet or less.
- An atmospheric oxygen concentration below 19.5% (oxygen-deficient atmosphere) or above 23.5% (oxygen-enriched atmosphere).
- An atmosphere which is immediately dangerous to life and health.

Immediately Danger to Life and Health (IDLH) means any condition which poses an immediate threat to loss of life; may result in irreversible or immediate-severe health effects; may result in eye damage, irritation, or other conditions which could impair escape from the space.

Protective system means a method of protecting employees from cave-ins, from material that could fall or roll from an excavation face or into an excavation, or from the collapse of adjacent structures. Protective systems include support systems, sloping and benching systems, shield systems and other systems that provide protection.

Ramp means an inclined walking or working surface that is used to gain access to one point from another, and is constructed from earth or from structural materials such as steel or wood.

Registered Professional Engineer means a person who is registered as a professional engineer in the state where the work is to be performed. However, a professional engineer, registered in any state is deemed to be a "registered professional engineer" within the meaning of this standard when approving designs for "manufactured protective systems" or "tabulated data" to be used in interstate commerce. To oversee an excavation/trench activity the PE must have experience with and expertise in excavation, soil and stability considerations.

Sheeting means the members of a shoring system that retain the earth in position and in turn are supported by other members of the shoring system.

Shield (Shield system) means a structure that is able to withstand the forces imposed on it by a cave-in and thereby protect employees within the structure. Shields can be permanent structures or can be designed to be portable and moved along as work progresses. Additionally, shield can be either pre-manufactured or job-built in accordance with 1926.652 (c)(3) or (c)(4). Shields used in trenches are usually referred to as "trench boxes" or "trench shields".

Shoring (Shoring system) means a structure such as a metal hydraulic, mechanical or timber shoring system that supports the sides of an excavation and which is designed to prevent cave-ins.

Sloping (Sloping system) means a method of protecting employees from cave-ins by excavating to form sides of an excavation that are inclined away from the excavation so as to prevent cave-ins. The angle of incline required to prevent a cave-in varies with differences in such factors as the soil type, environmental conditions of exposure, and application of surcharge loads.

Stable rock means natural solid mineral material that can be excavated with vertical sides and will remain intact while exposed. Unstable rock is considered to be stable when the rock material on the side or sides of the excavation is secured against caving-in or movement by rock bolts or by another protective system that has been designed by a registered professional engineer.

Support system means a structure such as underpinning, bracing, or shoring, which provides support to an adjacent structure, underground installation, or the sides of an excavation.

Trench means a narrow excavation (in relation to its length) made below the surface of the ground to which a person can bodily enter. In general, the depth is greater than the width, but the width of a trench (measured at the bottom) is not greater than 15 feet (4.6 meters). If forms or other structures are installed or constructed in an excavation so as to reduce the dimension measured from the forms or structure to the side of the excavation to 15 feet (4.6 meters) or less (measured at the bottom of the excavation), the excavation is considered to be a trench.

Cemented soil means a soil in which the particles are held together by a chemical agent, such as calcium carbonate, such that a hand size sample cannot be crushed into powder or individual soil particles by finger pressure.

Cohesive soil means clay (fine grained soil), or soil with a high clay content, which has cohesive strength. Cohesive soil does not crumble, can be excavated with vertical sides, and is plastic when moist. Cohesive soil is hard to break up when dry, and exhibits significant cohesion when submerged. Cohesive soils include clayey silt, sandy clay, silty clay, clay and organic clay.

Dry soil means soil that does not exhibit visible signs of moisture content.

Fissured means a soil material that has a tendency to break along definite planes of fracture with little resistance, or a material that exhibits open cracks, such as tension cracks, in an exposed surface.

Granular soil means gravel, sand, or silt (coarse grained soil) with little or no clay content. Granular soil has no cohesive strength. Some moist granular soils exhibit apparent cohesion. Granular soil cannot be molded when moist and crumbles easily when dry.

Layered system means two or more distinctly different soil or rock types arranged in layers. Micaceous seams or weakened planes in rock or shale are considered layered.

Moist soil means a condition in which a soil looks and feels damp. Moist cohesive soil can easily be shaped into a ball and rolled into small diameter threads before crumbling. Moist granular soil that contains some cohesive material will exhibit signs of cohesion between particles.

Plastic means a property of a soil which allows the soil to be deformed or molded without cracking, or appreciable volume change.

Saturated soil means a soil in which the voids are filled with water. Saturation does not require flow. Saturation, or near saturation, is necessary for the proper use of instruments such as a pocket penetrometer or sheer vane.

Soil classification system means, for the purpose of this procedure, a method of categorizing soil and rock deposits in a hierarchy of Stable Rock, Type A, Type B and Type C, in decreasing order of stability. The categories are determined based on an analysis of the properties and performance characteristics of the deposits and the characteristics of the deposits and the environmental conditions of exposure.

Submerged soil means soil which is underwater or is free seeping.

Unconfined compressive strength means the load per unit area at which a soil will fail in compression. It can be determined by laboratory testing, or estimated in the field using a pocket penetrometer, by thumb penetration tests, and other methods.

Wet soil means soil that contains significantly more moisture than moist soil, but in such a range of values that cohesive material will slump or begin to flow when vibrated. Granular material that would exhibit cohesive properties when moist will lose those cohesive properties when wet.

Exhibit 2 – Daily / Periodic Excavation Inspection Checklist

ARCADIS

Daily / Periodic Excavation Inspection Checklist

Project Name:	**Date / Time:**
Project Number:	**Location:**
Prepared By:	**Project Manager:**

This checklist must be completed for all excavations. It documents that daily and post-event / periodic inspections are conducted.

Soil Classified As: Stable Rock Type A Type B Type C

Soil Classified On: By:

Type of Protective System in Use: Sloping Shoring Other _______________

Description:

Inspection Item	YES	NO	Comments
Has the ARCADIS Utility Clearance Procedure been completed?			
Are underground installations protected from damage?			
Are adequate means of entry / exit available in the excavation – at least every 25 feet?			
If exposed to traffic, are personnel wearing reflective vests and adequate barriers/traffic controls installed?			
Do barriers exist to prevent equipment from rolling into the excavation?			
Was air monitoring conducted prior to and during excavation entry?			
Was the stability of adjacent structures reviewed by a registered P.E.?			
Are spoil piles at least 2 feet from the excavation edge?			
Is fall protection in use near excavations deeper than 6 feet?			
Are work tasks completed remotely if feasible?			
Is a protective system in place and in good repair?			
Is emergency rescue (lifeline / body harness) equipment used due to potential atmospheric hazard?			
Is excavation exposed to vibration?			
Are employees protected from falling / elevated material?			
Is soil classification adequate for current environmental / weather conditions?			
Do portable ladders extend at least 4 feet above the excavation?			
Are portable ladders or ramps secured in place?			
Have all personnel attended safety meeting on excavation hazards?			
Are support systems for adjacent structures in place?			
Is the excavation free from standing water?			
Is water control and diversion of surface runoff adequate?			
Are employees wearing required protective equipment?			

ARCADIS Excavation Competent Person:	**Date/Time:**

ARCADIS Infrastructure, environment, buildings	ARCADIS HS Procedure Name Personal Protective Equipment	Revision Number 01
Implementation Date 20 February 2009	ARCADIS HS Procedure No. ARC HSGE015	Revision Date 20 February 2009
Author Miriam Koesterich	Page 1 of 12	Approver Mike Thomas

1. POLICY

It is the policy of ARCADIS to assess the workplace to identify and assess hazards in order to appropriately implement controls for those hazards. In addition, it is ARCADIS policy to supply personal protective equipment (PPE) for employees in a working environment where engineering and administrative controls are not feasible or effective in the control of hazards. ARCADIS will train and supply this PPE at no cost to the employee.

2. PURPOSE AND SCOPE

2.1 Purpose

The purpose of PPE is to shield or isolate individuals from the chemical, physical, and biologic hazards that may be encountered in their work environment. A hazard analysis or assessment will be performed before a job task is begun to evaluate the if PPE is necessary to protect an employee from identified hazards and determine the type of PPE required. This analysis will include the identification of hazards/suspected hazards and their routes of exposure.

Combinations of protection may be needed to provide the appropriate level of protection for any given work environment. The level of PPE may change during a job, so periodic evaluation will be done to ensure that the most appropriate PPE is being used. Over-protection, as well as under-protection, can be hazardous and should be avoided where possible.

Subcontractors and other non-ARCADIS employees must supply their own PPE. ARCADIS will not supply PPE to any non-ARCADIS employees unless specific arrangements and agreements are made with the other party.

This Health and Safety Procedure (HSP) provides guidance on the proper selection, use, care and maintenance of PPE.

2.2 Scope

Whenever possible, engineering, substitution, and administrative controls will be used to reduce or eliminate hazards, but when they are not feasible, practical or adequate, PPE will be used to protect employees from exposure to hazards during ARCADIS-related work tasks.

3. DEFINITIONS

Eye/Face Protection - Equipment designed to provide eye or face protection when exposed to hazards from flying particles, molten metal, liquid chemicals, acids or caustic liquids, chemical gases or vapors, or potentially injurious light radiation.

Foot Protection - Footwear designed to provide foot and toe protection when working in areas where there is a danger of foot injuries due to falling or rolling objects, or objects piercing the sole, and/or where an employee's feet are exposed to electrical hazards. These include such measures as steel toe, metatarcal, and boot warmers

Hand and Body Protection - Equipment designed to provide protection to the hands and body during exposures to potential hazards such as potential for skin absorption of harmful substances, sharp objects, abrasive surfaces, punctures, temperature extremes and chemical contact.

Hazard Assessment - The process utilized to identify hazards in the workplace and to select the appropriate PPE to guard people against potential hazards (see attachment Hazard Assessment for Personal Protective Equipment (PPE)).

Head Protection - Equipment designed to provide protection to the head during exposure to potential hazards such as falling objects, striking against objects, or electrical hazards.

Hearing Protection - Equipment designed to provide protection to an individual's hearing during exposure to excessive noise levels and any 8hr work day with noise levels consistently 85dB or above.

Personal Protective Equipment (PPE) - Equipment designed to provide protection to the wearer from potential hazards to the eyes, face, hands, head, feet, ears, extremities, and respiratory system.

Respiratory Protection - Equipment designed to provide protection to the wearer from potential inhalation hazards such as vapors, mists, particulates, and gases.

4. RESPONSIBILITIES

4.1 ARCADIS Management

Is responsible for providing resources for the acquisition of PPE and for the conduct of hazard assessments.

4.2 Project Managers

Project Managers are responsible, as part of the project hazard assessment, for determining PPE necessary to complete the project. In addition, the project manager is responsible for determining client requirements with respect to PPE. Project Managers notify health and safety staff of biological, chemical, and physical hazards present or potentially present on the site.. Project Managers are also responsible for ensuring that project staff has the appropriate and applicable training for PPE use prior to those staff beginning work.

4.2 Corporate Health and Safety

Corporate Health and Safety is responsible for keeping this policy and procedure up-to-datewith current regulatory requirements and best practices and for assisting in determining the appropriate PPE for a particular task and work environment and for assisting in the identification of appropriate vendors of such PPE.

4.3 Health and Safety Staff

Project Health and Safety Staff including designated Writers and Reviewers of Project Health and Safety Plans (HASPs) are responsible for developing control processes andtechniques on specific projects based on the physical, chemical, and biological hazards expected to be encountered on project facilities.

4.4 ARCADIS Staff

ARCADIS staff are responsible for completing PPE training as required by this policy and procedure, and for following all hazard control processes designated by the Project Manager, Project Health and Safety Staff, and the project HASP. If project personnel believe that a hazard is present that was not previously identified or is at levels that are higher than expected, they should stop work and notify project health and safety staff or the project manager immediately and not proceed until authorized.

5. PROCEDURE

5.1 Minimum PPE Requirements

All staff who regularly conduct field work or visit project sites outside of office environments will be issued a field bag that contains, at a minimum, the following PPE:

- An ARCADIS logo’d hardhat
- Two pair of safety glasses, one clear pair and one tinted pair, or one pair of prescription safety glasses with transitional lenses
- Hearing protection
- A minimum, Type 2 reflective vest in either orange, lime green or yellow
- Steel toe safety boots

Office locations will stock extra bags with the equipment listed above for use by other staff who do not regularly go to field locations. Additional PPE and H&S equipment will be issued to staff based on the hazards they face on specific projects (i.e. respirators, goggles, chaps, etc.).

No ARCADIS staff should arrive at a field or project site without this minimum PPE.

5.2 The PPE Program

The basic objectives of a PPE program are to protect the wearer from safety and health hazards; and to prevent injury to the wearer from incorrect use and/or malfunction of the PPE. This document serves as the overall ARCADIS PPE program and is used as guidance for the development of a project-specific PPE program which becomes part of a project-specific health and safety plan. A project-specific PPE program in combination with this HSP will address the following:

- PPE selection based upon site hazards (Hazard Identification/Assessment).

  - Identify the hazards/suspected hazards and their potential routes of exposure (e.g., skin, inhalation, ingestion or eye contact).

- The use and limitations of the equipment including limitations during temperature extremes and under certain medical conditions;
- The work mission duration;
- Maintenance, storage, decontamination and disposal of PPE;
- Training including proper fit and how to properly put on and take off PPE;
- PPE inspection procedures prior to, during, and after use; and
- Periodic evaluation of the effectiveness of the PPE program.

5.3 PPE Selection

The use of PPE can itself create significant worker hazards, such as heat stress, physical and psychological stress and impaired vision, mobility and communication. Over-protection, as well as under-protection, can be hazardous and should be avoided where possible. Site or project-specific health and safety plans take into consideration engineering, substitution, and administrative controls first as a means to eliminate/reduce the need for PPE. When it is not feasible or practical to eliminate the use of PPE, PPE selection will be based on an evaluation of the performance characteristics of the PPE relative to the following:

- The requirements and limitations of the tasks or work environment;
- The task-specific conditions and duration; and
- The hazards and potential hazards identified at the site.

The level of protection will be increased whenever it is shown that increased protection is necessary to reduce employee exposures to the hazards. It may be decreased when it is shown that this will not result in hazardous exposure to employees.

5.4 Levels of PPE Protection

For work on hazardous sites, a combination of PPE may be categorized into levels A, B, C, or D with level A offering the highest level of protection and D the lowest. Monitoring the effectiveness of PPE will be done throughout a project to ensure that the appropriate level of protection is being worn. These levels of protection are described below.

Level A Protection

Level A PPE offers the highest level of respiratory and skin protection and should be worn when:

- The hazardous substance has been identified and requires the highest level of protection of the skin, eyes, and respiratory system based on either:
  - The measured (or potential) high concentrations of atmospheric gases, vapors, or particulates; or
  - If site operations and work functions involve a high potential for splash, immersion, or exposure to unexpected vapors, gases, or particulates which are harmful to skin eyes, or the respiratory system.
- There is a known or suspected high degree of hazard to the skin and skin contact is possible.
- Conducting work in a confined, poorly ventilated area and the other criteria requiring Level A PPE have not been determined.

Level A equipment includes:

- NIOSH approved positive pressure, full-face piece self contained breathing apparatus (SCBA), or positive pressure supplied airline respirator with escape SCBA;
- Totally encapsulating chemical-protective suit (material based on the hazard);
- Chemical resistant outer **and** inner gloves (type and material based on the hazard);
- Chemical resistant boots with steel toe and shank;
- Disposable protective suit, gloves and boots (depending on suit construction, may be worn over the totally encapsulating suit);
- Coveralls (optional, as applicable);
- Long underwear (optional, as applicable); and
- Hard-hat - under suit (optional, as applicable).

Level B Protection

Level B PPE offers a high degree of respiratory protection with lesser levels of skin protection. Level B PPE should be worn when:

- The type and atmospheric concentration of substances have been identified and require a high level of respiratory protection but less skin protection;
- The atmosphere contains less than 19.5 percent oxygen; or
- The presence of incompletely identified vapors or gases is indicated by direct reading organic vapor detection instruments, but the vapors and gases are not suspected of

containing high levels of chemical harmful to the skin or capable of being absorbed through the skin. Level B is the minimum level of protection that should be worn when there is insufficient information to determine the hazards or potential hazards of the substance.

Level B PPE equipment includes:

- NIOSH approved positive pressure, full face piece self contained breathing apparatus (SCBA), or positive pressure supplied air respirator with escape SCBA;
- Hooded chemical resistant clothing (overalls and long sleeve jacket; coveralls; one or two piece chemical splash suit; disposable chemical resistant overalls) (materials based on the hazards);
- Chemical resistant outer **and** inner gloves (material based on the hazards);
- Chemical resistant boots with steel toe and shank;
- Coveralls (optional, as applicable);
- Outer chemical resistant boot covers (optional, as applicable);
- Hard hat (optional, as applicable); and
- Face shield (optional as applicable).

Level C Protection

Level C PPE is used when the concentration and type of airborne substance is known, and the criteria for using an air purifying respirator are met. It should be worn when:

- Atmospheric contaminants, liquid splashes, or other direct contact will not adversely affect or be absorbed through any exposed skin;
- The types of air contaminants have been identified, concentrations measured, and an air purifying respirator is available that can remove the contaminants; and
- All criteria for the use of an air purifying respirator are met.

Level C PPE equipment includes:

- NIOSH approved full face or half mask air purifying respirator (with appropriate cartridges based on the hazards);
- Hooded chemical resistant clothing (overalls and long sleeve jacket; coveralls; one or two piece chemical splash suit; disposable chemical resistant overalls) (materials based on the hazards);

- Chemical resistant outer **and** inner gloves (materials based on the hazards);
- Chemical resistant boots with steel toe and shank;
- Coveralls (optional, as applicable);
- Outer chemical resistant boot covers (optional, as applicable);
- Hard hat (optional, as applicable);
- Escape mask (optional, as applicable); and
- Face shield (optional, as applicable).

Level D Protection

Level D PPE offers the least skin and respiratory protection and should be worn when the atmosphere contains no known hazards, and work functions preclude splashes, immersions or the potential for unexpected inhalation of or contact with hazardous levels of any chemicals.

Level D PPE equipment may include any or all of the following depending on the hazards of the site:

- Chemical resistant boots with steel toe and shank (optional, as applicable);
- Coveralls (optional, as applicable);
- Gloves (optional, as applicable);
- Outer chemical resistant boots (disposable) (optional, as applicable);
- Safety glasses or chemical splash goggles (optional, as applicable);
- Hard hat (optional, as applicable);
- Escape mask (optional as applicable); and
- Face shield (optional as applicable).

5.5 Combinations of Protection

Combinations of protection are acceptable if the task hazard analysis and the site conditions warrant modification of PPE levels.

5.6 Equipment List

Eye/Face Protection

All employees engaged in or working in or adjacent to areas with eye-hazardous activities or operations, such as but not limited to flying objects and hazardous chemicals shall wear appropriate eye protection.

- Safety glasses with side shields are required for impact protection and shall meet ANSI Standard Z87.1 requirements.
- Chemical goggles (for protection against chemical splash).
- Face shields (for face protection from chemical splash and are not a substitute for primary eye protection).
- Full-face respirators can provide eye and face protection in lieu of safety glasses, goggles, or face shields.
- Shaded eye protection meeting the minimum shade requirements established in 29 CFR 1910.133 (for employees exposed to sources of injurious light radiation [e.g., welding, cutting, lasers]).
- For prescription eye protection contact your supervisor to fill out an AOSafety order form available on APEX.

Respiratory Protection

Respirators will be provided and used in accordance with the ARCADIS Respiratory Protection Policy/Procedure ARC HSGE017 and 29CFR 1910.134.

Hearing Protection

Hearing protection will be provided and used in accordance with the ARCADIS Hearing Conservation Policy/Procedure ARC HSIH008 and 29CFR 1910.95.

Foot Protection

- Basic foot protection is required for all ARCADIS job sites and industrial locations. Specialized footwear will be provided as required by the nature of the work. Special foot protection may include, but is not limited to, chemically resistant, thermally shielded, metatarsal guards, etc.
- Leather Safety Boots will be provided for employees employees; one pair of leather safety boots will be provided as necessary by ARCADIS. The employee purchasing the footwear is required to ensure that it meets ANSI Standard Z41.
- For most work done by ARCADIS, safety boots will be equipped with steel toes and shanks. It is also required that puncture resistant soles or in-soles are equipped in the safety boots. Some clients require puncture resistant soles or in-soles.

- The maximum expenditure or reimbursement for approved safety shoe purchases will be $150. Reimbursement requests must be approved by the employee's supervisor.

- Athletic-style safety shoes ("safety sneakers") are prohibited (due to the difficulties created by these styles in supervising proper use of protective footwear).

Head Protection

Hard hats meeting ANSI Z89.1 will be provided to protect employees from impact, penetration, falling objects, and/or limited electrical shock and burn, as appropriate for work site hazards. A hard hat must be replaced when it becomes damaged, contaminated (and contamination cannot be removed) or it has been struck by an object of sufficient size to potentially compromise its integrity.

Hardhats must resist penetration by objects, be water resistant and slow burning, and have a chin strap if it is worn while working at elevation. It must be worn square on the head and not be pushed back, to the side or forward.

There are two types and three classes of head protection described in ANSI standard

Z89.1-1997. The types and classes are divided by the protection they provide from impact and electricity. It is important that the level of protection necessary be evaluated when during preparation of the site specifc HASP.

Other hazard situations to consider are:

- In areas of heavy vegetation or in any area where hunters may be present, it is recommended that some type of brightly colored head protection be worn. For example, a bright orange or yellow baseball cap or stocking cap.

- If cold exposure is an issue, hardhat liners are available (made specifically for the particular hardhat) or if a hardhat is not required, some type of insulated head protection such as a stocking cap should be worn.

Hand Protection

Appropriate hand protection will be provided if employee's hands are exposed to hazards while on the job.

such as:

- pinch points
- sharp/pointed tools or objects
- incorrect or inadequate tool use
- improper use
- rotating/energized/automated parts
- abrasive materials

- inadequate job planning
- lack of/inadequate protection
- changing weather conditions and extreme temperatures
- hazardous material
- jewelry and loose clothing.

Once these hazards are identified, the appropriate glove or hand protection must be selected. When choosing gloves, keep in mind:

- Hazardous Chemicals/Substances to be Contacted
- Nature of Contact (total immersion, splash, etc.)
- Duration of Contact
- Area of Protection (hand only, forearm, arm)
- Equipment (rotating, sharp edges, etc.)
- Grip (dry, wet, oily)
- Thermal Protection
- Abrasion/Cut/Puncture Resistance
- Tear/Tensile Strength
- Ergonomics (size, heat stress, dexterity)
- Decontamination/Disposal

In selecting chemically protective gloves, the toxic properties of the chemical(s) will be determined. Information provided on the manufacturer's label or by chemical compatibility charts regarding breakthrough time, permeation rate, and degradation should be considered during selection.

Body Protection

Protective clothing, gloves, boots, and other protective equipment will be provided as appropriate for the hazards associated with the tasks being performed.

Long pants are required for all field work unless approval is granted by corporate H&S. . Additional protection such as cooling vests may be required. In environments with potential biological hazards such as ticks, plantsor snakes, gloves and long sleeves should be worn along with head protection of somekind to protect the scalp. In areas of roadway work or other vehicle traffic high visibility Class II safety vests will be worn.

Chemically Protective Clothing (CPC) will be selected by evaluating the performance characteristics of the CPC against the requirements and limitations of the site and task-specific conditions. This selection should be performed by an employee with training and experience taking into consideration:

- Permeation, degradation, penetration of the CPC by the chemical and;
- Durability, flexibility, fit, temperature effects, ease of decontamination, compatibility with other necessary equipment (e.g., hardhats, SCBA, etc.); and duration of use that could effect the employees ability perform the task.

Specialized Equipment

All other specialized safety equipment required for an assignment (e.g., work gloves, specialized protective clothing, hip boots, field rain gear, personal floatation devices) will be provided by the Firm as specified in the HASP.

Extreme Cold Environments

Supervisors will provide as necessary any of the following to protect from extreme cold environments:

- Hats/hat liners and gloves
- Thermal clothing
- Hi-Visibility clothing
- Winter footwear

Use of specialized equipment will be charged to projects in accordance with established policy and rental rates.

5.7 Maintenance/Storage/Disposal

PPE Maintenance and Disposal

PPE must be inspected by the user before and after each use for defects, rips, tears, and/or damaged parts. Damaged or compromised PPE will not be used and must be repaired before re-use or disposed. PPE must be disposed of according to the HASP and other project plans for the site. If non-disposable, PPE must be decontaminated and sanitized before being reused according to the HASP Contaminated PPE which cannot be properly decontaminated by normal procedures must be disposed of accordingly.

PPE Storage

All PPE must be stored to protect against dust, sunlight, extreme heat and cold, excessive moisture, and damaging chemicals. Storage must be in accordance with the manufacturer's specifications.

Contaminated Boots

Boots contaminated or damaged on the job will be replaced. Contaminated boots will be disposed of with the site waste.

6. TRAINING

Training in the proper use of PPE will generally be provided in conjunction with HAZWOPER training or via coursework selected and approved by Corporate H&S. Training will be completed prior to the employee's use of PPE, when changes in the work place alter the use or type of PPE, and when inadequacies in the employee's knowledge or use of PPE are noted.

The training will include at a minimum:

- When and what PPE is necessary;
- How to put on, adjust, wear and take off the PPE;
- Limitations of the PPE; and
- Proper care, maintenance, useful life, and proper disposal of PPE.

7. REFERENCES

- 29 CFR 1910.120 "Hazardous Waste Operations and Emergency Response"
- 29 CFR 1910 Subpart I "Personal Protective Equipment"

8. RECORDS

Records of the PPE training are retained by the Employee and in the ARCADIS training database. Medical certification/recertification are retained by Corporate H&S.

9. APPROVALS AND HISTORY OF CHANGE

Approved by: Michael A. Thomas, CIH, Director H&S Environmental Division

Michael A Thomas

History of Change

Revision Date	Revision Number	Reason for change
20 February 2009	01	Original document

ARCADIS infrastructure, environment, facilities	ARCADIS HS Procedure Name Roadway Work Zone Safety, Traffic Control and Employee Safety on Private Roadways and Parking Areas	Revision Number 1
Implementation Date January 4, 2010	ARCADIS HS Procedure No. ARC DOT-302	Revision Date January 4, 2010
Author Sam Moyers	Page 1 of 7	Approver Mike Thomas

1. POLICY

It is the policy of ARCADIS that employees performing work in or adjacent to private roadways or in parking areas be adequately protected from vehicular and equipment traffic.

2. PURPOSE AND SCOPE

2.1 Purpose

This procedure provides guidance to employees on ARCADIS requirements for protection from site traffic on private roadways and work in parking areas not under the control of DOT or similar jurisdiction. This procedure provides requirements for preparation of a Site Traffic Awareness and Response (STAR) Plans for private property traffic control and management. This procedure also provides guidance on general H&S issues associated with safety and training requirements.

2.2 Scope

This procedure is specific to ARCADIS work performed in private roadways and parking areas. Work zone safety and traffic control on public roadways is addressed in the ARCADIS H&S Procedure ARC DOT-301, “Traffic Control and Employee Safety on Public Roadways” (ARC DOT-301).

This procedure does not address work on airport or railroad property where exposure to plane or train traffic is expected.

If the client has more restrictive requirements than what is presented in this procedure, the more restrictive requirement will apply.

3. DEFINITIONS

Engineering Judgment - The evaluation of available pertinent information and the application of appropriate principles, standards, guidance, and practices as contained in the Manual on Uniform Traffic Control Devices (MUTCD) and other sources, for the purpose of deciding upon the applicability, design, operation, or installation of traffic control devices. Engineering Judgment shall be exercised by a qualified engineer, or by an individual working under the supervision of a qualified engineer, through the application of procedures and criteria established by the engineer.

Client Site - Any portion of the client’s facility under the control of the client and is not part of the public right of way. Excludes airports and railroad property where exposure to plane or train traffic is expected..

“Must”, “Shall” and “Will” – means the requirement is mandatory. This definition also applies to equivalent terms in DOT Fact Sheets associated with this procedure.

Parking Area or Parking Lot – Any portion of the client site designated for parking vehicles or equipment. Paved or unpaved completion.

Reasonable Time and Place - A condition where the employee conducting the required task is permitted to perform the task in a rested state, free from any condition that would compel the employee to hurry through the assigned task due to:

- Work schedule,
- Budget, and/or
- Mandate requiring the task be performed during times normally considered by employees to be free of any obligation to perform work on behalf of ARCADIS (vacation, holidays, etc.)

Roadway – That portion of a road improved, designed, or ordinarily used for vehicular travel and parking lanes, but exclusive of the sidewalk, berm, or shoulder.

“Should” and “May” – Means the requirement is recommended, if the requirement(s) is not in conflict with an applicable regulatory or client requirement(s). This definition also applies to equivlanet terms in DOT Fact Sheets associated with this procedure.

STAR Plan – A plan for the control and management of traffic on private roadways or parking areas for the protection of project workers. The term includes any requirements presented in project specific HASPs, Job Loss Analyses (JLAs), and/or in a STAR Plan template.

Temporary Traffic Control Zone – An area of a highway where road user conditions are changed because of a roadway work zone or incident by the use of temporary traffic control devices, flaggers, uniformed law enforcement officers, or other authorized personnel.

Traffic Control Device – A sign, signal, marking, or other device used to regulate, warn, or guide traffic, placed on, over, or adjacent to a street, highway, pedestrian facility, or shared-use path by authority of a public agency having jurisdiction.

4. RESPONSIBILITIES

4.1 Corporate Health and Safety

Corporate Health and Safety (through the DOT Compliance Manager) has the overall responsibility for the development, revision and maintenance of the ARCADIS DOT Program for Work Zone Safety.

4.2 Managers and Supervisors

Each ARCADIS Manager acting in a Project Manager or Task Manager responsibility, over projects involving STAR work, will steward the DOT Program for Work Zone Safety and audit, to the extent necessary, each project team member’s compliance with this program. Each affected manager will:

- Ensure financial resources and appropriate time scheduled to adequately construct and implement required STAR Plans,

- Make employees that are responsible and accountable for implementing any STAR Plan requirements available at a reasonable time and place to review the relevant STAR Plan and provide an opportunity for the employees to ask questions regarding the plan's content.

- Hold employees implementing STAR Plan requirements accountable if found in non compliance with the STAR Plan requirements.

- Make employees responsible for the development of STAR Plans available at a reasonable time and place to obtain any required training mandated by this procedure or by federal or state regulation.

4.3 Employees

Each Employee having a responsibility to implement any aspect of a STAR Plan will follow the STAR Plan as instructed. Employees have the absolute right to stop work if asked to perform work (either by ARCADIS or the client) in a manner inconsistent with the STAR Plan. If deviations from the STAR Plan are necessary due to site or other conditions, these deviations will be documented.

5. PROCEDURE

5.1 General

All work performed on or adjacent to private roadways and parking areas will be conducted in a manner that will protect workers from site traffic. Requirements for private property traffic control will be addressed in the project specific HASP, JLA or in the STAR Plan template. Elements of the STAR Plan must be consistent with:

- The ARCADIS DOT Program for Roadway Work Zone Safety; and/or

- Any applicable client requirement relating to facility traffic control;

All employees working in a STAR area must have protection from traffic hazards beyond the use of personal protective equipment (PPE). This includes any work in a Temporary Traffic Control Zone on a private roadway or in a parking lot. Each STAR Plan will address the specific project methods and equipment to be used to address employee protection beyond PPE.

The STAR Plan is considered an extension of the project specific HASP. An ARCADIS STAR Plan Template is available on the DOT Team Site and in the HASP section of the Health and Safety Team Site of the APEX to facilitate adherence to this procedure, however, use of the template is not mandatory as long as effective written controls are addressed in the HASP or JLA for the project.. Alternate formats of a standalone STAR Plan are acceptable if a specific format is required by a client.

The STAR Plan template, if utilized, will be included as an appendix to the HASP for use by employees in the field.

5.2 Relationship of STAR Plan Requirements to the DOT MUTCD

STAR design and layout used should be consistent with the MUTCD to the extent practical. Traffic control devices used for traffic control on private roadways and parking areas will meet MUTCD requirements for color, shape, reflectivity, and design.

5.3 Relationship of STAR Plan to Traffic Control Plan (TCP) Requirements for Public Roadways

Project sites with on-site activities requiring development of a STAR Plan and also having off-site activities requiring a TCP, may have all requirements for both on-site and off-site traffic control presented in a TCP that is reviewed and approved by an employee with Engineering Judgment. See ARC DOT-301 for more information.

5.4 STAR Plan Requirements

5.4.1 Required STAR Plan Elements

Each project specific STAR Plan will be in writing (computer generated or hand written) and will contain the following elements:

- General Project Information (see note below)
    - Project name;
    - Project number;
    - Name of individual responsible for developing the STAR Plan;
    - HASP Reviewer name;
    - Name of individual responsible for implementing the STAR Plan ("TBD" or similar entry is not permitted). Back up persons may also be provided if they meet the requirements of this procedure.
- Project Description (see note below)
    - A brief description of the work to be performed, including estimated durations of time the worker will be in the roadway or parking area.
- Anticipated vehicles or equipment expected to be encountered on the client site.
- STAR Layout
    - A narrative or pictorial (created by drafting or legibly hand drawn) description, of the traffic control device layout. Compliance may be achieved by:
        - Attaching the applicable DOT Facts 301 or 302 series fact sheet(s) to the STAR Plan; or

        - Attaching the relevant diagram, drawing or narrative from client specific documents; or
        - Attaching a specific legible drawing relevant to the condition expected to be encountered during project work.
        - Manual revisions of the above documents are authorized to ensure project specific control.

- Indication of the traffic control devices, as appropriate and applicable, to be used on the project:
    - Number of warning signs including wording to be used on the signs.
    - Number of cones, barrels, etc.
    - Number of flaggers, etc.
- Approvals (see note below)
    - Signature and date STAR Plan developer.
    - Signature of HASP Reviewer (an employee with Engineering Judgement may also approve a STAR Plan even if not designated as a HASP Reviewer).
- Reviews (see note below)
    - Signature and of each employee performing work within the private roadway or parking area

Note: Information does not need to be duplicated if embedding the STAR Plan requirements within a HASP or JLA (project numbers, names, signatures, etc).

5.5 General PPE Requirements

Each employee will wear, at a minimum, a Class II reflective vest meeting ANSI 107-1999 requirements either orange or lime green in color (the latter color is preferred). A Class III reflective vest is required if night work.

Other required PPE includes:

- Safety glasses;
- Hard hat;
- Sturdy boot with ankle support (steel toe may be required based on project safety requirements); and
- Other PPE, including any other required high visibility clothing, required by the project specific HASP, JLA or STAR Plan template will also be worn, as specified.

5.6 Cone Requirements

Cones used in parking areas will meet recommended height and visibility requirements in DOT Facts-302b. At least one 42 inch high channelizer cone will be available at each work location in the parking area. Cones may be augmented with flags to increase visibility.

5.7 Use of Safety Fencing and High Visibility Caution Tape

Use of orange safety fencing and/or high visibility caution tape is recommended for static work activities in parking areas having durations greater than 1 hour per location. These devices may be used for shorter duration work if specific pedestrian control is required. Example work activities where use of these devices is recommended include, but is not limited to:

- Drilling operations;
- Excavation operations; and/or
- Long term aquifer testing activities.

Use of these devices should be used in conjunction with 42 inch tall channelizer cones. Caution tape and safety fencing will be inspected and maintained in good, secure condition at all times when used on the project.

5.8 Vehicle Requirements

Each ARCADIS operated vehicle operated in a private roadway work zone should be equipped with high intensity rotating, flashing, oscillating, or strobe lights. If the supplementary lighting is present, the lighting will be maintained in operational condition at all times. Standard vehicle flashers will be when used if other suggested lighting is not available.

6. Training

Each employee having a responsibility to implement any aspect of the STAR Plan will be trained to the level appropriate to perform the required duty. The minimum training requirement includes review of any applicable DOT Fact Sheet(s) relevant to the work being performed and/or other applicable work standard.

7. Reference Documents

- Manual on Uniform Traffic Control Devices, US Department of Transportation, Federal Highway Administration, current edition.
- ARC DOT-301, “Roadway Work Zone Safety, Traffic Control and Employee Safety on Public Roadways.”

- ARCADIS STAR Plan Template for Private Roadways and Parking Areas
- ARCADIS DOT Facts 300 series fact sheets

8. Records

STAR Plans, including any attached documents, will be retained with the project specific HASP in the project files. The STAR Plan and associated documents may be kept in hard copy or electronic formats. File retention for STAR Plans and associated documents will be consistent with ARCADIS requirements for document retention.

9. APPROVALS AND HISTORY OF CHANGE

Approved By: Sam Moyers, DOT Compliance Manager, ARCADIS

Approved By: Mike Thomas, H&S Director for Environment Division

Michael A. Thomas

History of Change

Revision Date	Revision Number	Reason for change
October 30, 2009	DRAFT	Original draft document
November 4, 2009	DRAFT	Revised draft
January 4, 2010	1	Final document

ARCADIS Infrastructure, environment, buildings	ARCADIS HS Procedure Name Tailgate Health and Safety Meetings	Revision Number 03
Implementation Date 14 September 2009	ARCADIS HS Procedure No. ARC HSGE001	Revision Date 22 February 2010
Author Mike Thomas	Page 1 of 3	Approver Mija Coppola

1. POLICY

It is ARCADIS US policy that ARCADIS staff will participate in tailgate meetings to be held at least once daily on ARCADIS project sites that occur outside of an office environment to ensure that the health and safety issues of the day's activities are understood by all affected parties and that appropriate controls are in place.

2. PURPOSE AND SCOPE

2.1 Purpose

This procedure describes the requirements for implementing an incident- and injury-free workplace by providing guidance on tailgate safety meetings to be performed prior to all projects performed by ARCADIS staff outside of an office-setting or environment.

2.2 Scope

This procedure applies to all non-office related activities performed by ARCADIS or on behalf of ARCADIS. If the site and project is controlled by ARCADIS, tailgate meetings will include the participation of all ARCADIS staff, ARCADIS subcontractors and other involved site personnel as appropriate. If the site is controlled by another party (e.g., a construction site on which ARCADIS is providing a resident engineer or owner's representative), then ARCADIS staff should attend the tailgate meeting held by the controlling party, if one is held. If the tailgate meeting does not address ARCADIS activities or is not deemed adequate, then the ARCADIS staff will hold their own tailgate meeting following this procedure.

If there is only one ARCADIS staff on the site for the day, then the PM and field staff will conduct the tailgate via phone as deemed appropriate.

It is also ARCADIS US policy that more than one tailgate meeting may be held as appropriate for the activities.

3. DEFINITIONS

Definitions applicable to this procedure may be found in ARC HSMS000 – Health and Safety Management System.

4. RESPONSIBILITIES

Field Supervisor – In the scope of this practice, the designated field supervisor will lead or designate an alternative leader to lead the tailgate meeting. In addition, the field supervisor will verify that in the tailgate meeting, the following are clearly established, communicated and reinforced, and that the workforce understands them:

- A process for the transfer of control of work between work groups as appropriate and applicable

- Specific procedures and policies that will be followed (e.g., Health and Safety Plan (HASP), Job Loss Analysis (JLA), H&S Procedures, Field H&S Handbook, etc.)

- Assignment of other responsibilities based on the site activities and hazards to competent staff

Project and Task Managers – are responsible for ensuring that all appropriate hazard assessments have been completed, that all project requirements have been communicated to the field supervisor and other responsible parties, that competent personnel, based on the activities and hazards, have been assigned to the project, and that all employees including ARCADIS, subcontractors and other site personnel know of their requirement and participation in all tailgate meetings conducted for the project.

Health and Safety Staff and Project Site Safety Officers or Supervisors – assist with the completion of hazard identification and assessments as appropriate for the project. In addition, these staff will assist with determining the proper controls and provide information for the tailgate meetings that is relevant to the site activities and the hazards to be encountered by employees.

Employees – are responsible for actively participating in the tailgate meetings, acknowledging their presence at the tailgate meetings, and participating in hazard assessments for the activities in which they will be involved. Employees are responsible for understanding the hazards of their activities, implementing the controls for the hazards and using Stop Work Authority if they don't understand the hazards, their job tasks, or if they do not feel safe.

5. PROCEDURE

5.1 Tailgate Meetings

Tailgate meetings will be held, at a minimum, at the start of each work day, shift or task change. It may be necessary to hold tailgate meetings at other times based on the site, activities, and personnel on the site. Tailgate meetings are usually conducted by the field supervisor, the site safety officer or both. At times, the Project Manager or Task Manager may lead the tailgate meeting.

Work crews that include a lone worker will hold a tailgate meeting by telephone with the Project or Task manager as appropriate. The lone worker or small workgroup will call in at the end of the day to complete the tailgate meeting form per this procedure.

Tailgate meetings will review the planned work activities for the work period, discuss and resolve the risks and mitigations, discuss any health, safety, security and environment concerns and raise the consciousness of each worker before they start work. Utilizing the Tailgate Meeting form in Exhibit 1 will ensure that relevant topics are addressed.

5.2 Tailgate H&S Meeting Form

The *Tailgate H&S Meeting Form* (Exhibit 1) will be used to document the conduct of the tailgate H&S meeting. Copies of the completed form will be kept in the project files. It will be completed by the designated leader of the meeting during the completion of the meeting and for post day activities review as indicated on the form.

5.3 Participation and Preparation

Effective tailgate meetings require participation. When selecting the location of the meetings, the meeting leader will ensure it is in a place free from distraction and that allows for interaction and participant comfort. This will help encourage participation.

6. TRAINING

No specific training or competence is required related to the conduct of the Tailgate Meeting.

7. REFERENCES

ARCADIS Health and Safety Plan procedure – ARC HSFS010

8. RECORDS

Tailgate Meeting forms to be kept on-site and then in project files per ARCADIS project recordkeeping requirements

9. APPROVALS AND HISTORY OF CHANGE

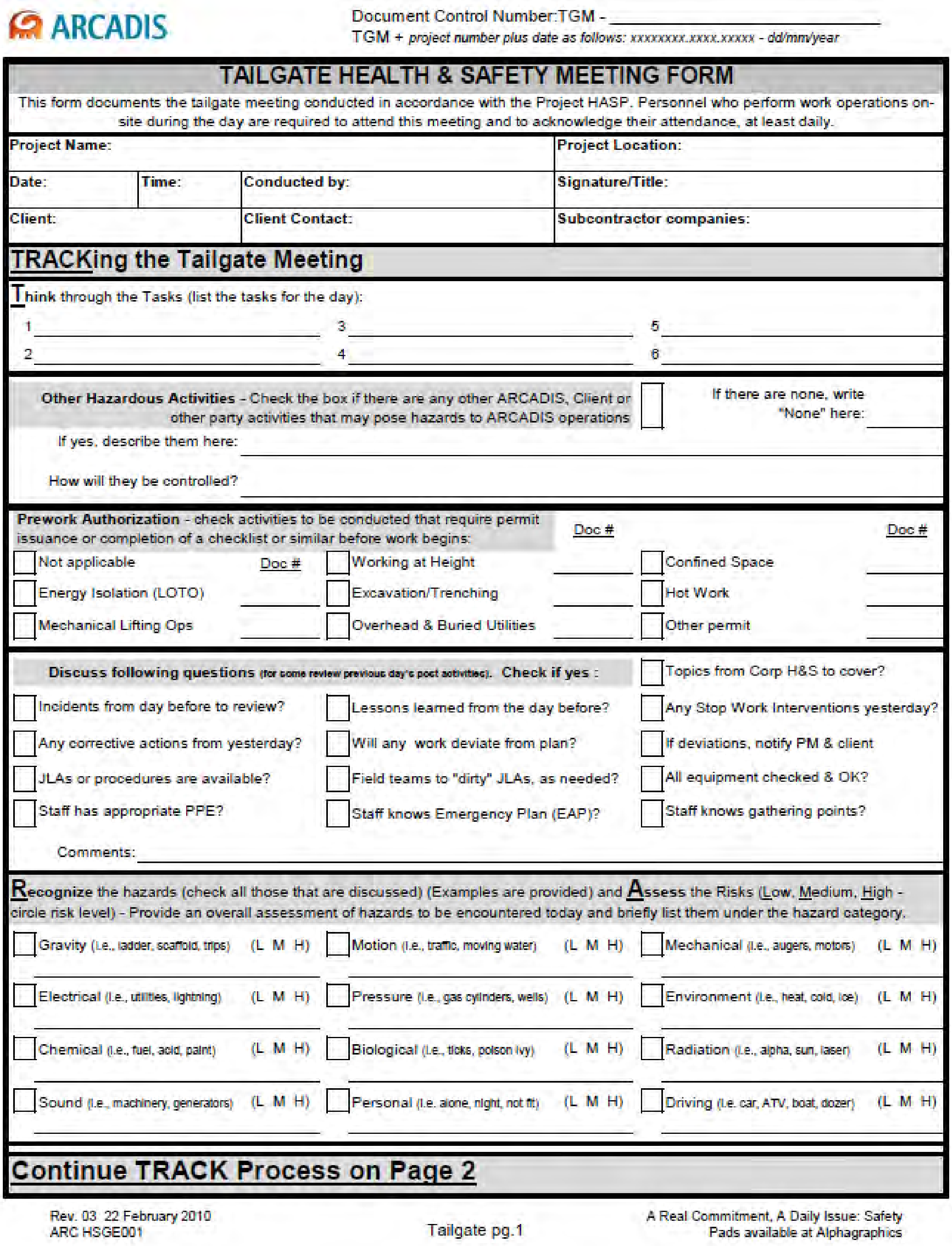
Approved By: Mija Coppola, Director of H&S

Mija A. Coppola

History of Change

Revision Date	Revision Number	Reason for change
14 September 2009	01	Original document
1 Feburary 2010	02	Made minor edits to text. Also, made modifications to Tailgate Meeting form. Changed JSA to JLA.
22 February 2010	03	Added tagline to the Tailgate meeting form

Exhibit 1 –Tailgate Meeting form



ARCADIS

Document Control Number:TGM - ____________________

TGM + *project number plus date as follows: xxxxxxxx.xxxx.xxxxx - dd/mm/year*

TAILGATE HEALTH & SAFETY MEETING FORM

This form documents the tailgate meeting conducted in accordance with the Project HASP. Personnel who perform work operations on-site during the day are required to attend this meeting and to acknowledge their attendance, at least daily.

Project Name:			**Project Location:**
Date:	**Time:**	**Conducted by:**	**Signature/Title:**
Client:		**Client Contact:**	**Subcontractor companies:**

TRACKing the Tailgate Meeting

Think through the Tasks (list the tasks for the day):

1 ____________ 3 ____________ 5 ____________

2 ____________ 4 ____________ 6 ____________

Other Hazardous Activities - Check the box if there are any other ARCADIS, Client or other party activities that may pose hazards to ARCADIS operations ☐

If there are none, write "None" here: ____________

If yes, describe them here: ____________

How will they be controlled? ____________

Prework Authorization - check activities to be conducted that require permit issuance or completion of a checklist or similar before work begins:

	Doc #		Doc #		Doc #
☐ Not applicable		☐ Working at Height		☐ Confined Space	
☐ Energy Isolation (LOTO)		☐ Excavation/Trenching		☐ Hot Work	
☐ Mechanical Lifting Ops		☐ Overhead & Buried Utilities		☐ Other permit	

Discuss following questions (for some review previous day's post activities). **Check if yes** :

☐ Incidents from day before to review?
☐ Any corrective actions from yesterday?
☐ JLAs or procedures are available?
☐ Staff has appropriate PPE?
☐ Lessons learned from the day before?
☐ Will any work deviate from plan?
☐ Field teams to "dirty" JLAs, as needed?
☐ Staff knows Emergency Plan (EAP)?
☐ Topics from Corp H&S to cover?
☐ Any Stop Work Interventions yesterday?
☐ If deviations, notify PM & client
☐ All equipment checked & OK?
☐ Staff knows gathering points?

Comments: ____________

Recognize the hazards (check all those that are discussed) (Examples are provided) and **A**ssess the Risks (Low, Medium, High - circle risk level) - Provide an overall assessment of hazards to be encountered today and briefly list them under the hazard category.

☐ Gravity (i.e., ladder, scaffold, trips) (L M H)	☐ Motion (i.e., traffic, moving water) (L M H)	☐ Mechanical (i.e., augers, motors) (L M H)
☐ Electrical (i.e., utilities, lightning) (L M H)	☐ Pressure (i.e., gas cylinders, wells) (L M H)	☐ Environment (i.e., heat, cold, ice) (L M H)
☐ Chemical (i.e., fuel, acid, paint) (L M H)	☐ Biological (i.e., ticks, poison ivy) (L M H)	☐ Radiation (i.e., alpha, sun, laser) (L M H)
☐ Sound (i.e., machinery, generators) (L M H)	☐ Personal (i.e. alone, night, not fit) (L M H)	☐ Driving (i.e. car, ATV, boat, dozer) (L M H)

Continue TRACK Process on Page 2

Rev. 03 22 February 2010
ARC HSGE001

Tailgate pg.1

A Real Commitment, A Daily Issue: Safety Pads available at Alphagraphics

TAILGATE HEALTH & SAFETY MEETING FORM - Pg. 2

Control the hazards (Check all and discuss those methods to control the hazards that will be implemented for the day): Review the HASP, applicable JLAs, and other control processes. Discuss and document any additional control processes.

☒ **STOP WORK AUTHORITY** (Must be addressed in every Tailgate meeting - (*See statements below*)

- ☐ Elimination
- ☐ Engineering controls
- ☐ General PPE Usage
- ☐ Personal Hygiene
- ☐ Emergency Action Plan (EAP)
- ☐ JLA to be developed/used *(specify)*
- ☐
- ☐ Substitution
- ☐ Administrative controls
- ☐ Hearing Conservation
- ☐ Exposure Guidelines
- ☐ Fall Protection
- ☐ LPO conducted *(specify job/JLA)*
- ☐
- ☐ Isolation
- ☐ Monitoring
- ☐ Respiratory Protection
- ☐ Decon Procedures
- ☐ Work Zones/Site Control
- ☐ Traffic Control
- ☐ Other *(specify)*

Signature and Certification Section - Site Staff and Visitors

Name/Company/Signature	Initial & Sign in Time	Initial & Sign out Time	I have read and understand the HASP

Important Information and Numbers

All site staff should arrive fit for work. If not, they should report to the supervisor any restrictions or concerns.

In the event of an injury, employees will call WorkCare at 1.800.455.6155 and then notify the field supervisor who will, in turn, notify Corp H&S at 1.720.344.3844.

In the event of a motor vehicle accident, employees will notify the field supervisor who will then notify Corp H&S at 1.720.344.3844 and then Corp Legal at 1.720.344.3756.

In the event of a utility strike or other damage to property of a client or 3rd party, employees will immediately notify the field supervisor, who will then immediately notify Corp Legal at 1.678.373.9556 and Corp H&S at 1.720.344.3500

Visitor Name/Co - not involved in work

In Out

In Out

In Out

In Out

I will STOP the job any time anyone is concerned or uncertain about health & safety or if anyone identifies a hazard or additional mitigation not recorded in the site, project, job or task hazard assessment.

I will be alert to any changes in personnel, conditions at the work site or hazards not covered by the original hazard assessments.

If it is necessary to **STOP THE JOB**, I will perform **TRACK**; and then amend the hazard assessments or the HASP as needed.

I will not assist a subcontractor or other party with their work unless it is absolutely necessary and then only after I have done TRACK and I have thoroughly controlled the hazard.

Post Daily Activities Review - Review at end of day or before next day's work (Check those applicable and explain:)

☐ Lessons learned and best practices learned today: ______

☐ Incidents that occurred today: ______

☐ Any Stop Work interventions today? ______

☐ Corrective/Preventive Actions needed for future work: ______

☐ Any other H&S issues: ______

Keep H&S 1st in all things

WorkCare - 1.800.455.6155
Near Loss Hotline - 1.866.242.4304

1. POLICY

It is the practice of ARCADIS and its affiliated companies to implement appropriate, reasonable and practical procedures within acceptable and customary industry practices to promote the health and safety of its employees, and avoid and mitigate exposure of risk in the performance of their work. In furtherance of this policy, ARCADIS promotes and encourages compliance by all employees with this policy and procedures relating to subsurface work and/or investigations (SWI) and working in the vicinity of above ground utilities.

- This procedure is followed by all responsible ARCADIS personnel. Such procedures are included in the project planning processes utilized by ARCADIS personnel.

- Project management procedural requirements are outlined in Section 5.2. All employees included in SWI and above ground utility work are familiar with these procedures.

- Contract Terms: In agreements for SWI with a client, prime contractor, or subcontractors, required terms (Exhibit 1) shall be included for the appropriate allocation of risk of damage to subsurface facilities. If such provisions cannot be agreed upon, the reasons are documented and other risk-management actions identified, such as limits of liability, additional physical investigations, additional lines of evidence of utility location, assignment of risk to subcontractors, etc.

- The policy of ARCADIS encourages and empowers all employees to take such action as they deem appropriate to assure compliance with this policy and procedures both in project planning and field site operations. Such authority is delegated to those on the project site to immediately stop any SWI work or work in the vicinity of above ground utilities where the employee believes that injury to persons or damage to property could occur. Such action is taken without regard to costs or schedule. Personnel should immediately notify their supervisor of any concerns they have when observing any SWI work or work in the vicinity of above ground utilities. In all agreements between ARCADIS and SWI subcontractors, (e.g., drilling subcontractors), provisions shall be included in the subcontract, work authorization or purchase order. These provisions (Exhibit 1) are found on the ARCADIS intranet at the Legal Department team site.

All ARCADIS personnel involved in SWI work or work in the vicinity of above ground utilities will be appropriately trained on this procedure and have the appropriate professional experience for oversight of or involvement in SWI work or work in the vicinity of above ground utilities. ARCADIS Corporate Health & Safety can answer further questions about this policy or the hazards associated with and the control procedure for work in the vicinity of subsurface or above ground utilities.

Again, to support the efforts of ensuring the health and safety of its employees and mitigating risk to ARCADIS, ARCADIS requires that these policies and procedures be followed and implemented at all levels of project management and field implementation.

2. PURPOSE AND SCOPE

2.1 Purpose

This procedure directs general safety procedures associated with the identification and management of above ground and subsurface utility locations on project sites.

2.2 Scope

2.2.1 **Management Requirements** - ARCADIS personnel managing or working on any project requiring SWI and requiring work in the vicinity of above ground utilities must incorporate this procedure into their project planning and field work activities to ensure that all reasonable means to identify utilities are implemented and that appropriate controls have been put in place to minimize or eliminate damage to these utilities and the hazards associated with these utilities. All applicable procedures described in this document must be completed prior to initiating intrusive field work or field work in the vicinity of above ground utilities, or the work cannot proceed.

2.2.2 **Project Management Requirements** - Where SWI are required to be performed by a subcontractor to ARCADIS under its subcontract, project management shall require the subcontractor to adequately incorporate SWI procedures described herein into the subcontractor's scope of work.

3. DEFINITIONS

Above Ground Utilities - For the purpose of this procedure, above ground utilities include, but are not limited to: any above ground line, system, or facility used for producing, storing, conveying, transmitting or distributing communication or telecommunications, electricity, gas, petroleum and petroleum products, coal slurry, hazardous liquids or gases, water under pressure, steam, or other hazardous materials.

Subsurface Utilities - For the purposes of this procedure, subsurface utilities include, but are not limited to: any underground line, system, or facility used for producing, storing, conveying, transmitting or distributing communication or telecommunications, electricity, gas, petroleum and petroleum products, coal slurry, hazardous liquids or gases, water under pressure, steam, or sanitary sewage; underground storage tanks; tunnels and cisterns; and septic tanks.

4. RESPONSIBILITIES

4.1 Project Manager Responsibilities

To prevent injury to employees, avoid disruption to utility services, and help eliminate damage to subsurface and above ground utilities, project managers have the responsibility for utility identification, location, and marking prior to initiating field activities. Most states, provinces, municipalities, and clients have rules, general statutes, or laws that specify the requirements of subsurface utility location prior to intrusive subsurface field activities (i.e., excavation, trenching, boring, and all forms of drilling operations, etc.). The project manager ensures that these laws are followed, and that the directives outlined in this procedure are met for every project involving SWI and work in the vicinity of above ground utilities.

In addition, if field activities are completed in the vicinity of above ground utilities, the project manager is responsible for working with the client to identify the nature of the utilities, and to determine what control processes need to be implemented to prevent damage to these utilities and to minimize any injury in the event there is damage.

4.2 Field Personnel Responsibilities

Field personnel conducting SWI activities and activities where above ground utilities are in the vicinity of the work have the responsibility to read, understand, and follow this procedure and complete the appropriate checklists during the on-site utility locate process. ARCADIS personnel assisting in the identification of underground utilities need to have previous related experience of a minimum of 1 year. Those implementing remote sensing technologies must complete training in those techniques and have 6 months experience operating and interpreting results.

If utilities cannot be located to eliminate any reasonable concern, field personnel can use their Stop Work authority until utility locations can be identified. Field personnel must review this procedure onsite with ARCADIS subcontractors, and ensure they follow the procedures detailed in this document. Any ARCADIS subcontractor not following these procedures will be asked to stop work, and the project manager contacted. Any diversion from this procedure by ARCADIS field personnel must be approved by the project manager with input from Corporate Health & Safety as necessary.

5. PROCEDURE

A flow chart/decision tree of the procedure is presented in Exhibit 2 of this document.

5.1 Lines of Evidence

The following three actions (lines of evidence) are required for the utility location process:

- Contact the State One Call
- Obtain a detailed site utility plan drawn to scale, preferably an “as-built” plan
- Conduct a detailed visual site inspection

In the event that one or more of the above lines of evidence cannot be conducted, or if the accuracy of utility location is questionable, a minimum of one additional line of evidence must be utilized as appropriate or suitable to the conditions. Examples of additional lines of evidence include but are not limited to:

- Private utility locating service
- Research of state, county or municipal utility records and maps including computer-drawn maps or geographical information systems (GIS)
- Contact with the utility provider to obtain their utility location records

- Hand augering or digging
- Hydro-knife
- Air-knife
- Radio Frequency Detector (RFD)
- Ground Penetrating Radar (GPR)
- Any other method that may give ample evidence of the presence or location of subgrade utilities

5.2 Project Management Procedural Requirements

Field activities are planned and designed to avoid contact with and damage to, and minimize interference with subsurface and above ground utilities in the vicinity of ARCADIS work activities. During the planning phase of a project the project manager will insure the appropriate allocation of utility location responsibilities and verify their completion. The utility location activities will implement the lines of evidence as defined in Section 5.1.

5.2.1 Communication and Coordination

The PM or their designated Task Manager:

- Communicates verbally and in writing the responsibilities for utility location with each party
- Provides the list to the site safety officer for inclusion in the site-specific health and safety plan (HASP);
- Communicates potential hazards to field staff prior to mobilization;
- Instructs field staff to be aware of and implement the procedures in the Section 5.1 of this procedure and utilize the appropriate utility location checklists.
- When practical, schedules a joint meeting between the public/private utility locators and field staff to oversee the subsurface utility locating and marking in the field.
- Communicates with and provides utility location documentation to the subcontractors to verify with them the utility locations and discusses methods to be used to protect those utilities.
- Understands the subcontractor’s methods for utility location and documenting the process with a clear delineation of responsibilities for utility location.

In general, subsurface utility locations marked by public utility locators are only good for 2 weeks (research your state-specific requirements). If SWI activities are not conducted during this time period, the site is remarked.

NOTE: At no time is SWI conducted based on old markings, hand-drawn maps/sketches, photographs, or by recollection/memory of field staff. If markings are smeared, removed, damaged, or impacted in any way, the site must be remarked before SWI begins. Flag markings are used in addition to paint markings wherever possible.

5.2.2 Utility Request Notifications for Public Property

Prior to intrusive work on public property (i.e., right-of-ways, easements, etc.), notification of a public one-call service center is completed a minimum of 48-72 hours (states/localities requirements vary, so the PM is responsible for verifying this) prior to initiating field activities (excluding Saturdays, Sundays, and legal holidays). Specific state or local laws related to utility location are evaluated with respect to notification and liability in the event of utility damage. During the call, the responsible party:

- Provides accurate description of the location of all areas of the SWI;
- Documents the utility locate request to record the time and date of the call, the area to be marked, the list of utility companies and municipalities that the one call service center will notify;
- Records the associated ticket (or dig) number provided by the one call service center;
- Cross references the notification list provided by the one-call service center with the list of known or suspected utilities for the property; and
- Provides accurate contact (responsible party name and phone numbers) information for the one call service center so they can subsequently communicate potential questions and/or delays related to the utility location and marking.

After receiving a request, the one-call service center sends requests to participating utility operators who have utilities in the area of the intrusive field activities. Each underground utility operator dispatches their own locators to mark their facilities with paint or flags. The project manager attempts to have field staff present during the marking of the utilities by the locator organization to ensure that the area of the SWI is included in the locating activities. It is important to note:

- Not all utility operators and municipalities participate in one call programs. In some instances, one-call programs provide a list of utility providers that participate, and a list of those that do not. The utility providers that do not participate are contacted individually so that they can mark their own lines, and this call is documented (date of call, person receiving call, date lines will be marked, etc.);

- Public utility locators are usually only required to mark utilities within the public spaces (i.e., right of ways) or at most up to a meter on private property; and
- Knowledge of existing or suspected, but unmarked utilities are documented and communicated to the site safety officer, field staff, and the client prior to implementing field activities.

If a known or suspected subsurface utility does not participate in the state one-call program, and that provider has not been individually contacted prior to the start of SWI, then the field activities are postponed. If these utility providers are contacted and do not provide utility location services, then SWI are not performed until a private utility locating company is contracted and the locating tasks completed.

5.2.3 Nation-wide Utility Locate Call Number 811

State and local utility notification centers participate in a “Call before you Dig” number for public safety and to protect underground infrastructure. This national number is: **811**. The number is designed to help prevent professional excavators, drillers and homeowners from damaging underground utility lines, or causing an injury or service outage while digging/drilling. For more information about the 811 services, visit www.call811.com

The number 811 is an FCC designated national n-11 number. This quick and efficient one call service will notify the appropriate utilities, who participate in the one call program. **However,** callers must still verify who the one call service contacts, and then determine which utilities may need to be contacted directly (e.g. those utilities not participating in the one call service) by following the requirements outlined in this procedure.

5.3 Field Protocol

At no time do field activities that involve SWI or work in the vicinity of above ground utilities commence without the field staff having knowledge of the location of subsurface and above ground utilities. In addition, as stated above and in general, subsurface utility locations marked by public utility locators are only good for 2 weeks (research your state-specific requirements). If SWI activities are not conducted during this time period, all lines of evidence must be re-verified.

NOTE: At no time is SWI conducted based on old markings, hand-drawn maps/sketches, photographs, or by recollection/memory of field staff. If markings are smeared, removed, damaged, or impacted in any way, the site must be remarked before SWI begins. Flag markings are used in addition to paint markings wherever possible.

5.3.1 SWI and Subsurface Utilities

Prior to the start of intrusive activities, all utilities are located and measures instituted to avoid subsurface utility hazards. No SWI will be conducted within 30 inches of a line marking. If SWI must take place within 30 inches of the line marking, an additional line of evidence must be used that will ensure the avoidance of the line. An additional safety measure can include the use of lockout/tagout to render the utility controlled.

Prior to mobilizing to the site for SWI work, field staff reviews the task details with the project manager or their designated authorized TM. This may include but is not limited to review of boring logs, excavation permits, etc. Any special site or client requirements are also discussed. Prior to initiation of any intrusive activities, the utilities and structures checklist (Exhibit 3) is reviewed and completed. Generally, the following colors apply for different types of utilities/operations:

Red – Electric;
Yellow – Natural gas/oil;
Orange – Communication/cable television;
Blue – Water;
Green – Sewer;
Pink – Temporary survey marking;
White – Proposed excavation; and
Purple – Reclaimed water

In addition, the SWI subcontractor marks (i.e., paint, stakes, etc.) the location of their operations to ensure they fall within the area that has been investigated for utilities.

Once the checklist is completed and all utilities identified, any client/site specific utility location or other utility (subsurface or above ground utilities) protection procedures (i.e. such as hand digging to a specified depth, covering or shielding lines, etc.) is completed at each location where work will be completed. If a known or suspected public subsurface utility has not been marked or the markings are not clear, the state one-call number is contacted to determine if an “emergency” locate can be requested. If so, follow the procedures outlined by the locate service and contact the project manager. If it is a private utility that is not marked, the facility manager and/or the project manager should be contacted.

If unexpected conditions are encountered (refusal, debris, pea gravel, etc.) while completing the intrusive activity, all work is immediately halted. Note that subsurface utilities at many industrial facilities are often placed in conduits or concrete to prevent damage. If a utility or subsurface structure is compromised, the field staff initiates the Emergency Action Plan Guidelines (Exhibit 5); however, more detailed emergency action procedures should be reviewed with the client and documented in the site specific health and safety plan prior to initiating work.

5.3.2 Work in the Vicinity of Above Ground Utilities

If activities take place in the vicinity of an above ground utility, the utility line can be rendered controlled (i.e. through lockout/tagout procedures) or protected from damage (i.e. covering overhead power lines). The following table is used to develop acceptable work distances for work involving machinery with high extensions (backhoes, drilling rig masts, etc.) in the vicinity of overhead power lines:

Power Line Voltage Phase to phase (kV)	Minimum Safe Clearance (feet)
50 or below	10
Above 50 to 200	15
Above 200 to 350	20
Above 350 to 500	25
Above 500 to 750	35
Above 750 to 1,000	45

ANSI Standard B30.5-1994, 5-3.4.5

The distance may be lengthened if directed by the client or the electric company, and any specified distances are strictly followed. In addition, work involving machinery, vehicles or equipment that may come in contact with above ground utilities is not completed until those utilities are protected or control processes are in place to avoid damage to those utilities.

If an above ground utility is discovered that has not been previously identified prior to mobilizing to the field, the field staff notifies the project manager who requests the client to assist in the identification of the utility and the implementation of control procedures as appropriate. In addition, if a utility or subsurface structure is compromised, the field staff initiates the Emergency Action Plan Guidelines (Exhibit 5); however, more detailed emergency action procedures should be reviewed with the client and documented in the site specific health and safety plan prior to initiating work.

6. RECORDS

6.1 Utilities Location Records

All records (maps and documentation of communications) used to determine the location of utilities should be retained and kept in the project file.

7. APPROVALS AND HISTORY OF CHANGE

Approved By: Mija Coppola, Director H&S, Infrastructure and PM/CM Divisions

Mija A. Coppola

History of Change

Revision Date	Revision Number	Reason for change
13 December 2006	01	Original document
26 March 2007	02	Put in new company format
15 May 2007	03	Added nation-wide 811 number
6 September 2007	04	Changing over to new template format
22 February 2008	05	Changing over to new template format
13 January 2009	06	Define lines of evidence

Exhibit 1 - Contract Term Language

INSERT INTO ALL CLIENT CONTRACTS OR WORK ORDERS WHERE DRILLING, EXCAVATION, INTRUSIVE WORK IS TO BE PERFORMED.

Site Conditions: ARCADIS shall not be liable for: (i) damage or injury to any subterranean structures (including, but not limited to, utilities, mains, pipes, tanks, and telephone cables) or any existing subterranean conditions; or the consequences of such damage or injury, if (with respect to this clause (i)) such structures or conditions were unknown and were not identified or shown, or were incorrectly shown, in information or on plans furnished to or obtained by ARCADIS in connection with the Services; (ii) concealed conditions encountered in the performance of the Services; (iii) concealed or unknown conditions in an existing structure at variance with the conditions indicated by the Scope of Services or Work Authorization; or (iv) unknown physical conditions below the surface of the ground that differ materially from those ordinarily encountered and are generally recognized as inherent in work of the character provided under this Agreement.

Client shall provide to ARCADIS all plans, maps, drawing and other documents identifying the location of any subterranean structures on the Site. Prior to location of any drilling or excavation below the ground surface, ARCADIS shall obtain the concurrence of the Client as to the location for such drilling or excavation.

Should: (i) concealed conditions be encountered in the performance of the Services; (ii) concealed or unknown conditions in an existing structure be at variance with the conditions indicated by the Scope of Services or Work Authorization; or (iii) unknown physical conditions below the surface of the ground differ materially from those ordinarily encountered and generally recognized as inherent in work of the character provided under this Agreement; then the amount of this Agreement and/or time for performance shall be equitably adjusted by change order upon timely notice.

INSERT INTO ALL DRILLING, EXCAVATION, INTRUSIVE WORK SUBCONTRACTS.

Site Conditions: SUBCONTRACTOR acknowledges that time is of the essence with respect to the performance and completion of its work under this Contract. SUBCONTRACTOR shall adhere to, commence and complete its work in accordance with any schedule incorporated into this Contract, or any schedule submitted by SUBCONTRACTOR or attached hereto; and with respect to any Changes, out of scope or additional work, SUBCONTRACTOR shall expeditiously perform such work according to any schedule therefore agreed to by the parties. In the event any schedule is incorporated in this Contract or attached to this Contract, SUBCONTRACTOR acknowledges and agrees that such schedule has accounted for all inherent or reasonably anticipated delays, including but not limited to those inherent in obtaining site information, access sufficient labor, supplies, tools, equipment and utilities required for the project work, and SUBCONTRACTOR waives any claim of extra compensation or damages therefore.

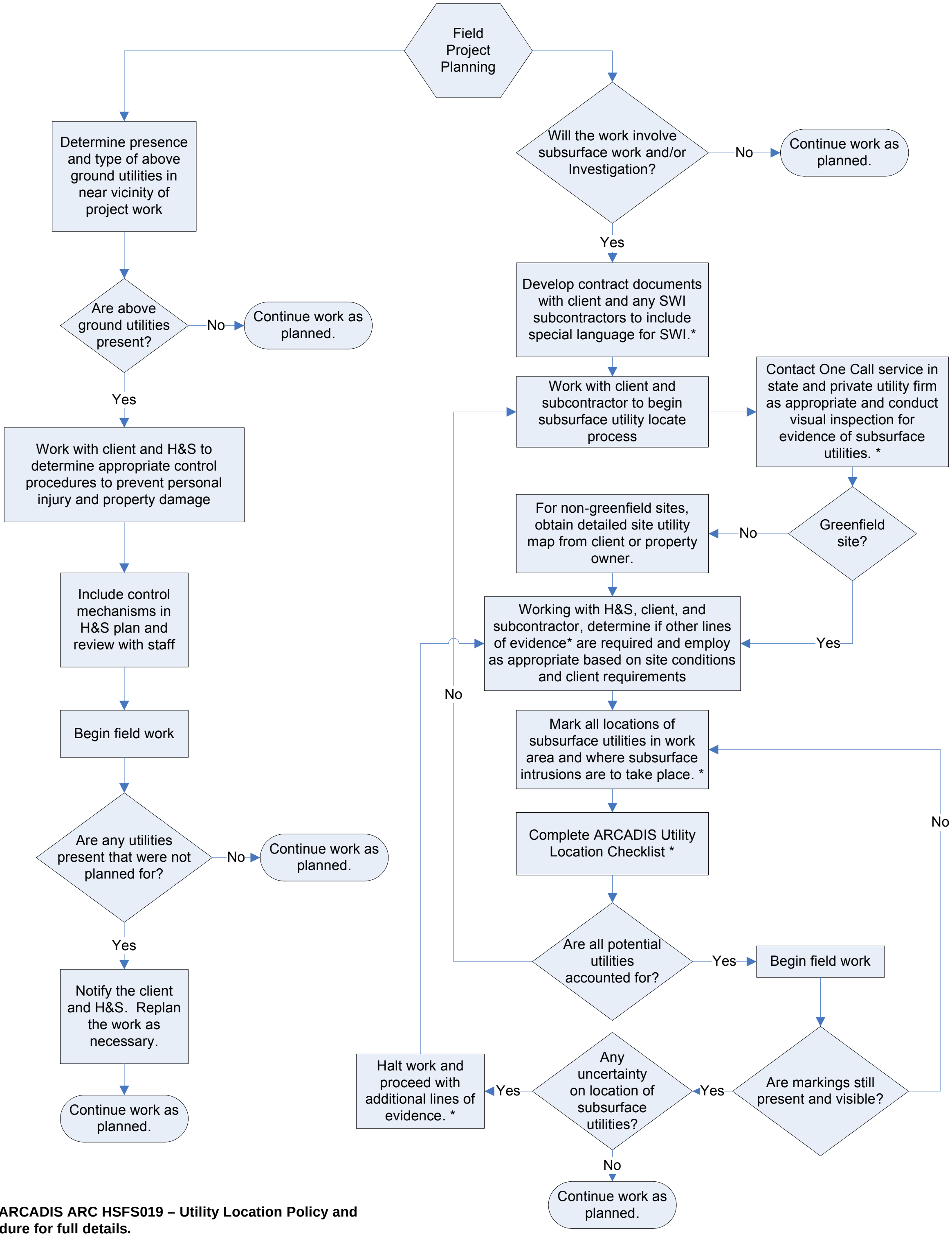
Subcontractor represents and warrants that it has had an opportunity to review and/or has carefully examined all necessary drawings, maps, schematics, specifications, governmental restrictions, permits and license requirements, and all applicable laws, regulations and rules relating to the Work to be done and the Site, it surroundings and local conditions, and has made all investigations based on reasonably available information that are necessary to develop a full understanding of the hazards and difficulties which can be encountered and are likely to impact the cost or schedule to perform the Work. SUBCONTRACTOR is thus familiar with conditions at the Site as are pertinent to or which may affect the Work and has been granted the right to conduct, and has conducted, all investigations it deems appropriate to determine that it can fulfill the requirements of this Contract. Notwithstanding any other provision of this Contract, SUBCONTRACTOR assumes the risk of all conditions, as specified in this Contract, that may affect SUBCONTRACTOR'S ability to perform the Work and will, regardless of such conditions, or the expense or difficulty of performing the Work or the negligence, if any, of ARCADIS, with respect to same, fully complete the Work for the stated price without further recourse to ARCADIS. Information on the Site and local conditions at the Site furnished by ARCADIS are not guaranteed by ARCADIS to be accurate, and is furnished only for the convenience of SUBCONTRACTOR.

The discovery of concealed conditions which could not reasonably have been anticipated by the SUBCONTRACTOR from information available to SUBCONTRACTOR may constitute a changed condition, which, to the extent such condition materially affects the cost or schedule to perform the Work, would entitle the SUBCONTRACTOR to a change and an equitable adjustment of the Contract price or time. SUBCONTRACTOR warrants that it shall conduct appropriate investigations to determine, with reasonable certainty, the location of utility and service lines, underground storage systems, and other subsurface structures of any kind before commencement of any drilling, excavation, or other work that has the potential to disturb these structures. SUBCONTRACTOR further warrants that it shall conduct independent field investigations to confirm the location of subsurface structures before commencement of subsurface work and shall not relay exclusively on plot plans or other drawings provided to SUBCONTRACTOR in conducting these investigations.

Exhibit 2 – Utility Location Decision Tree

Exhibit B- Utility Location Decision Tree*

(PMs or TMs are required to follow both sides of flowchart)



*** See ARCADIS ARC HSFS019 – Utility Location Policy and Procedure for full details.**

Exhibit 3 - Utilities and Structures Checklist

Project:	**Project Number:**
Site Location:	**Date:**

Instructions: This checklist will be used as a safety measure to insure that all underground utility lines, other underground structures as well as above ground utilities are clearly marked out and identified in the area selected for boring or excavation. **DRILLING, EXCAVATION, OR ANY TYPE OF GROUND INTRUSIVE WORK MAY NOT PROCEED UNTIL LINES ARE MARKED AND THIS CHECKLIST HAS BEEN COMPLETED.**

Pre-Field Work Requirements		
Was the state one-call notified with the required advanced notice (usually 48 to 72 hours) (or 811 Nation-wide number)	YES NO	
State one-call confirmation number		
List utility companies who do not participate in the state one call program. Were they contacted directly?		
What additional lines of evidence are used for utility clearance?		
Was a plot plan showing site features and subsurface utilities provided by the PM/TM?	YES NO	
Subgrade Utility Line Location		
Where is the gas line located?		
Where is the gas meter located on the site building(s)?		
Are the electric lines subsurface or overhead? Where are they located?		
Where is electric meter located on the site building(s)?		

Where are the telephone/cable lines located? Are there any overhead lines?	
Where do these lines enter the site building(s)?	
Where are the water lines located?	
Does the site occupant use water (bathrooms, industrial uses, fire suppression, etc.)? If so where do the water lines enter the building for these purposes?	
Are there small manholes/vault covers indicating water lines? If so, where?	
Was the local municipality contacted to mark sanitary lines?	
Where are the sanitary lines located?	
Where might the sanitary lines enter the building? (i.e. what side of the building are the bathrooms, kitchens, water treatment plant, etc?)	
Where are the storm sewer lines located?	
Are there storm sewer inlets located on the property? Check inlets for direction of subsurface lines.	
Are there any gutters directing storm water to the subsurface? Evaluate for direction of lines.	
Underground Storage Tank Sites	
Where are the USTs located? How many USTs are at the site (very number of USTs by counting fill ports and vent lines)?	
Where do the vent lines run?	
Where does the piping run? (Evaluate the path between USTs to dispenser islands).	
Where are the sub-surface electrical lines located which feed power to the UST system?	
General Underground Utility Location Signs	
Are there any cracks resembling straight lines that may indicate the settling of utility lines?	

Are there any patched areas where subsurface repairs may have been conducted?	
Are there any manhole covers or valve boxes that are not associated with marked lines?	
Above ground Utility Line Location	
Are there overhead power lines? If, so where are they located?	
What is the voltage of the overhead power lines?	
Are there any above ground structures (utilities, piping, etc.) that are used by the client? If so, are they located proximal to the work area?	
Do these lines need controlled (locked out) or protected prior to starting work?	
Interviews: Site Owners/Occupants MUST be interviewed for location of private utility lines at the site (if practicable) before start of work	
Name of Owner/Occupant.	
How is this person affiliated with the Site?	
Who interviewed Owner/Occupant?	
Date of Interview	
Specific comments that should be noted from the interview:	

NOTE: If any subsurface utilities listed above are not located, do not proceed with subsurface activities. Contact PM/TM immediately.

Name and signature of person who conducted utility line checklist

Name (print) **Signature** **Date**

Exhibit 4 - Use and Limitations of Utility Locating Methods

Ground Penetrating Radar (GPR)

The GPR system transmits high frequency electromagnetic waves into the ground and detects the energy reflected back to the surface. Energy is reflected along boundaries that possess different electrical properties. Reflections typically occur at lithologic contacts or where subsurface materials have high electrical contrasts, including metal objects such as underground storage tanks (USTs), drums, and utility pipes. These reflections are detected by the antenna and are processed into an electrical signal that can be used to image the subsurface feature. The GPR data will be reviewed in the field to assist in the delineation of potential piping or other subsurface structures.

The detection of subsurface structures located at the site depends on the electrical properties of the soil and the structure's depth, diameter, and composition. GPR is limited to the detection of smaller diameter pipes with depth. Generally, a pipe must increase in diameter by one 1 inch for each foot in depth to be seen using GPR. Also, plastic piping is more difficult to detect than metal piping using GPR, and caution should be used if plastic utility lines are suspected.

Radio Frequency Detection (RFD)

This instrument operates on the principle of radio frequency transmission and detection. The transmitter applies a known frequency to the pipe and the receiver is able to detect this frequency along the length of the structure. The success of RFD in tracing underground utilities is based on the composition of the structure (metal or plastic) and the ability to accurately position the transmitter unit so that it can be attached to, or placed directly over the structure. RFD should only be used to verify the location of utility mark-outs, and not as the primary method of utility identification.

Soil Vacuum Excavation

This method uses nondestructive vacuum excavation methods to create a visual test hole allowing the confirmation of buried utilities. This method is very accurate and relatively fast and can be performed prior to or during the drilling program. The limiting factors for this method are cost and availability. As with specialty drilling methods, a limited number of firms have the equipment to perform vacuum excavation.

The location of the structures to be cleared relative to the source and depth of impacted soil or groundwater is considered. If the zone to be cleared is known not to contain hazardous vapors or petroleum hydrocarbons via previous testing, continuous air monitoring is implemented using a lower explosive level (LEL)/O2 meter and photoionization detector (PID) or flame ionization detector (FID) to the depth of the boring. Also consistent with the site health and safety plan (HASP), air monitoring should be conducted continuously with the LEL/O2 meter during any activity if flammable or explosive vapors are suspected to be present. Prior to any subsurface investigation activities, air monitoring should be conducted to establish background levels for total organic vapors using a PID or FID. All work activity must STOP where tests indicate the concentration of flammable vapors exceeds 10% of the LEL, and the source of vapors must be investigated.

Vacuum-assisted soil excavation utility clearance will not be used in areas know to contain hazardous vapors or petroleum hydrocarbons unless the equipment to be used is suitable for flammable/explosive atmospheres. There is a significantly increased risk of explosion if these materials are encountered while performing this type of utility clearance. Cautions will be performed, as identified below.

Cautions

Many vacuum systems that are commonly used for utility clearance are considered unsuitable for use for environmental investigation sites. Most vacuum units are “Not for use with Hydrocarbons, Explosives, Corrosive or Toxic Material,” and are “Not Intrinsically Safe.”

Given that many units and associated tanking are not explosion-proof, the following steps will be considered prior to using vacuum- assisted utility clearance units where soils could be impacted with petroleum hydrocarbons or flammable vapors.

1. Request from the manufacturer and/or the contractor doing the work to supply manufacturers’ documentation and specifications for use of the unit at environmental sites.

2. Request documentation that the unit is intrinsically safe and may be used in areas where petroleum hydrocarbon may be present.

3. Obtain the procedures for grounding portable units to discharge potential static electricity during operation.

4 If none of the above are available, then hand auger instead and do not use vacuum-assisted methods.

Exhibit 5 - Emergency Action Plan Guidelines

When work activities result in the contact or compromise of a utility line, an appropriate response is critical to prevent injury, death or significant property damage. Although circumstances and response vary depending on site specific conditions, the following guidelines provide information that is factored into emergency action planning associated with utility damage. In any event, emergency planning is coordinated with the entity that owns the utility and the client prior to the start of work. This planning and the appropriate response actions are documented in the project health and safety plan and reviewed with all field staff.

Contact with Above or Underground Electric

Contact with above ground or underground electric lines may result in the equipment being energized. Field personnel do not assume rubber tires on equipment are insulating the equipment from the ground. For underground electric strikes, contact with the line may not be immediately noticeable but indications of a strike include: power outage, smoke, explosion, popping noises, or arching electricity. If contact with an electric line is made or is suspected, the following guidelines are followed:

- Under most circumstances, the equipment operator or any worker on a seat of the equipment should stay on the equipment. These workers should not touch anything, especially metal, on the equipment.

- If it is determined that the equipment should be vacated due to a life threatening circumstance, the worker(s) should jump clear as far as possible from the equipment. When jumping keep both feet together and hop away to a safe distance after landing on the ground. Do not use hand holds or touch any part of the equipment when preparing to jump off.

- Workers on the ground should move away from the equipment.

- Keep others away from the equipment and the area.

- If anyone is injured and in contact with the line or equipment, any attempted rescue should be performed with extreme caution. Only use long, dry, clean, unpainted pieces of wood or fiberglass pole or long dry, clean rope to retrieve the victim. Perform first aid/CPR only after the victim is sufficiently clear from the electrical hazard.

- Notify the electric utility or the client as appropriate for the site. Call 911or the client’s emergency response phone number, as appropriate, for any serious injury or any situation that may result in fire or other hazard that could produce injury or property damage.

Natural Gas

If a natural gas line of any size is compromised, immediately:

- Shut off the equipment and remove any other ignition sources.

- Evacuate the area as quickly as possible.

- DO NOT attempt to turn off any gas valves.
- Call 911 or the designated client emergency response number as appropriate.
- Call the gas utility, if site response is not controlled by the client.
- Do not return to the area until permitted by the utility or by the approved client emergency response personnel, as appropriate.

Water Lines (all types)

Compromised water lines may rapidly become a significant hazard especially if the line is under considerable pressure. Ruptured pressurized water lines may undermine and wash out unconsolidated materials beneath equipment or structures causing them to become unstable. If a pressurized water line is ruptured, the following guidelines should be followed:

- Promptly shut off all equipment.
- Lower masts or other high extension components of the equipment.
- Evacuate area and call the water utility or client emergency response number, as appropriate.
- Turn off the water if the valve location is known and on the site property.
- If potable water lines have been ruptured, attempt to divert any flow away from structures prone to being flooded. Use caution and keep a safe distance from the line break since the ground surface may be compromised.
- For raw process water or other water of unknown quality, do not attempt to divert or contain. Avoid skin contact or accidental ingestion of any water.
- When returning to the area of the break, survey the area for signs of compromised land surface (cracks in asphalt or concrete, depressions in ground, observations of undercutting, etc.) and avoid moving any equipment until these conditions are repaired or resolved.

Sewers (all types)

Use the same general guidelines for water lines when responding to compromised sewers. If a sanitary sewer is compromised additional guidelines should be followed to avoid contracting any bacterial illnesses. These include:

- Promptly evacuate the area.
- Avoid contact with any sewage material.
- If contaminated, promptly was with soap (antimicrobial) and water and promptly change impacted clothing.

- If sewage is accidentally ingested or infiltrates any breach of the skin or enters the eyes, seek medical attention as a precautionary measure.

- Decontaminate equipment with commercially available disinfectant solutions or a 10% chlorine bleach solution.

Communication Lines

Contact and compromise of communication lines are generally considered more of a financial concern than a concern associated with injury. However, eye damage may occur if looking into the ends of a cut fiber optic line. Do not look into the ends of fiber optic lines or other communication lines of unknown type. Promptly contact the communication company owning the line.

Product Lines and Underground Storage Tanks (all types)

Compromise of a product line or underground storage tank (UST) requires immediate action to mitigate impact to the environment. For gasoline stations and similar facilities the following guidelines should be followed during a line or UST breach:

- Immediately shut down equipment and turn off the emergency shutoff switch for the facility dispensers.

- If there are no injuries, attempt to contain any flowing product using absorbent materials and/or by physically pumping or bailing product out of the breached area.

- If product is flowing on the surface away from the break area, attempt to protect downgradient storm drains, sewer drains, and surface water features form impact of the petroleum product using any readily available materials.

- If the bottom of a UST has been breached, immediately contact a pump truck to remove product from the affected UST.

- For releases involving diesel fuel, care will be taken to avoid any situation where diesel may be injected into the body from impalement by coated nails, wood splinters, etc. If diesel is injected into the body, seek prompt medical attention, even if no apparent symptoms of a problem exist.

- Clear area and arrange for prompt repair.

For industrial sites with lines or USTs containing multiple products with varying hazards, similar guidelines may be followed as above if the material encountered is known and workers have a fundamental understanding of the hazards associated with the material. Upon discovery of a line or UST breach due to work activities at these sites:

- Immediately stop work and notify the client representative or call the client designated emergency number. For abandoned sites call 911.

- If the material is not known, promptly evacuate the area and let HAZMAT teams deal with the release.

ARCADIS

Appendix D

PPE Equipment List

PPE CHECKLIST

R = Equipment required to be present on the site. **O** = Optional equipment. **NR =** Not Required. Subcontractors must have the same equipment listed here as a minimum.

Description (Put Specific Material or Type in Box)	**Level Of Protection**		
	D	**C**	**B**
Body			
Coveralls	NR		
Chemical Protective Suit (include type in cell, e.g., Tyvek, Saranex, PVC, etc.)	NR		
Splash Apron	NR		
Rain Suit	O		
Traffic Safety Vest (reflective)	R		
Head			
Hard Hat (if does not create other hazard)	R		
Head Warmer (depends on temperature and weather conditions)	O		
Eyes & Face			
Safety Glasses (incorporate sun protection as necessary)	R		
Goggles (based on hazard)	O		
Splash Guard (based on hazard)	NR		
Ears			
Ear Plugs	R (task appropriate)		
Ear Muffs	O		
Hands and Arms			
Outer Chemical Resistant Gloves (specify the type of glove based on chemical hazard)	R		
Inner Chemical Resistant Gloves (specify the type of glove based on chemical hazard)	O		
Insulated Gloves	O		
Work Gloves*	R (task appropriate)		
Foot			
Safety Boots (steel toe and shank)	R		
Rubber, Chemical Resistant Boots	O		
Rubber Boots	O		
Disposable Boot Covers	O		
Respiratory Protection			
1/2 Mask APR	NR		
Full Face APR	NR		
Dust Protection	O		
Powered APR	NR		
SCBA	NR		
Air Line	NR		

Appendix E

Forms and MSDSs

Forms

ARCADIS

Document Control Number:TGM - ____________________

TGM + *project number plus date as follows: xxxxxxxx.xxxx.xxxxx - dd/mm/year*

TAILGATE HEALTH & SAFETY MEETING FORM

This form documents the tailgate meeting conducted in accordance with the Project HASP. Personnel who perform work operations on-site during the day are required to attend this meeting and to acknowledge their attendance, at least daily.

Project Name:			**Project Location:**
Date:	**Time:**	**Conducted by:**	**Signature/Title:**
Client:		**Client Contact:**	**Subcontractor companies:**

TRACKing the Tailgate Meeting

Think through the Tasks (list the tasks for the day):

1 ____________________
2 ____________________
3 ____________________
4 ____________________
5 ____________________
6 ____________________

Other Hazardous Activities - Check the box if there are any other ARCADIS, Client or other party activities that may pose hazards to ARCADIS operations ☐

If there are none, write "None" here: ________

If yes, describe them here: ____________________

How will they be controlled? ____________________

Prework Authorization - check activities to be conducted that require permit issuance or completion of a checklist or similar before work begins:

	Doc #		Doc #		Doc #
☐ Not applicable		☐ Working at Height	________	☐ Confined Space	________
☐ Energy Isolation (LOTO)	________	☐ Excavation/Trenching	________	☐ Hot Work	________
☐ Mechanical Lifting Ops	________	☐ Overhead & Buried Utilities	________	☐ Other permit	________

Discuss following questions (for some review previous day's post activities). **Check if yes :**

☐ Incidents from day before to review?
☐ Any corrective actions from yesterday?
☐ JLAs or procedures are available?
☐ Staff has appropriate PPE?
☐ Lessons learned from the day before?
☐ Will any work deviate from plan?
☐ Field teams to "dirty" JLAs, as needed?
☐ Staff knows Emergency Plan (EAP)?
☐ Topics from Corp H&S to cover?
☐ Any Stop Work Interventions yesterday?
☐ If deviations, notify PM & client
☐ All equipment checked & OK?
☐ Staff knows gathering points?

Comments: ____________________

Recognize the hazards (check all those that are discussed) (Examples are provided) and **Assess** the Risks (Low, Medium, High - circle risk level) - Provide an overall assessment of hazards to be encountered today and briefly list them under the hazard category.

☐ Gravity (i.e., ladder, scaffold, trips) (L M H) ____________________
☐ Electrical (i.e., utilities, lightning) (L M H) ____________________
☐ Chemical (i.e., fuel, acid, paint) (L M H) ____________________
☐ Sound (i.e., machinery, generators) (L M H) ____________________
☐ Motion (i.e., traffic, moving water) (L M H) ____________________
☐ Pressure (i.e., gas cylinders, wells) (L M H) ____________________
☐ Biological (i.e., ticks, poison ivy) (L M H) ____________________
☐ Personal (i.e. alone, night, not fit) (L M H) ____________________
☐ Mechanical (i.e., augers, motors) (L M H) ____________________
☐ Environment (i.e., heat, cold, ice) (L M H) ____________________
☐ Radiation (i.e., alpha, sun, laser) (L M H) ____________________
☐ Driving (i.e. car, ATV, boat, dozer) (L M H) ____________________

Continue TRACK Process on Page 2

TAILGATE HEALTH & SAFETY MEETING FORM - Pg. 2

Control the hazards (Check all and discuss those methods to control the hazards that will be implemented for the day): Review the HASP, applicable JLAs, and other control processes. Discuss and document any additional control processes.

☒ **STOP WORK AUTHORITY** (Must be addressed in every Tailgate meeting - (*See statements below*)

☐ Elimination
☐ Engineering controls
☐ General PPE Usage
☐ Personal Hygiene
☐ Emergency Action Plan (EAP)
☐ JLA to be developed/used ***specify***
☐
☐

☐ Substitution
☐ Administrative controls
☐ Hearing Conservation
☐ Exposure Guidelines
☐ Fall Protection
☐ LPO conducted ***specify job/JLA***
☐
☐

☐ Isolation
☐ Monitoring
☐ Respiratory Protection
☐ Decon Procedures
☐ Work Zones/Site Control
☐ Traffic Control
☐ Other ***specify***
☐

Signature and Certification Section - Site Staff and Visitors

Name/Company/Signature	Initial & Sign in Time	Initial & Sign out Time	I have read and understand the HASP

Important Information and Numbers

All site staff should arrive fit for work. If not, they should report to the supervisor any restrictions or concerns.

In the event of an injury, employees will call WorkCare at 1.800.455.6155 and then notify the field supervisor who will, in turn, notify Corp H&S at 1.720.344.3844.

In the event of a motor vehicle accident, employees will notify the field supervisor who will then notify Corp H&S at 1.720.344.3844 and then Corp Legal at 1.720.344.3756.

In the event of a utility strike or other damage to property of a client or 3rd party, employees will immediately notify the field supervisor, who will then immediately notify Corp Legal at 1.678.373.9556 and Corp H&S at 1.720.344.3500

Visitor Name/Co - not involved in work

In Out

In Out

In Out

In Out

I will STOP the job any time anyone is concerned or uncertain about health & safety or if anyone identifies a hazard or additional mitigation not recorded in the site, project, job or task hazard assessment.

I will be alert to any changes in personnel, conditions at the work site or hazards not covered by the original hazard assessments.

If it is necessary to **STOP THE JOB,** I will perform **TRACK**; and then amend the hazard assessments or the HASP as needed.

I will not assist a subcontractor or other party with their work unless it is absolutely necessary and then only after I have done TRACK and I have thoroughly controlled the hazard.

Post Daily Activities Review - Review at end of day or before next day's work (Check those applicable and explain:)

☐ Lessons learned and best practices learned today: ____________________
☐ Incidents that occurred today: ____________________
☐ Any Stop Work interventions today? ____________________
☐ Corrective/Preventive Actions needed for future work: ____________________
☐ Any other H&S issues: ____________________

Keep H&S 1st in all things

WorkCare - 1.800.455.6155
Near Loss Hotline - 1.866.242.4304

GENERAL PERMIT TO WORK /TAILGATE MEETING - Chevron

Project Name:

Chevron Facility #:

Project Location (Address):

Total # On-site Workers:

Chevron Project Manager:

Project Type:

Valid Date: **Time Written:** **Time Closed-out:**

1.	a. Have necessary work permits been obtained (including those for subcontractors)?	☐ Yes ☐ No ☐ N/A
	b. If **CONFINED SPACE** activities are to be conducted has the CONFINED SPACE portion of the HIGH RISK WORK PERMIT been completed?	☐ Yes ☐ No ☐ N/A
	c. If **EXCAVATION / TRENCHING / DRILLING / OVERHEAD CLEARANCE** activities deeper than 4 feet and/or within 10 feet of a high pressure gas line and/or within 3 feet of a buried active product or electric line or overhead work involving equipment within 15 feet of an overhead electric line or pole supporting the line are to be conducted has the applicable portion of the HIGH RISK WORK PERMIT been completed?	☐ Yes ☐ No ☐ N/A
	d. If **HOTWORK** activities are to be conducted has the HOTWORK and FIRE MONITOR ACTIVITY LOG portions of the HIGH RISK WORK PERMIT been completed?	☐ Yes ☐ No ☐ N/A
	e (i). If **LOCK OUT / TAG OUT (LO/TO)** activities are to be conducted has the LO/TO portion of the HIGH RISK WORK PERMIT been completed? *See e (ii) below.*	☐ Yes ☐ No ☐ N/A
	f. If **DIVING** activities are to be conducted has the DIVING portion of the HIGH RISK WORK PERMIT been completed?	☐ Yes ☐ No ☐ N/A
	g. If workers are **WORKING AT an ELEVATION over 6 feet** has the WORKING AT ELEVATION portion of the HIGH RISK WORK PERMIT been completed?	☐ Yes ☐ No ☐ N/A
	h. If workers are **BOATING** or **WORKING NEAR OPEN WATER** has the applicable portion of the HIGH RISK WORK PERMIT been completed?	☐ Yes ☐ No ☐ N/A
	i. If **DEMOLITION, REMOVAL OF PIPELINES AND BURIED STRUCTURES** work activities are to be conducted has the EXCAVATION / TRENCHING / DRILLING / OVERHEAD CLEARANCE and DEMOLITION, REMOVAL OF PIPELINES AND BURIED STRUCTURES portions of the HIGH RISK WORK PERMIT been completed?	☐ Yes ☐ No ☐ N/A
2.	Have applicable pre-trip paperwork (JMP and vehicle inspection checklists) been completed by ARCADIS personnel and ARCADIS subcontractors (if applicable)? Has subcontractor paperwork been inspected?	☐ Yes ☐ No ☐ N/A
3.	Have all sections of the Health & Safety Plan (E-HASP) applying to today's tasks been reviewed?	☐ Yes ☐ No ☐ N/A
4.	Has the E-HASP been signed by appropriate on-site personnel?	☐ Yes ☐ No ☐ N/A
5.	Have all visitors and workers completed the E-HASP written test? Have all tested been graded? Deficiencies reviewed?	☐ Yes ☐ No ☐ N/A
6.	Has the scope of work or work plan been reviewed and fully understood?	☐ Yes ☐ No ☐ N/A
7.	Where will the scope of work or work plan be kept on site?	**Location:**
8.	Are appropriate tools on-site to complete tasks safety and appropriately?	☐ Yes ☐ No ☐ N/A
9.	Has Chevron's hand safety policy, including FOBK prohibition, been discussed and highlighted during the health and safety meeting?	☐ Yes ☐ No ☐ N/A
10.	Have Operational Excellence Tenets been discussed and highlighted during the daily health and safety meeting?	☐ Yes ☐ No ☐ N/A
11.	Has everyone reviewed the applicable SOPs and JLAs for their assigned work duties?	☐ Yes ☐ No ☐ N/A
12.	Has JLA been modified in the field to include up-to-minute site conditions and notation of puncture resistant footwear required/not required? Class identification of high visibility vests?	☐ Yes ☐ No ☐ N/A
13.	Where will the E-HASP be kept on site?	**Location:**
14.	Have the action levels and work zones been identified and reviewed?	☐ Yes ☐ No ☐ N/A
15.	Are employees prepared to follow Chevron work zone requirements, including but not limited to: proper delineator height, quantity, vest class?	☐ Yes ☐ No ☐ N/A
16.	What level of PPE is required (See note on following page)?	☐ A ☐ B ☐ C ☐ D ☐ Other
17.	a. If monitoring is required, what type of monitoring will be performed at the site?	**Type:** *(See 17b-17d)* ☐ N/A
	b. Is monitoring equipment present and properly calibrated?	☐ Yes ☐ No ☐ N/A
	c. Have E-HASP requirements for air monitoring been reviewed?	☐ Yes ☐ No ☐ N/A
	d. Will Air Monitoring Log be completed by Health & Safety Supervisor (HSS)?	☐ Yes ☐ No ☐ N/A
18.	Will work conducted by others in the area affect/conflict your work area?	☐ Yes ☐ No ☐ N/A
19.	Will GFCI in-line protectors, positioned next to the power source, be tested and utilized if AC-powered equipment is used?	☐ Yes ☐ No ☐ N/A
20.	Are above-ground utilities identified and clearly visible by equipment operators? Underground utilities marked?	☐ Yes ☐ No ☐ N/A
21.	Has the underground/overhead utilities checklist been completed?	☐ Yes ☐ No ☐ N/A
22.	Have shut-off switches/valves been located (as required by scope work)?	☐ N/A ☐ Electric ☐ Gas ☐ Water
23.	Are proper traffic and/or fire control measures in-place?	☐ Traffic ☐ Fire Prevention ☐ N/A
24.	Who is the designated HSS?	**Name:**
25.	Does everyone know his/her role during an emergency? E-HASP section related to emergency roles and responsibilities reviewed?	☐ Yes ☐ No ☐ N/A
26.	Does everyone know the location, directions, and name of the nearest hospital? Has route and emergency contact numbers been confirmed?	☐ Yes ☐ No ☐ N/A
27.	Has a plan been established to ensure emergency responders can easily locate or be escorted to the work site?	☐ Yes ☐ No ☐ N/A
28.	Where is the support zone located?	**Location:**
29.	Has an evacuation signal (i.e. emergency alarm, hand signal) been communicated to personnel?	☐ Yes ☐ No ☐ N/A
30.	Where is the nearest working phone located (non-cell phone)	**Location:**
31.	What is the local emergency phone number?	**Number:**
32.	Where is the location of the primary first aid kit (to include portable eyewash & CPR shield)?	**Location:**
33.	Where is the location of the primary fire extinguisher?	**Location:**
34.	Document last inspection date of primary fire extinguisher and expiration of primary first aid kit contents:	**Extinguisher -** **First Aid Kit -**
35.	Have modifications to safety procedures or documentation (ie. JMP, TCP) been made and communicated to onsite personnel?	☐ Yes ☐ No ☐ N/A
36.	Has a plan been established to sample, store, label and dispose of waste properly?	☐ Yes ☐ No ☐ N/A
37.	Are MSDS for ALL chemicals being used at site (oils, detergents, preservatives, etc.) included in E-HASP?	☐ Yes ☐ No ☐ N/A
38.	Are personnel qualified to perform work at site? Training records verified?	☐ Yes ☐ No ☐ N/A
39.	Has operating vehicle or machinery in reverse been discouraged during TCP review? Will spotters be used when available?	☐ Yes ☐ No ☐ N/A

Special Safety Concerns For Today

Personnel wishing to volunteer information relating to allergies/ailments/illnesses AND whether s/he is wearing contact lenses:

Last Name:	Note:	Last Name:	Note:
Last Name:	Note:	Last Name:	Note:
Last Name:	Note:	Last Name:	Note:

Short Service Employee (requires prior Chevron PM approval – no more than 1 SSE if < 5 Workers on Site)

Short Service Employees On-Site	Short Service Mentor

HAVE A SAFETY MEETING BEFORE WORK AND MID-DAY

Stop Work Authority – As an employee or contractor for Chevron, you are responsible and authorized to stop any work that is not safe or does not comply with the OE tenets. There will be **NO** repercussions for initiating Stop Work Authority.

NOTICE TO ALL WORKERS: – By signing below, you agree that you have read and fully understand the JLAs applicable to you and your assigned duties.

Signature Section

Name (PRINT)	Company	Before Work (Time)	Signature	Mid Day (Time)	Initials (Mid- Day Meeting)
1.					
2.					
3.					
4.					
5.					
6.					
7.					
8.					

Call the **ARCADIS Near Miss Hotline** to report all near misses!! : **866-242-4304**

"I have assessed the risks posed by work activities planned for today and steps to mitigate those risks (e.g. E-HASP, JLA, PTW, tailgate meeting, coordination with other parties on site, etc.) have been completed to the best of our ability. Work is safe to proceed. If site conditions change, Stop Work Authority will be used to assess work conditions."

Permit Writer/Holder: ____ Date (___/___/___)

Reference Section

5 Keys to Operational Discipline

1. **Everyone knows how to do their job correctly and safely.**
2. **Workers recognize hazards and anticipate unusual situations.**
3. **When unusual situations occur, work is stopped and change is effectively managed.**
4. **Supervisors and leaders reinforce the right behaviors and correct poor behavior and performance.**
5. **Workers expect and demand that their coworkers follow procedures.**

Chevron Work Zone Table

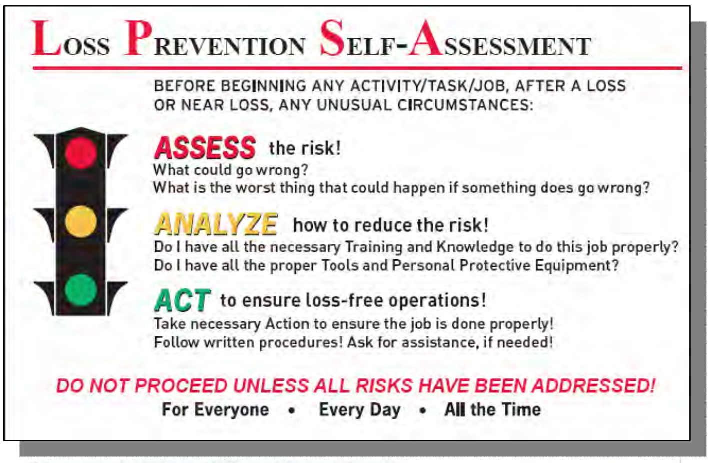
Condition	Level 1	Level 2	Level 3	Level 4
Traffic Volume or Activity	none-low	moderate	high	high
Duration of Activity	< 10 min.	> 10 min.	extended	extended
3rd Party Concerns	low	low	medium	high
Size of Work Area	small	medium	large	large
Use of Heavy Equipment	no	no	yes	yes
DOT Requirements (U.S.)	no	no	no	yes

Safety Vest Classes

Vest Class	When to Use
Class I – high visibility safety vest with no reflective bands	**NOT ALLOWED ON CHEVRON PROJECT SITES**
Class II – high visibility safety vest with reflective bands around chest, shoulders and back	Minimum class allowed at Chevron project sites; used in standard lighting conditions and mod. traffic conditions
Class III – high visibility safety vest with reflective bands around chest or waist, shoulders and sleeves	When performing night-time work or work in low light conditions; when working near areas where traffic flow is over 50 mph

Chevron Work Zone Level Summary

Level 1	Min. of 4 delineators, with max. space between of 4 feet. Use buddy system and vehicle with flashers as necessary.
Level 2	Barrier tape on delineators with flags and the use of field vehicles (with flashing yellow light or vehicle hazard lights) and\or other lane control devices. Metal barricades may be used in conjunction because of the noise they make if knocked over. The buddy system should be used in areas where the worker cannot give undivided attention to traffic when exposed to traffic hazards. Use for temporary securing of an area (for sampling ground water, hand digging, etc.) or for temporary isolation of an area for storing or moving equipment/supplies.
Level 3	Plastic Security Fencing and\or Type 2 or 3 barricades. A flashing yellow light on the vehicle may be used for additional visibility. Be cognizant of blocking of pump islands and any other dealer concerns. To be used when there is heavy equipment, power tools, in areas near electrically energized or rotating equipment, as protection for workers or public near slow moving traffic or with high traffic volume, in driveways, around tripping hazards, and to control third party foot traffic. If there is no third party traffic potential then Level 3 is NOT required.
Level 4	Often requires oversight by a licensed traffic controller. In the U.S., the DOT requires signage and protection devices – refer to the Manual on Uniform Traffic Control Devices (MUTCD). Your country/state/region may have more stringent requirements (e.g., Caltrans WATCH Manual in California). A thorough Job Safety Analysis is required. Note: If the likelihood of vehicles backing in a public right-of-way is low, the 48" minimum height of control devices may not apply.



LOSS PREVENTION SELF-ASSESSMENT

BEFORE BEGINNING ANY ACTIVITY/TASK/JOB, AFTER A LOSS OR NEAR LOSS, ANY UNUSUAL CIRCUMSTANCES:

ASSESS the risk!
What could go wrong?
What is the worst thing that could happen if something does go wrong?

ANALYZE how to reduce the risk!
Do I have all the necessary Training and Knowledge to do this job properly?
Do I have all the proper Tools and Personal Protective Equipment?

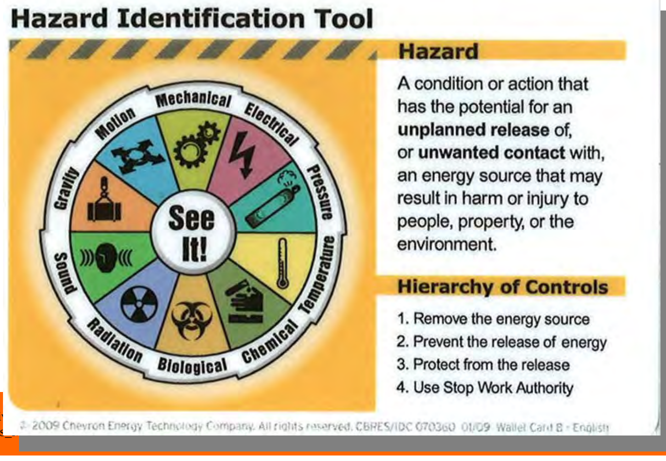
ACT to ensure loss-free operations!
Take necessary Action to ensure the job is done properly!
Follow written procedures! Ask for assistance, if needed!

DO NOT PROCEED UNLESS ALL RISKS HAVE BEEN ADDRESSED!

For Everyone • Every Day • All the Time

Chevron Tenets of Operation

1. **Always operate within design and environmental limits.**
2. **Always operate in a safe and controlled condition.**
3. **Always ensure safety devices are in place and functioning.**
4. **Always perform safe work practices and procedures.**
5. **Always meet or exceed customers' requirements.**
6. **Always maintain integrity of dedicated systems.**
7. **Always comply with all applicable rules and regulations.**
8. **Always address abnormal conditions.**
9. **Always follow written procedures for high-risk or unusual situations.**
10. **Always involve the right people in decisions that affect procedures and equipment**



Hazard Identification Tool

Hazard

A condition or action that has the potential for an **unplanned release** of, or **unwanted contact** with, an energy source that may result in harm or injury to people, property, or the environment.

Hierarchy of Controls

1. Remove the energy source
2. Prevent the release of energy
3. Protect from the release
4. Use Stop Work Authority

Gravity - *falling object, collapsing roof, and a body tripping or falling*

Motion - *vehicle, vessel, or equipment movement; flowing water; wind; and body positioning when lifting, straining, or bending*

Mechanical - *rotating equipment, compressed springs, drive belts, conveyors, and motors*

Electrical - *power lines, transformers, static charges, lightning, energized equipment, wiring, and batteries*

Pressure - *pressure piping, compressed cylinders, control lines, vessels, tanks, hoses, and pneumatic and hydraulic equipment*

Temperature - *open flame; ignition sources; hot or cold surfaces, liquids, or gases; steam; friction; and general environmental and weather conditions*

Chemical - *flammable vapors, reactive hazards, carcinogens or other toxic compounds, corrosives, pyrophorics, combustibles, oxygen-deficient atmospheres, welding fumes, and dusts*

Biological - *animals, bacteria, viruses, insects, blood-borne pathogens, improperly handled food, and contaminated water*

Radiation - *lighting issues, welding arcs, solar rays, microwaves, lasers, X-rays, and NORM scale*

Sound - *equipment noise, impact noise, vibration, high-pressure release, and the impact of noise to communication*

HIGH RISK PERMIT TO WORK

Project Name: Chevron Former Bulk Fuel Terminal
Chevron Facility #: 6517863
Project Location (Address): 431 Veterans Memorial Parkway, East Providence, RI
Total # On-site Workers:
Chevron Project Manager: Peter Kasbohm
Project Type:
Valid Date:
Time Written:
Time Closed-out:

THIS HIGH RISK PERMIT REQUIRES COMPLETION BY AN AUTHORIZED PERMIT APPROVER. EVERY WORKER SHOULD READ AND UNDERSTAND, IN THEIR ENTIRETY, APPLICABLE SECTIONS IN THE SITE HEALTH AND SAFETY PLAN. THIS PERMIT DOES NOT REPLACE TASK-SPECIFIC CHECKLISTS AND FORMS PRESENT IN THE SITE HEALTH AND SAFETY PLAN.

CONFINED SPACE

Confined Space = Space with restricted entry/exit, large enough to contain worker and not intended for worker occupancy.
Permit-required Confined Space = Confined space with a hazard to health or life associated with it.

1.	Location and Description of Confined Space:		
2.	Entry objectives:		
3.	Equipment / Materials required for entry:		
4a.	Time of entry:	4b. Expiration of entry:	
5.	Type of respirator:	6. Level of PPE protection: ☐ Level A ☐ Level B ☐ Level C ☐ Level D	
7.	Air monitoring: ☐ Continuous ☐ 5 minutes ☐ 10 minutes ☐ 15 minutes for ☐ % Oxygen ☐ % LEL ☐ H_2S ☐ CO ☐ Other:		
	Has each entrant received confined space training?	☐ Yes (Stop Work) ☐ No	
8.	Has each entrant developed / reviewed JSA for scope of work in confined space?	☐ Yes (Stop Work) ☐ No	
9.	Have entrants and attendants agreed upon hand signals?	☐ Yes (Stop Work) ☐ No	
10.	Do entrants have any medical conditions or currently taking medication that could affect their safety in the confined space?	☐ Yes (Stop Work) ☐ No	
11.	Is there a rescue plan in place that includes a worker safety line or self-retriever?	☐ Yes ☐ No (Stop Work) ☐ N/A	
12.	Is a trained rescue team available at the entry point?	☐ Yes ☐ No (Stop Work) ☐ N/A	
13a.	Number of Entrants:	13b. Number of Attendants:	
	Name of Entrants	Name of Attendants	
14.	Authorized Permit Approver (PRINT):	Authorized Permit Approver (SIGNATURE):	
15.	Chevron PM acceptance? *Must be accepted by Chevron PM or pre-approved company representative*	☐ E-mail ☐ Fax ☐ Phone (w/ documentation) ☐ No	

EXCAVATION / TRENCHING / DRILLING / OVERHEAD CLEARANCE

Required for excavations deeper than 4 feet, excavations or drilling within 10 feet of a buried high pressure gas line, excavation or drilling within 3 feet of a buried active product line or active electric line, or work involving equipment (such as pulling unit, mast truck, backhoe or excavator) within 15 feet of active overhead electrical line or pole supporting an electric line.

1.	Soil classified as:	☐ Stable rock ☐ Type A ☐ Type B ☐ Type C
2a.	Soil classified on:	2b. Soil classified by:
3.	Type of protection system in use: ☐ Sloping ☐ Shoring ☐ N/A ☐ Other :	
4.	Description of excavation/trench/drilling:	
5.	Have underground utilities been marked by contracted utility locating company?	☐ Yes ☐ No ☐ N/A
6.	Are underground installations including utilities in danger?	☐ Yes ☐ No ☐ N/A
7.	Has the Chevron AND ARCADIS Underground / Overhead Utility Checklist been completed?	☐ Yes ☐ No ☐ N/A
8.	Has the Daily / Periodic Excavation Inspection Checklist been completed?	☐ Yes ☐ No ☐ N/A
8.	Document if the following are present:	
	☐ Electric Power Line (method of marking [i.e. red flags]):	☐ Drain Line (method of marking):
	☐ Natural Gas Line (method of marking):	☐ Underground storage tank (method of marking):
	☐ Telephone Line (method of marking):	☐ Underground Cable (method of marking):
	☐ Water Line (method of marking):	☐ Overhead Power Line (method of marking):
	☐ Product Line (method of marking):	☐ Overhead Product Line (method of marking):
	☐ Sewer Line (method of marking):	☐ Steam Line (method of marking):
9.	Authorized Permit Approver (PRINT):	Authorized Permit Approver (SIGNATURE):
10.	Chevron PM acceptance? *Must be accepted by Chevron PM or pre-approved company representative*	☐ E-mail ☐ Fax ☐ Phone (w/ documentation) ☐ No

HIGH RISK WORK PERMIT

HOT WORK

1.	Location and Description of Hot Work:	
2.	Objective of Hot Work:	
3.	Equipment / Material used:	
4a.	Time of Hot Work:	4b. Time of Hot Work Completion:
5a.	Available fire suppression: ☐ Fixed water supply ☐ Fire blanket ☐ Portable extinguisher (*See 5b.→*)	5b. Quantity, size, and type of extinguisher:
6.	Are fire barriers in place to control sparks?	☐ Yes ☐ No ☐ N/A
7.	Air monitoring: ☐ Continuous ☐ 5 minutes ☐ 10 minutes ☐ 15 minutes	for ☐ % Oxygen ☐ % LEL ☐ H2S ☐ CO ☐ Other:
8.	Have workers developed / reviewed JSA for scope of work?	☐ Yes ☐ No ☐ N/A
9.	Have workers been trained on using fire suppression system at site?	☐ Yes ☐ No ☐ N/A
10.	Are adequate monitors available to maintain continuous, unobstructed vision of the hot work being performed?	☐ Yes ☐ No ☐ N/A
11	Has fire monitor been designated with the sole responsibility of monitoring hot work activity and verifying there is no fire condition AFTER hot work has ended?	☐ Yes ☐ No (Stop Work) ☐ N/A
12a.	Name of Employees Performing Hot Work	12b. Name of Dedicated Fire Monitors
13.	**FIRE MONITOR ACTIVITY LOG**	
14.	14a. Time of pre-work survey:	14b. Pre-work LEL%:
	14c. Pre-work Oxygen %:	14d. LEL% after __________ minutes of work:
	14d. Oxygen % after __________ minutes of work:	
15.	Have hot work areas been monitored for up to an hour after completion of hot work activity?	☐ Yes ☐ No ☐ N/A
16.	Authorized Permit Approver (PRINT):	Authorized Permit Approver (SIGNATURE):
17.	Chevron PM acceptance? *Must be accepted by Chevron PM or pre-approved company representative*	☐ E-mail ☐ Fax ☐ Phone (w/ documentation) ☐ No

LOCKOUT / TAGOUT

1.	Have authorized and affected employees been training in the Lockout / Tagout (LO/TO) program?	☐ Yes ☐ No ☐ N/A
2.	Has the LO/TO Equipment-Specific Energy Control Procedure form been completed?	☐ Yes ☐ No ☐ N/A
2.	Are there multiple keys for the lock?	☐ Yes (Stop Work) ☐ No ☐ N/A
3.	Equipment Identification:	

Hazardous Energy Source		Isolation Device			Verifying Lockout Means of Verification of Lockout
Type & Magnitude	Function	Type	Location	ID #	
Electrical 120V					
Electrical (over 120V)					
Pneumatic					
Hydraulic					
Mechanical					
Potential					
Gravity					
Other					

4.	Have current LO/TO locations been inspected by an employee not involved in the energy control process periodically?	☐ Yes ☐ No ☐ N/A
5.	PERMISSION TO REMOVE LOCKOUT / TAGOUT FROM AUTHORIZED EMPLOYEE (PRINT NAME):	☐ E-mail ☐ Fax ☐ Phone (w/ documentation) ☐ No (STOP WORK) OSHA allows removal of LO/TO devices only if worker that applied the lock is not available and permission has been given by that individual. If permission has not been given, documentation of efforts to obtain permission must be kept. The worker that applied the LO/TO must be notified of its removal prior to returning to the work site. This notification and acceptance should be documented.
6.	Authorized Permit Approver (PRINT):	Authorized Permit Approver (SIGNATURE):
7.	Chevron PM acceptance? *Must be accepted by Chevron PM or pre-approved company representative.* ***Exception: O&M performed AT LEAST Quarterly.***	☐ E-mail ☐ Fax ☐ Phone (w/ documentation) ☐ No

HIGH RISK WORK PERMIT

DIVING

United States Coast Guard: VHF Channel 16 **Dives Alert Network (DAN): 1-919-684-4326**

1.	Location and Description of Diving:	
2.	Objective of Dive:	
a.	Time of First Dive:	3b. Time of Last Dive Completion:
4a.	Name of Dive Safety Officer:	4b. Proposed # of Dives Per Day:
5.	Are divers preparing a dive log?	☐ Yes ☐ No (Stop Work) ☐ N/A
6.	What is the minimum surface time interval between dives?	☐ 1 hour ☐ 2 hours ☐ Other ______ hours
7.	Have divers developed / reviewed JSA for scope of work, which includes entry procedures, depth and bottom time limits, emergency procedures, etc.?	☐ Yes ☐ No (Stop Work) ☐ N/A
8.	Are dive physicals available for each diver?	☐ Yes ☐ No (Stop Work) ☐ N/A
9.	Are divers mentally and physically prepared for diving?	☐ Yes ☐ No (Stop Work) ☐ N/A
10a.	Name of Certified Divers	10b. Name of Support Personnel
11.	Have divers and support team reviewed hand signals, emergency recall signals and emergency procedures?	☐ Yes ☐ No (Stop Work) ☐ N/A
12.	Is dive platform stable, seaworthy and outfitted with proper safety equipment?	☐ Yes ☐ No (Stop Work) ☐ N/A
13.	Has all dive gear been inspected by the user and found to be fully operational?	☐ Yes ☐ No (Stop Work) ☐ N/A
14.	Is a means of communication available? (cellular telephone, landline phone, NAVSAT,etc.)?	☐ Yes ☐ No (Stop Work) ☐ N/A
15.	Is location of diving first aid kit, drinking water, oxygen resuscitator and backboard and rescue equipment known to divers and support personnel?	☐ Yes ☐ No (Stop Work) ☐ N/A
16.	What is the address and phone number to the closest hospital to the dive site?	
17.	What is the address and phone number to the nearest recompression chamber to the site?	
18.	Authorized Permit Approver (PRINT):	Authorized Permit Approver (SIGNATURE):
19.	Chevron PM acceptance? *Must be accepted by Chevron PM or pre-approved company representative*	☐ E-mail ☐ Fax ☐ Phone (w/ documentation) ☐ No

WORKING AT ELEVATION

ersons working at or above an elevation of 6 feet must have fall protection. OSHA 29 CFR 1926 Subpart M governs fall protection.

1.	Do employees know appropriate anchorage points for each task that requires a fall-arrest or restraint system?	☐ Yes ☐ No (Stop Work) ☐ N/A
2.	Are all anchorage points stable, substantial and have sufficient strength to withstand twice the potential impact energy of the free-fall?	☐ Yes ☐ No (Stop Work) ☐ N/A
3.	Are all anchorage points for body harnesses located at shoulder height?	☐ Yes ☐ No (Stop Work) ☐ N/A
4.	Are anchorage points for self-retracting lifeline systems located overhead?	☐ Yes ☐ No (Stop Work) ☐ N/A
5.	If the lifeline, lanyard, or self-retracting lifeline is not permanently attached to an anchorage point at the elevated work area, is the first worker up or the last worker down protected while climbing and traversing?	☐ Yes ☐ No (Stop Work) ☐ N/A
6.	Does the lifeline have a minimum breaking strength of 5,000 pounds?	☐ Yes ☐ No (Stop Work) ☐ N/A
7.	Is the lifeline free from abrasive or cutting edges?	☐ Yes ☐ No (Stop Work) ☐ N/A
8.	Does the system provide fall protection as the worker connects to and releases from the lifeline?	☐ Yes ☐ No (Stop Work) ☐ N/A
9.	Is the lifeline arranged so workers never have to hold it for balance?	☐ Yes ☐ No (Stop Work) ☐ N/A
10.	Is the vertical segment integrated with the horizontal segment to provide continuous fall protection?	☐ Yes ☐ No (Stop Work) ☐ N/A
11.	Has equipment been inspected thoroughly for signs of abrasion, fraying, punctures, etc. prior to use?	☐ Yes ☐ No (Stop Work) ☐ N/A
12.	Is the fall arrester equipment with a locking mechanism that prevents unintentional opening of the device and subsequent disengagement from the lifeline?	☐ Yes ☐ No (Stop Work) ☐ N/A
13.	Is the lanyard length as short as necessary and in no case greater than 6 feet?	☐ Yes ☐ No (Stop Work) ☐ N/A
14.	Does the lanyard have a shock absorbing feature to limit the arresting forces to 500-600 pounds?	☐ Yes ☐ No (Stop Work) ☐ N/A
15.	Has the free-fall distance been considered, so that a worker will not strike a lower surface or object before the fall is arrested?	☐ Yes ☐ No (Stop Work) ☐ N/A
16.	Have pendulum-swing fall hazards been eliminated?	☐ Yes ☐ No (Stop Work) ☐ N/A
17.	Have safe methods to retrieve fallen workers been planned?	☐ Yes ☐ No (Stop Work) ☐ N/A
18.	Have harnesses been inspected ON THE USER by Health & Safety Supervisor or Authorized Permit Writer?	☐ Yes ☐ No (Stop Work) ☐ N/A
19.	Have ladders and scaffolds been inspected prior to use?	☐ Yes ☐ No (Stop Work) ☐ N/A
20.	Authorized Permit Approver (PRINT):	Authorized Permit Approver (SIGNATURE):
21.	Chevron PM acceptance? *Must be accepted by Chevron PM or pre-approved company representative.*	☐ E-mail ☐ Fax ☐ Phone (w/ documentation) ☐ No

HIGH RISK WORK PERMIT

BOATING OR WORKING NEAR OPEN WATER

1.	Does boat have current registration on board?	☐ Yes ☐ No (Stop Work) ☐ N/A
2.	Does boat operator have appropriate training (US Coast Guard Boating Safety Course or equivalent)?	☐ Yes ☐ No (Stop Work) ☐ N/A
3.	Have all passengers been acquainted with use and location of safety equipment, radio, scope of work, etc.?	☐ Yes ☐ No (Stop Work) ☐ N/A
.	Have emergency docking location(s) been assessed by boat operator?	☐ Yes ☐ No (Stop Work) ☐ N/A
5.	Are properly fitted personal flotation devices (PFDs) available for every boat passenger?	☐ Yes ☐ No (Stop Work) ☐ N/A
6.	Have PFDs been inspected for tears/holes, discolored or weakened material, insecure straps, zippers, buckles or labels that are no longer readable?	☐ Yes ☐ No (Stop Work) ☐ N/A
7.	Is a throw ring (Class IV PFD) on the vessel?	☐ Yes ☐ No (Stop Work) ☐ N/A
8.	If sampling on or near water below 50° F, will cold water immersion suits be worn by all passengers?	☐ Yes ☐ No (Stop Work) ☐ N/A
9.	Is a horn available capable of producing a 4-second blast audible for at least ½ mile?	☐ Yes ☐ No (Stop Work) ☐ N/A
10.	If using a portable air horn as the primary audible device is a second can of air or an alternative audible device available?	☐ Yes ☐ No (Stop Work) ☐ N/A
11.	Are all navigational AND instrument lights in working order?	☐ Yes ☐ No (Stop Work) ☐ N/A
12.	Are visual signaling devices (flares, days signals, etc.) stored in a dry location and available at all times?	☐ Yes ☐ No (Stop Work) ☐ N/A
13.	Is there a plug and material for bailing water from the boat?	☐ Yes ☐ No (Stop Work) ☐ N/A
14.	Is an additional propulsion device (oar, paddles, smaller motor) in the boat?	☐ Yes ☐ No (Stop Work) ☐ N/A
15.	Is there a US Coast Guard-approved marine fire extinguisher, a stocked first aid kit and batteries on board?	☐ Yes ☐ No (Stop Work) ☐ N/A
16.	Have the fuel, oil, and battery levels been checked?	☐ Yes ☐ No (Stop Work) ☐ N/A
17.	Is the weather forecast favorable for boating and is a weather band radio operational?	☐ Yes ☐ No (Stop Work) ☐ N/A
18.	Is an anchor and suitable length of anchor line available on the boat?	☐ Yes ☐ No (Stop Work) ☐ N/A
19.	Are cell phones and a marine radio available and operational?	☐ Yes ☐ No (Stop Work) ☐ N/A
20.	Has a float plan been prepared for the boating activities and has it been given to on-shore personnel?	☐ Yes ☐ No (Stop Work) ☐ N/A
21.	Are PFDs available for every employee working near or above open water?	☐ Yes ☐ No (Stop Work) ☐ N/A
22.	If working near or above open water does the JSA adequately address "fall in" hazards?	☐ Yes ☐ No (Stop Work) ☐ N/A
23.	Has a rescue plan been added to the JSA for "fall in" emergencies?	☐ Yes ☐ No (Stop Work) ☐ N/A

"I have assessed the risks posed by work activities planned on or near the water and have taken steps to mitigate those risks. If working on a boat I will familiarize myself with federal, state and local boating regulations and/or International Inland Navigation Rules. I will wear a US Coast Guard-approved personal flotation device AT ALL TIMES when I am in a boat or working near or above water."

24.	Name of Boaters (PRINT)	Name of Boaters (SIGNATURE)
25a.	Any boat passengers have allergies/ailments/illnesses?	25b. ☐ Yes (If YES, provide details below: ☐ No
26.	Authorized Permit Approver (PRINT):	Authorized Permit Approver (SIGNATURE):
27.	Chevron PM acceptance? *Must be accepted by Chevron PM or pre-approved company representative*	☐ E-mail ☐ Fax ☐ Phone (w/ documentation) ☐ No

DEMOLITION, REMOVAL OF PIPELINES AND BURIED STRUCTURES

Complete the EXCAVATION / TRENCHING / DRILLING / OVERHEAD CLEARANCE portion of this permit before proceeding below.

1.	Have necessary federal, state and local permits been obtained?	☐ Yes (Stop Work) ☐ No ☐ N/A
2.	Has the Excavation / Trenching portion of this permit been completed?	☐ Yes ☐ No ☐ N/A
3.	Have storage tanks and pipelines been identified on drawings?	☐ Yes ☐ No ☐ N/A
4.	Have underground installations been identified using ground penetrating radar?	☐ Yes ☐ No ☐ N/A
5.	Have tanks and pipelines been emptied and filled with inert material prior to removal?	☐ Yes ☐ No ☐ N/A
6.	Will there be steps to prevent spills or releases?	☐ Yes (Stop Work) ☐ No ☐ N/A
7.	Are there adequate materials for containment and cleanup of spills or releases?	☐ Yes (Stop Work) ☐ No ☐ N/A
8.	Has a debris pile been selected at a safe location away from the immediate work area?	☐ Yes (Stop Work) ☐ No ☐ N/A
9.	Have fire department personnel been notified of today's work activities?	☐ Yes ☐ No ☐ N/A
10.	Is there a need to decontaminate debris prior to removal from site?	☐ Yes ☐ No ☐ N/A
11.	Have plans been made for scrap material to be removed from the site expeditiously?	☐ Yes ☐ No ☐ N/A
12.	Authorized Permit Approver (PRINT):	Authorized Permit Approver (SIGNATURE):
3.	Chevron PM acceptance? *Must be accepted by Chevron PM or pre-approved company representative.* ***Exception: O&M performed AT LEAST Quarterly.***	☐ E-mail ☐ Fax ☐ Phone (w/ documentation) ☐ No

ARCADIS
Infrastructure, environment, buildings

Air Monitoring Log

Project:

Date:

Monitoring Instruments:

Air Monitor:

Activity:

Level of Protection:

Time	Location	Instrument Reading	Comments

Employee Signature Form

I certify that I have read, understand, and will abide by the safety requirements outlined in this HASP.

Printed Name	Signature	Date

Subcontractor Acknowledgement: Receipt of HASP Signature Form

ARCADIS claims no responsibility for the use of this HASP by others although subcontractors working at the site may use this HASP as a guidance document. In any event, ARCADIS does not guarantee the health and/or safety of any person entering this site. Strict adherence to the health and safety guidelines provided herein will reduce, but not eliminate, the potential for injury at this site. To this end, health and safety becomes the inherent responsibility of personnel working at the site.

Printed Name	Company	Signature	Date

Visitor Acknowledgement and Acceptance of HASP Signature Form

By signing below, I waive, release and discharge the owner of the site and ARCADIS and their employees from any future claims for bodily and personal injuries which may result from my presence at, entering, or leaving the site and in any way arising from or related to any and all known and unknown conditions on the site.

Name	Company	Reason for Visit	Date/Time On Site	Date/Time Off Site

Hazardous Materials Transportation Form

	Vehicle (place X in box)	Type (pick-up, car, box truck, etc.)
Personal		
Rental		
ARCADIS owned/leased		
Government owned		
Trailer		

Materials Transported	Quantity	Storage/Transport Container

List Trained Drivers:

____

____

Hazardous Materials Shipment Form

Material Description and Proper Shipping Name (per DOT or IATA)	Shipment Quantity	DOT Hazard Classification	Shipment Method (air/ground)

List Shipper (i.e., who we are offering the shipment to):

____

____

List Trained Employee(s):

____

____

Name/Company:____Date:______________

Health and Safety Plan Written Test

Please answer all the following questions.

1) What is the address of the site?

2) If working conditions or behaviors are considered unsafe, what is your responsibility to do?

3) Where is the HASP located?

4) Name 3 tools used in the LPS Program.

5) Where is the support zone?

6) Where is the exclusion zone?

7) Where are the emergency equipment kept (e.g. fire extinguishers, first aid kits, etc)?

8) What are the components of the Loss Performance Self Assessment?

9) Have you read the Journey Management Plan?

10) What section summarizes emergency procedures and presents directions to the nearest hospitals and emergency telephone numbers?

11) What LPS tool should you use every time before you perform a new activity?

12) If a near miss occurs, who should you notify…and when?

13) After leaving the exclusion zone, before eating, drinking, smoking, or using the toilet facilities, what must you do?

14) Who is the ARCADIS Site Health and Safety Supervisor?

15) Where is the evacuation assembly point?

Name/Company:____Date:______________

Visitor Written Test

Please answer all the following questions.

1) What is the address of the site?

2) Who is the ARCADIS Site Health and Safety Supervisor?

3) Who is responsible for escorting you at all times while on site?

4) In the event of an on-site emergency where should everyone meet?

5) Where is the nearest phone located?

6) What is the local emergency number?

7) Where is the location of first aid equipment and fire extinguisher?

8) What allergies or medical conditions do you have that may require attention in the event of an emergency?

CHEVRON Environmental Management Company	**Management of Change Report Form** **Attach additional pages if needed.**	
INTRODUCTION	**Change Owner**:	**Affected Location(s)/Operation(s):**
MOC #:	**Type of Change:**	**Date Submitted:**
Reason for Change:		
Describe the Change:		
Public Affairs	**Certain changes require an update of the PGPA screeening questionnaire. If required, this task should be listed below.**	
TASKS	**List the work tasks required to complete the change. List the responsible Subject Matter Experts and the due date.**	

MOC Tasks:	**Subject Matter Experts**	**Due Date**	**Completed**
1.			
2.			
3.			
4.			
5.			

RISKS	**Identify associated risks and how they are to be mitigated. List the responsible party and the due date.**

MOC Risks	**Responsible**	**Due Date**	**Completed**
1.			
2.			
3.			
4.			

COMMUNICATION	**Who needs to know about the change and how will you communicate the change to them?**

APPROVALS	
Initial MOC Plan Authorized by:	**Date Authorized**:
Change Approved by:	**Date Approved**:

Management of Change Form Instructions

INTRODUCTION

Change Owner:	List who is preparing the MOC Form. This is the person who has identified the need for the change.
Affected Location(s)/Operation(s):	List which locations or operations impacted by the change.
MOC #:	The Change Owner assigns the MOC number. The format for the number is: Sequential number The Business Unit The location The last two digits of the year. For example, 001-Abandonment-New Orleans-05 indicates it is the first MOC for the Abandonment Business Unit, New Orleans office in 2005.
Type of Change:	Select the type of change from the options: • Operational, • Organizational, or • Business Process.
Date Submitted:	Enter the date of the MOC.
Reason for Change	Select the reason for the change from the options: • Safety/risk reduction • Cost savings • Regulatory requirement • Legal requirement • Customer requirement • Business plan • Process improvement • Operational excellence • Capital stewardship • Profitable growth

Public Affairs

Review the Public Affairs Identification Process to determine if updated screening questionnaire is required.	Questionnaire data should be reviewed and updated when the following occurs (not necessarily a complete list): • Change in surrounding land use(s) • Change in use of the property • Sale of the property • Plume/contamination migrates off-site • A large excavation for the project is planned (when was not planned or expected previously) • Project/site receives media attention (when had none previously) • Public complaints made about the project/site (when were none previously) • Litigation/claims filed or threatened against the company regarding the project/site (when were

	none previously) • New development or buildings being built, or have been built, over the plume/contamination • Announcement of new plans for development or redevelopment of the property • Discovery that a nearby municipal or private water supply, or surface water body has been contaminated or possibly contaminated • New information suggests that there is potential for vapor intrusion into a non-service station structure • New information indicates the nearby presence of community or environmental receptors or special/valued sites.
TASKS	
List the work tasks required to complete the change.	The Tasks section is used to list the specific actions required to implement the change. These are the key activities that need to occur during the change. Keep the tasks parallel in scope. Don't microplan one part and gloss over the tasks in another part of the change. Each task should be discrete and assignable to someone. Review the task list to ensure that all the major steps of your change are included. This section could be quite lengthy, depending upon the type of change and its scope.
List the Subject Matter Experts, the due date and finally, record the date completed.	Each task needs to be assigned to someone. The Change Owner defines the needed subject matter experts. Depending on the complexity and type of change, the Authorizing Supervisor may add Subject Matter Experts. This section contains the names of the personnel or departments that have been assigned to each task. The Subject Matter Experts named for each task should be the person responsible for completing the task or review. Establish a date for the completion of the task. Record the actual completion date when it occurs.
RISKS	
List the risks associated with the change. These may be risks during the change as well as risks that may exist following the change.	These are the risks to people, equipment, products, the business, or the environment during and after the change. It is not intended to take the place of a JSA or a PHA.
List the responsible party, the due date and finally, record the date completed.	Each risk needs to be assigned to someone who will be resonsible for mitigating the risk. In addition, establish a date for the completion of the risk mitigation.
COMMUNICATION	
Who needs to know about the change and how will you communicate the change to them?	Other organizations can be affected by a change; therefore, good communication is essential to reduce losses due to fines, accidents, or other incidents associated with change. Other Chevron entities, Contractors, and government agencies are just a few of the organizations to consider contacting and communicating with when implementing a change. In this section, you identify the groups impacted by

	this change and plan how you will communicate the change to those groups. You can attach existing communication plans, define necessary communication tasks, assign individuals to those tasks, and determine target completion dates. Implementing the Change involves assuring that everyone who needs to know about the Change is informed. This might be as simple as distributing an e-mail or it could be formal training or issuing revised procedures. Identify who needs to know about the Change and list how you will communicate the Change to them. If in doubt, err on the side of telling too many people instead of assuming that an individual or group does not need to know about the change.
APPROVALS	
Initial Authorization	The Authorizing Supervisor must approve the MOC Plan prior to the Change Owner forwarding it to any subject matter experts.
Final Approval	After all MOC tasks and reviews have been performed, the change can be considered complete and the Authorizing Supervisor can sign-off on the MOC.
Attach all work documents for the project to the MOC Form. Many changes will include other work documents in addition to the MOC Form. For example, permits, drawings, project schedules, and letters might be produced for a change. These should be attached to the MOC Form.	
Document Storage - The MOC Form and any attachment to the Form are to be filed in the shared directory identified by the Business Unit. The MOC Log will be updated and filed in the same directory. The files may be maintained by the individual Business Units in either the regional office locations or the project locations, as determined appropriate by the Business Unit.	
Document Retention - The MOC documents shall be maintained for three years, or for the life of facility affected by the change, whichever is longer.	

Management of Change Tracking Form

MOC #	Date Submitted	Description of Change	Date of Completion

Exhibit 3 - Utilities and Structures Checklist

Project:	**Project Number:**
Site Location:	**Date:**

Instructions: This checklist will be used as a safety measure to insure that all underground utility lines, other underground structures as well as above ground utilities are clearly marked out and identified in the area selected for boring or excavation. **DRILLING, EXCAVATION, OR ANY TYPE OF GROUND INTRUSIVE WORK MAY NOT PROCEED UNTIL LINES ARE MARKED AND THIS CHECKLIST HAS BEEN COMPLETED.**

Pre-Field Work Requirements		
Was the state one-call notified with the required advanced notice (usually 48 to 72 hours) (or 811 Nation-wide number)	YES	NO
State one-call confirmation number		
List utility companies who do not participate in the state one call program. Were they contacted directly?		
What additional lines of evidence are used for utility clearance?		
Was a plot plan showing site features and subsurface utilities provided by the PM/TM?	YES	NO
Subgrade Utility Line Location		
Where is the gas line located?		
Where is the gas meter located on the site building(s)?		
Are the electric lines subsurface or overhead? Where are they located?		
Where is electric meter located on the site building(s)?		

Where are the telephone/cable lines located? Are there any overhead lines?	
Where do these lines enter the site building(s)?	
Where are the water lines located?	
Does the site occupant use water (bathrooms, industrial uses, fire suppression, etc.)? If so where do the water lines enter the building for these purposes?	
Are there small manholes/vault covers indicating water lines? If so, where?	
Was the local municipality contacted to mark sanitary lines?	
Where are the sanitary lines located?	
Where might the sanitary lines enter the building? (i.e. what side of the building are the bathrooms, kitchens, water treatment plant, etc?)	
Where are the storm sewer lines located?	
Are there storm sewer inlets located on the property? Check inlets for direction of subsurface lines.	
Are there any gutters directing storm water to the subsurface? Evaluate for direction of lines.	
Underground Storage Tank Sites	
Where are the USTs located? How many USTs are at the site (very number of USTs by counting fill ports and vent lines)?	
Where do the vent lines run?	
Where does the piping run? (Evaluate the path between USTs to dispenser islands).	
Where are the sub-surface electrical lines located which feed power to the UST system?	
General Underground Utility Location Signs	
Are there any cracks resembling straight lines that may indicate the settling of utility lines?	

Are there any patched areas where subsurface repairs may have been conducted?	
Are there any manhole covers or valve boxes that are not associated with marked lines?	
Above ground Utility Line Location	
Are there overhead power lines? If, so where are they located?	
What is the voltage of the overhead power lines?	
Are there any above ground structures (utilities, piping, etc.) that are used by the client? If so, are they located proximal to the work area?	
Do these lines need controlled (locked out) or protected prior to starting work?	
Interviews: Site Owners/Occupants MUST be interviewed for location of private utility lines at the site (if practicable) before start of work	
Name of Owner/Occupant.	
How is this person affiliated with the Site?	
Who interviewed Owner/Occupant?	
Date of Interview	
Specific comments that should be noted from the interview:	

NOTE: If any subsurface utilities listed above are not located, do not proceed with subsurface activities. Contact PM/TM immediately.

Name and signature of person who conducted utility line checklist

Name (print) **Signature** **Date**

CHEVRON-SPECIFIC POLICIES

TABLE OF CONTENTS

I. Excavation Safety and Borehole Clearance 2

II. Verification and Validation 2

III. One Team Incident Prevention System (OTIPS) 2

IV. Photographs 2

V. Operational Control 2

VI. Traffic Control 2

- A. Requirement for Two-Person Crew 3
- B. Level 1 3
- C. Level 2 4
- D. Level 3 4
- E. Level 4 4

VII. Waste Management 4

VIII. Jewelry, Hair and Loose Clothing 5

IX. Training 5

- A. General 5
- B. Permit-to-Work Approvers 5
- C. Management and Supervisors 6

I. EXCAVATION SAFETY AND BOREHOLE CLEARANCE

The overall goal of this program is to provide a procedural outline for utility clearance that can be applied to all facility investigations, and to minimize, if not eliminate, potential for damage caused by hitting utilities during intrusive operations. When applicable, crews must hand clear subsurface utilities to a depth of 8 feet below the ground surface.

II. VERIFICATION AND VALIDATION

Safety processes are verified and validated through use of OTIPS and through Level III safety audits.

III. ONE TEAM INCIDENT PREVENTION SYSTEM (OTIPS)

CEMC utilizes a behavior-based safety system database known as One Team Incident Prevention System (OTIPS). ARCADIS' LPS database is similar to CEMC's OTIPS, in that both systems use a set of tools to systematically prevent incidents or, at a minimum, reduce their severity. ARCADIS employees are required to have ARCADIS-led LPS training or CEMC OTIPS training prior to working on a CEMC job site.

The OTIPS site may be accessed at:

https://project.bbl-inc.com/IPS/index.cfm?TabID=2

IV. PHOTOGRAPHS

The taking of photographs is not permitted while onsite without approval from the client.

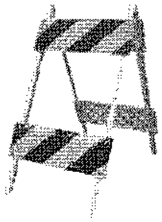
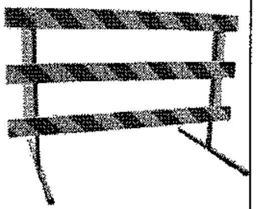
V. OPERATIONAL CONTROL

Operational control determines whether CEMC personnel or ARCADIS personnel are responsible for managing CEMC's Permit to Work process. Section 12.1 of the E-HASP provides information relating to CEMC operational control at this facility. When CEMC is the permit issuing authority at the project site, ARCADIS personnel are expected to assist with form completion and meet all of the hazard assessment and pre-job work requirements at the site. When CEMC does NOT have operational control at the facility, ARCADIS personnel are required to complete the General Permit-to-Work and High-Risk Permit-to-Work, when applicable. These forms are tailored for CEMC projects.

VI. TRAFFIC CONTROL

A JMP must be generated for each facility and updated in accordance with the JMP guidelines. The JMP incorporates a TCP to be implemented at the subject facility; this includes the designation of pedestrian and motor vehicle areas at the site. The HSS may make modifications to the TCP, as needed, to address potential changes is site conditions. A detailed TCP, including a site map depicting traffic control measures is included with the JMP in Appendix H of the E-HASP.

All personnel working at the project site must wear Class 2 or higher high-visibility vests. The Class identification is located on the garment tag. Class 3 vests are required for any night-time work or work where traffic flows at over 50 mph. If drums, cones or tubular markers are used to channelize pedestrians, the devices shall be located so that there are no gaps between the bases in order to create a continuous bottom. The cap height of each individual drum, cone or tubular marker shall be no less than 36 inches to be detectable to people who use walking canes. Type II or Type III Barricades are acceptable for use on Chevron project sites.

Type 1 Barricade	
Type 2 Barricade	
Type 3 Barricade	

When establishing work zones in parking lots or other areas with the potential for vehicle traffic, high-visibility traffic cones (with one or more flags) or delineators with a height of 48-inches must be utilized.

A. REQUIREMENT FOR TWO-PERSON CREW

If there are any questions regarding the number of personnel required to work safely at the site, a two-person crew must be dispatched for the first site visit. Subsequent review with these employees will determine if the site remains a two-person job. A two-person crew (minimum) is required if the location requires traffic to be redirected into another lane, a public traffic lane is temporarily closed, work is completed along the shoulder of a heavily trafficked roadway or it is deemed otherwise necessary for the safety of the field crew. Consider the buddy system also for work in areas where hybrid or electric vehicles are popular since they can't be heard at slow speeds.

B. LEVEL 1

Use of Level 1 protection may be acceptable for routine tasks of very short duration (less than 10 minutes in one area) where a worker can stay aware of traffic in the vicinity, such as during initial well gauging or surveying.

Maintain a maximum distance of 4-feet between cones and always leave an exit point from the work zone. If possible, use vehicle with flashers activated to provide visual warning and protection from traffic. Use the buddy system or watchperson when conditions warrant.

C. LEVEL 2

If tasks of any duration are such that the worker cannot remain alert regarding traffic, traffic control should be elevated to Level 2. Level 2 traffic control is also necessary when working more than 10 minutes in one location, where Level 3 control is not warranted.

Use barrier tape on delineators with flags. Use field vehicles with flashing yellow light or vehicle hazard lights and/or other lane control devices. Barricades may also be used in conjunction with tape to improve warning because of the noise they make if overturned. The buddy system should be used in areas where the worker cannot give undivided attention to traffic when exposed to traffic hazards. Level 2 should be used when temporarily securing an area for such activities as groundwater sampling, hand digging or temporarily isolating an area for storage or moving equipment/supplies.

D. LEVEL 3

High task loads that distract attention from vehicle traffic (including noise), work that is performed in low light conditions or when there is a closure of access from public right-of-way to a work site (e.g. station entrance is closed) warrants Level 3 traffic control.

Use plastic security fencing and/or Type 2 or Type 3 barricades. A flashing yellow light on the vehicle may be used for additional visibility. Be cognizant of blocking of pump island or any other facility concerns. Use Level 3 traffic control when where heavy equipment is or power tools in areas near electrically energized or rotating equipment. Use Level 3 as protection for workers or the public near slow-moving traffic or with high traffic volume, in driveways, around tripping hazards and to control third-party foot traffic. If there is no third-party traffic potential, Level 3 is NOT required.

E. LEVEL 4

Level 4 traffic control is warranted when work is being performed in a public right-of-way. Traffic control in Level 4 often requires oversight by a licensed traffic controller. Traffic control requirements may vary depending upon the authority having jurisdiction over the roadway, city, county, state or country.

VII. WASTE MANAGEMENT

The handling and disposal of investigation-derived waste (IDW) material generated at the site during investigative activities will be managed in accordance with ARCADIS Standard Operating Procedure (SOP): Handling and Storing Investigation-Derived Waste. The SOP will be used in accordance with state, local, and Chevron guidelines. Additionally, the site-specific requirement for the COCs will be taken into account for the handling of the IDW at this facility. The Investigative-Derived Waste SOP is available in Appendix I.

VIII. JEWELRY, HAIR AND LOOSE CLOTHING

Jewelry, hair and loose clothing can easily get caught in moving machinery, which may result in serious bodily injury or death. Always remove all jewelry before beginning work and make certain clothing and hair cannot become entangled in moving equipment.

Jewelry should not be worn if it could compromise the integrity of hand protection by tearing or puncturing protective gloves.

IX. TRAINING

A. GENERAL

All onsite project personnel who work in areas where they may be exposed to site contaminants must be receive the following training:

- OSHA HAZWOPER (29 CFR 1910.120). This certification requires an annual 8-hour refresher, a portion of which must be job-specific. Personnel who completed their initial training more than 12 months prior to the start of the project must have completed an 8-hour refresher course within the past 12 months.

- Loss Prevention System. This does not require an annual refresher; however, personnel are expected to remain proficient with the system by participating in JLAs, LPOs and IINLIs.

- Smith System® Defensive Driving – Hands-On. Personnel are expected to complete this course every 3 years. A commentary drive must accompany this training program.

B. PERMIT-TO-WORK APPROVERS

In addition the training specified in Section 4.1, Permit-to-Work Approvers must receive OSHA Supervisor training. Additionally, Permit-to-Work Approvers must be familiar with the ARCADIS/CEMC Permit-to-Work Procedure.

Authorized PTW Approvers are designated by the PIC or Program Manager based on appropriate experience and training including:

- OSHA HAZWOPER (40-hour initial and current 8-hour refresher).

- OSHA HAZWOPER Supervisor Training.

- Loss Prevention System (including OE Tenets and Stop Work Authority).

- At least six months in current job (not a Short Service Employee).

- Demonstrated understanding of ARCADIS PTW procedure.

Chevron EMC Retail & Terminal Business Unit

BOREHOLE CLEARANCE REVIEW
(Specific for service station assessments)

Chevron Site #: ______________________ Project #: _______________________________

Borehole #s Reviewed: ________________________________ Date: _____________________

Clearance Inspected by: _______________________ Reviewed by _______________________
(Contractor/Consultant Rep.) (Contractor/Consultant PM Initial)

Yes	No		
☐	☐	1.	Is a scaled site plan showing the proposed borehole locations attached to this form?
☐	☐	2.	Are all of the proposed borehole locations at least 5 feet from any subsurface utilities (including product lines) shown on ChevronTexaco's building plans?
☐	☐	3.	Are all of the proposed borehole locations at least 7 feet from the pad surrounding the underground storage tanks (USTs) shown on ChevronTexaco's building plans?
☐	☐	4.	Are all of the proposed borehole locations at least 5 feet from any subsurface utilities shown on public right-of-way street improvement plans? PM to check here ☐ if applicable to this job.
☐	☐	5.	Does the station manager have any knowledge of any subsurface utilities within 5 feet of the proposed borehole locations? (Review locations with the station manager).
☐	☐	6.	Are all of the proposed borehole locations at least 5 feet from any subsurface utilities identified during a geophysical survey? PM to check here ☐ if applicable to this job.
☐	☐	7.	Have all underground service alert providers notified by USA marked out their facilities in the vicinity of the borehole locations or otherwise notified EMC'S Contractor/Consultant that they do not have any facilities near the proposed borehole locations?
☐	☐	8.	Do any of the proposed borehole locations lie on a line connecting two similar manhole covers?
☐	☐	9.	Do any of the proposed borehole locations lie on a line perpendicular to the street from the water, gas, and electrical meter?
☐	☐	10.	Has the pavement in the vicinity of any of the proposed borehole locations subsided or does it give the appearance it may be covering a former trench?
☐	☐	11.	Have you carefully cleared the hole (using an air knife, hand auger, or other mechanical methods) to a minimum depth of 8 feet below grade and greater than the outer diameter of the drilling tools you will be using before using the drill rig?
☐	☐	12.	Were you able to carefully lower the drilling tools into the previously cleared hole without resistance to a minimum of 8 feet below grade?
☐	☐	13.	Does the soil you encountered in the cleared hole consist of clean gravel, clean sand, aggregate base (gravelly sand with ~10% fines), or non-native looking material?

Questions 1 thru 10 must be answered prior to mobilizing a drilling rig to the site. Questions 11 thru 13 should be answered prior to drilling by the field staff. DO NOT DRILL, if you answered NO to questions 1, 2, 3, 4, 6, 7, 11, or 12 or answered YES to questions 5, 8, 9, 10, or 13. Contact the EMC Contractor/Consultant project manager for instructions prior to drilling and describe the conflict on the back of this form.

ARCADIS
Infrastructure, environment, buildings

Daily / Periodic Excavation Inspection Checklist

Project Name:	**Date / Time:**
Project Number:	**Location:**
Prepared By:	**Project Manager:**

This checklist must be completed for all excavations. It documents that daily and post-event / periodic inspections are conducted.

Soil Classified As: Stable Rock Type A Type B Type C

Soil Classified On: By:

Type of Protective System in Use: Sloping Shoring Other ________________

Description:

Inspection Item	YES	NO	Comments
Is the underground / overhead utilities checklist completed?			
Are underground installations protected from damage?			
Are adequate means of entry / exit available in the excavation?			
If exposed to traffic, are personnel wearing reflective vests?			
Do barriers exist to prevent equipment from rolling into the excavation?			
Was air monitoring conducted prior to and during excavation entry?			
Was the stability of adjacent structures reviewed by a registered P.E.?			
Are spoil piles at least 2 feet from the excavation edge?			
Is fall protection in use near excavations deeper than 6 feet?			
Are work tasks completed remotely if feasible?			
Is a protective system in place and in good repair?			
Is emergency rescue (lifeline / body harness) equipment used due to potential atmospheric hazard?			
Is excavation exposed to vibration?			
Are employees protected from falling / elevated material?			
Is soil classification adequate for current environmental / weather conditions?			
Do portable ladders extend at least 4 feet above the excavation?			
Are portable ladders or ramps secured in place?			
Have all personnel attended safety meeting on excavation hazards?			
Are support systems for adjacent structures in place?			
Is the excavation free from standing water?			
Is water control and diversion of surface runoff adequate?			
Are employees wearing required protective equipment?			

BBL Excavation Competent Person:	**Date/Time:**

PROJECT WORK REQUEST

PROJECT NAME:
STREET ADDRESS:
CITY, STATE:
Facility Number:
Site Contact:
Chevron PM:
Laboratory:
Laboratory SO # :
Date Needed:
Budgeted Hours:
Date Requested:
Project Category: ☐ MT2 ☐ Non-MT2

PROJ. NUMBER:
TASK NUMBER:
Proj. Manager:
Site Safety Officer:
Field Supervisor:
Site Worker:
Site Worker:
Site Worker:
Site Worker:
Site Worker:
Site Worker:
Site Worker:

CHECK THE FOLLOWING BOXES THAT ARE APPLICABLE. BY CHECKING THE BOXES AND SIGNING BELOW, PERSONNEL ARE ASSURING THE TASKS HAVE BEEN COMPLETED PER COMPANY POLICIES AND PROCEDURES.

☐ Reviewed Work Packet
☐ Reviewed HASP
☐ Conducted Vehicle Inspection
☐ Reviewed/Modified JMP
☐ Conducted Tailgate Safety Meeting
☐ Completed PTW

☐ Completed Borehole Clearance Form
☐ Completed Survey Form
☐ Took 15 Min. Break Every 3 Hrs. While Driving
☐ Reviewed/Modifed JSA
☐ Assessed Need for Puncture Resistant Footwear on JSA
☐ Reviewed TCP

☐ Completed Utility Markout
☐ Reviewed OE Tenets
☐ Completed Equipment Rental Form
☐ Conducted SPSA as Needed
☐ Reviewed Stop Work Authority
☐ Photocopied Field Notes

☐ Task 1 Complete	
	Title one of the pages near the end of the yellow field book "Emergency Drills".
	Perform an emergency drill and document date/time near end of field book.
	Emergency drills should be conducted at each site annually.
☐ Task 2 Complete	
☐ Task 3 Complete	
☐ Task 4 Complete	
☐ Task 5 Complete	
☐ Task 6 Complete	

Project Manager **Pre-task** Acceptance
Name:
Date:

Project Manager **Post-task** Acceptance
Name:
Date:

Name:
Work Date:
Hours Billed: ____________
Signature/Date:
Page: 1 of

Generator → Project Manager → Field Supervisor → Project Manager → Document Technician → Central File

☐ Task 7 Complete	
☐ Task 8 Complete	
☐ Task 9 Complete	
☐ Task 10 Complete	
☐ Task 11 Complete	
☐ Task 12 Complete	
☐ Task 13 Complete	
☐ Task 14 Complete	
☐ Task 15 Complete	
☐ Task 16 Complete	
☐ Task 17 Complete	

Name: ______________________________

Work Date: ______________________________

MSDSs

ALCONOX MSDS

Section 1 : MANUFACTURER INFORMATION

Product name: Alconox

Supplier: Same as manufacturer.

Manufacturer: Alconox, Inc.
30 Glenn St.
Suite 309
White Plains, NY 10603.

Manufacturer emergency phone number: 800-255-3924.
813-248-0585 (outside of the United States).

Manufacturer: Alconox, Inc.
30 Glenn St.
Suite 309
White Plains, NY 10603.

Supplier MSDS date: 2009/04/20

D.O.T. Classification: Not regulated.

Section 2 : HAZARDOUS INGREDIENTS

C.A.S.	CONCENTRATION %	Ingredient Name	T.L.V.	LD/50	LC/50
25155-30-0	10-30	SODIUM DODECYLBENZENESULFONATE	NOT AVAILABLE	438 MG/KG RAT ORAL 1330 MG/KG MOUSE ORAL	NOT AVAILABLE
497-19-8	7-13	SODIUM CARBONATE	NOT AVAILABLE	4090 MG/KG RAT ORAL 6600 MG/KG MOUSE ORAL	2300 MG/M3/2H RAT INHALATION 1200 MG/M3/2H MOUSE INHALATION
7722-88-5	10-30	TETRASODIUM PYROPHOSPHATE	5 MG/M3	4000 MG/KG RAT ORAL 2980 MG/KG MOUSE ORAL	NOT AVAILABLE
7758-29-4	10-30	SODIUM PHOSPHATE	NOT AVAILABLE	3120 MG/KG RAT ORAL 3100 MG/KG MOUSE ORAL >4640 MG/KG RABBIT DERMAL	NOT AVAILABLE

Section 2A : ADDITIONAL INGREDIENT INFORMATION

Note: (supplier).
CAS# 497-19-8: LD50 4020 mg/kg - rat oral.
CAS# 7758-29-4: LD50 3100 mg/kg - rat oral.

Section 3 : PHYSICAL / CHEMICAL CHARACTERISTICS

Physical state: Solid

Appearance & odor: Almost odourless.
White granular powder.

Odor threshold (ppm): Not available.

Vapour pressure (mmHg): Not applicable.

Vapour density (air=1): Not applicable.

By weight: Not available.

Evaporation rate (butyl acetate = 1): Not applicable.

Boiling point (°C): Not applicable.

Freezing point (°C): Not applicable.

pH: (1% aqueous solution).
9.5

Specific gravity @ 20 °C: (water = 1).
0.85 - 1.10

Solubility in water (%): 100 - > 10% w/w

Coefficient of water\oil dist.: Not available.

VOC: None

Section 4 : FIRE AND EXPLOSION HAZARD DATA

Flammability: Not flammable.

Conditions of flammability: Surrounding fire.

Extinguishing media: Carbon dioxide, dry chemical, foam.
Water
Water fog.

Special procedures: Self-contained breathing apparatus required.
Firefighters should wear the usual protective gear.

Auto-ignition temperature: Not available.

Flash point (°C), method: None

Lower flammability limit (% vol): Not applicable.

Upper flammability limit (% vol): Not applicable.

Not available.

Sensitivity to mechanical impact: Not applicable.

Hazardous combustion products: Oxides of carbon (COx).
Hydrocarbons.

Rate of burning: Not available.

Explosive power: None

Section 5 : REACTIVITY DATA

Chemical stability:	Stable under normal conditions.
Conditions of instability:	None known.
Hazardous polymerization:	Will not occur.
Incompatible substances:	Strong acids. Strong oxidizers.
Hazardous decomposition products:	See hazardous combustion products.

Section 6 : HEALTH HAZARD DATA

Route of entry:	Skin contact, eye contact, inhalation and ingestion.
Effects of Acute Exposure	
Eye contact:	May cause irritation.
Skin contact:	Prolonged contact may cause irritation.
Inhalation:	Airborne particles may cause irritation.
Ingestion:	May cause vomiting and diarrhea. May cause abdominal pain. May cause gastric distress.
Effects of chronic exposure:	Contains an ingredient which may be corrosive.
LD50 of product, species & route:	> 5000 mg/kg rat oral.
LC50 of product, species & route:	Not available for mixture, see the ingredients section.
Exposure limit of material:	Not available for mixture, see the ingredients section.
Sensitization to product:	Not available.
Carcinogenic effects:	Not listed as a carcinogen.
Reproductive effects:	Not available.
Teratogenicity:	Not available.
Mutagenicity:	Not available.
Synergistic materials:	Not available.
Medical conditions aggravated by exposure:	Not available.
First Aid	
Skin contact:	Remove contaminated clothing. Wash thoroughly with soap and water. Seek medical attention if irritation persists.
Eye contact:	Check for and remove contact lenses. Flush eyes with clear, running water for 15 minutes while holding eyelids open: if irritation persists, consult a physician.
Inhalation:	Remove victim to fresh air. Seek medical attention if symptoms persist.
Ingestion:	Dilute with two glasses of water. Never give anything by mouth to an unconscious person. Do not induce vomiting, seek immediate medical attention.

Section 7 : PRECAUTIONS FOR SAFE HANDLING AND USE

Leak / Spill: Contain the spill.
Recover uncontaminated material for re-use.
Wear appropriate protective equipment.
Contaminated material should be swept or shoveled into appropriate waste container for disposal.

Waste disposal: In accordance with municipal, provincial and federal regulations.

Handling procedures and equipment: Protect against physical damage.
Avoid breathing dust.
Wash thoroughly after handling.
Keep out of reach of children.
Avoid contact with skin, eyes and clothing.
Launder contaminated clothing prior to reuse.

Storage requirements: Keep containers closed when not in use.
Store away from strong acids or oxidizers.
Store in a cool, dry and well ventilated area.

Section 8 : CONTROL MEASURES

Precautionary Measures

Gloves / Type: Neoprene or rubber gloves.

Respiratory / Type: If exposure limit is exceeded, wear a NIOSH approved respirator.

Eye / Type: Safety glasses with side-shields.

Footwear / Type: Safety shoes per local regulations.

Clothing / Type: As required to prevent skin contact.

Other / Type: Eye wash capability should be in close proximity.

Ventilation requirements: Local exhaust at points of emission.

Section 1 - Chemical Product and Company Identification 61

Material Name: Benzene **CAS Number:** 71-43-2
Chemical Formula: C_6H_6
Structural Chemical Formula: C_6H_6
EINECS Number: 200-753-7
ACX Number: X1001488-9
Synonyms: Benzene; BENZENE; (6)ANNULENE; BENZEEN; BENZEN; BENZIN; BENZINE; BENZOL; BENZOL 90; BENZOLE; BENZOLENE; BENZOLO; BICARBURET OF HYDROGEN; CARBON OIL; COAL NAPHTHA; CYCLOHEXATRIENE; EPA PESTICIDE CHEMICAL CODE 008801; FENZEN; MINERAL NAPHTHA; MOTOR BENZOL; NITRATION BENZENE; PHENE; PHENYL HYDRIDE; POLYSTREAM; PYROBENZOL; PYROBENZOLE
General Use: Manufacture of chemicals including styrene, dyes, and many other organic chemicals. Has been used in artificial leather, linoleum, oil cloth, airplane dopes, lacquers; as solvent for waxes, resins, oils etc.
May also be a minor component of gasoline, petrol.
Exposure should be minimized by use in closed systems.
Handling procedures and control measures should be evaluated for exposure before commencement of use in plant operations.

Section 2 - Composition / Information on Ingredients

Name	CAS	%
benzene	71-43-2	99.9

OSHA PEL
TWA: 1 ppm; STEL: 5 ppm.

NIOSH REL
TWA: 0.1 ppm; STEL: 1 ppm.

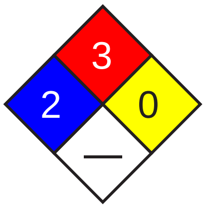
DFG (Germany) MAK
Skin.

ACGIH TLV
TWA: 0.5 ppm; STEL: 2.5 ppm; skin.

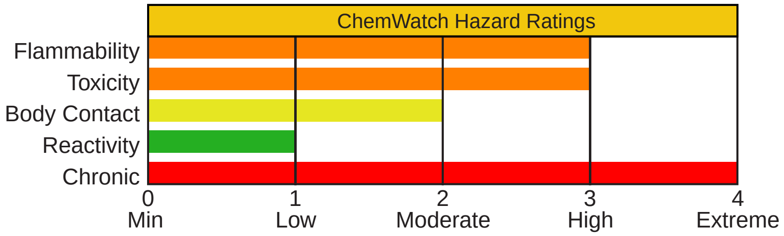
IDLH Level
500 ppm.

EU OEL
TWA: 1 ppm.

Section 3 - Hazards Identification



Fire Diamond



HMIS	
3	Health
3	Flammability
0	Reactivity

ANSI Signal Word
Danger!


Flammable

☆☆☆☆☆ Emergency Overview ☆☆☆☆☆

Colorless liquid; sweet odor. Irritating to eyes/skin/respiratory tract. Toxic. Other Acute Effects: headache, dizziness, drowsiness. Absorbed through skin. Chronic Effects: dermatitis, leukemia, bone marrow damage. Carcinogen. Reproductive effects. Flammable.

Potential Health Effects

Target Organs: blood, central nervous system (CNS), bone marrow, eyes, upper respiratory system, skin
Primary Entry Routes: inhalation, skin contact
Acute Effects
Inhalation: The vapor is discomforting to the upper respiratory tract and lungs and may be harmful if inhaled.

If exposure to highly concentrated solvent atmosphere is prolonged this may lead to narcosis, unconsciousness, even coma and possible death.

Acute effects from inhalation of high concentrations of vapor are pulmonary irritation, including coughing, with nausea; central nervous system depression - characterized by headache and dizziness, increased reaction time, fatigue and loss of coordination.

Inhalation hazard is increased at higher temperatures.

The symptoms of acute exposure to high vapor concentrations include confusion, dizziness, tightening of the leg muscles and pressure over the forehead followed by a period of excitement. If exposure continues the casualty quickly becomes stupefied and lapses into a coma with narcosis.

Effects of inhalation may include nausea, vomiting headache, dizziness, drowsiness, weakness, sometimes preceded by brief periods of exhilaration, or euphoria, irritability, malaise, confusion, ataxia, staggering, weak and rapid pulse, chest pain and tightness with breathlessness, pallor, cyanosis of the lips and fingertips and tinnitus. Severe exposures may produce blurred vision, shallow, rapid breathing, delirium, cardiac arrhythmias, unconsciousness, deep anesthesia, paralysis and coma characterized by motor restlessness, tremors and hyperreflexia (occasionally preceded by convulsions). Polyneuritis and persistent nausea, anorexia, muscular weakness, headache, drowsiness, insomnia and agitation may also occur. Two-three weeks after the exposure, nervous irritability, breathlessness and unsteady gait may still persist; cardiac distress and an unusual dicoloration of the skin may be evident for up to four weeks.

Hemotoxicity is not normally a feature of acute exposures although anemia, thrombocytopenia, petechial hemorrhage, and spontaneous internal bleeding have been reported. Fatal exposures may result from asphyxia, central nervous system depression, cardiac and respiratory failure and circulatory collapse; sudden ventricular fibrillation may also be fatal.

Death may be sudden or may be delayed for 24 hours. Central nervous system, respiratory or hemorrhagic complications may occur up to five days after the exposure and may be lethal; pathological findings include respiratory inflammation with edema, and lung hemorrhage, renal congestion, cerebral edema and extensive petechial hemorrhage in the brain, pleurae, pericardium, urinary tract, mucous membrane and skin.

Exposure to toxic levels has also produced chromosome damage.

Eye: The liquid is highly discomforting to the eyes, may be harmful following absorption and is capable of causing a mild, temporary redness of the conjunctiva (similar to wind-burn), temporary impairment of vision and/or other transient eye damage/ulceration.

The vapor is moderately discomforting to the eyes.

The material may produce severe irritation to the eye causing pronounced inflammation. Repeated or prolonged exposure to irritants may produce conjunctivitis.

Skin: The liquid may produce skin discomfort following prolonged contact.

Defatting and/or drying of the skin may lead to dermatitis. Open cuts, abraded or irritated skin should not be exposed to this material.

Toxic effects may result from skin absorption.

The material may cause skin irritation after prolonged or repeated exposure and may produce a contact dermatitis (nonallergic). This form of dermatitis is often characterized by skin redness (erythema) and swelling (edema) which may progress to vesiculation, scaling and thickening of the epidermis. Histologically there may be intercellular edema of the spongy layer (spongiosis) and intracellular edema of the epidermis.

Ingestion: The liquid is discomforting to the gastrointestinal tract and may be harmful if swallowed.

Ingestion may result in nausea, pain, vomiting. Vomit entering the lungs by aspiration may cause potentially lethal chemical pneumonitis.

Carcinogenicity: NTP - Class 1, Known to be a carcinogen; IARC - Group 1, Carcinogenic to humans; OSHA - Listed as a carcinogen; NIOSH - Listed as carcinogen; ACGIH - Class A2, Suspected human carcinogen; EPA - Class A, Human carcinogen; MAK - Class A1, Capable of inducing malignant tumors as shown by experience with humans.

Chronic Effects: Liquid is an irritant and may cause burning and blistering of skin on prolonged exposure.

Chronic exposure may cause headache, fatigue, loss of appetite and lassitude with incipient blood effects including anemia and blood changes.

Benzene is a myelotoxicant known to suppress bone-marrow cell proliferation and to induce hematologic disorders in humans and animals.

Signs of benzene-induced aplastic anemia include suppression off leukocytes (leukopenia), red cells (anemia), platelets (thromocytopenia) or all three cell types (pancytopenia). Classic symptoms include weakness, purpura, and hemorrhage. The most significant toxic effect is insidious and often irreversible injury to the blood forming tissue. Leukemia may develop.

Section 4 - First Aid Measures

Inhalation: Remove to fresh air.

Lay patient down. Keep warm and rested.

If breathing is shallow or has stopped, ensure clear airway and apply resuscitation. Transport to hospital or doctor.

Eye Contact: Immediately hold the eyes open and flush continuously for at least 15 minutes with fresh running water. Ensure irrigation under eyelids by occasionally lifting the upper and lower lids.

See DOT ERG

Transport to hospital or doctor without delay. Removal of contact lenses after an eye injury should only be undertaken by skilled personnel.

Skin Contact: Immediately remove all contaminated clothing, including footwear (after rinsing with water).
Wash affected areas thoroughly with water (and soap if available).
Seek medical attention in event of irritation.

Ingestion: Contact a Poison Control Center.
Do NOT induce vomiting. Give a glass of water.

After first aid, get appropriate in-plant, paramedic, or community medical support.

Note to Physicians: For acute or short-term repeated exposures to petroleum distillates or related hydrocarbons:

1.Primary threat to life from pure petroleum distillate ingestion and/or inhalation is respiratory failure.

2.Patients should be quickly evaluated for signs of respiratory distress (e.g. cyanosis, tachypnea, intercostal retraction, obtundation) and given oxygen. Patients with inadequate tidal volumes or poor arterial blood gases (pO_2 <50 mm Hg or pCO_2 >50 mm Hg) should be intubated.

3.Arrhythmias complicate some hydrocarbon ingestion and/or inhalation and electrocardiographic evidence of myocardial injury has been reported; intravenous lines and cardiac monitors should be established in obviously symptomatic patients. The lungs excrete inhaled solvents, so that hyperventilation improves clearance.

4.A chest x-ray should be taken immediately after stabilization of breathing and circulation to document aspiration and detect the presence of pneumothorax.

5.Epinephrine (adrenalin) is not recommended for treatment of bronchospasm because of potential myocardial sensitization to catecholamines.
Inhaled cardioselective bronchodilators (e.g. Alupent, Salbutamol) are the preferred agents, with aminophylline a second choice.

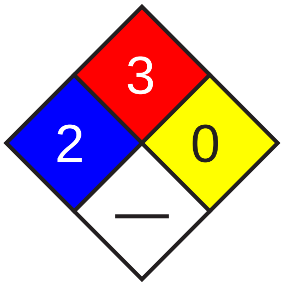
6.Lavage is indicated in patients who require decontamination; ensure use of cuffed endotracheal tube in adult patients.
Consider complete blood count. Evaluate history of exposure.

Section 5 - Fire-Fighting Measures

Flash Point: -11 °C Closed Cup
Autoignition Temperature: 562 °C
LEL: 1.3% v/v
UEL: 7.1% v/v

Extinguishing Media: Foam, dry chemical powder, BCF (where regulations permit), carbon dioxide.
Water spray or fog - Large fires only.

See
DOT
ERG



Fire Diamond

General Fire Hazards/Hazardous Combustion Products: Liquid and vapor are highly flammable.
Severe fire hazard when exposed to heat, flame and/or oxidizers.
Vapor forms an explosive mixture with air.
Severe explosion hazard, in the form of vapor, when exposed to flame or spark. Vapor may travel a considerable distance to source of ignition.
Heating may cause expansion/decomposition with violent rupture of containers.
On combustion, may emit toxic fumes of carbon monoxide (CO).

Fire Incompatibility: Avoid contamination with oxidizing agents i.e. nitrates, oxidizing acids, chlorine bleaches, pool chlorine etc. as ignition may result.

Fire-Fighting Instructions: Contact fire department and tell them location and nature of hazard.
May be violently or explosively reactive. Wear full body protective clothing with breathing apparatus. Prevent, by any means available, spillage from entering drains or waterways. Consider evacuation.
Fight fire from a safe distance, with adequate cover.
If safe, switch off electrical equipment until vapor fire hazard removed.
Use water delivered as a fine spray to control fire and cool adjacent area.
Avoid spraying water onto liquid pools.
Do not approach containers suspected to be hot.
Cool fire-exposed containers with water spray from a protected location.
If safe to do so, remove containers from path of fire.
Equipment should be thoroughly decontaminated after use.

Section 6 - Accidental Release Measures

Small Spills: Remove all ignition sources. Clean up all spills immediately.
Avoid breathing vapors and contact with skin and eyes.
Control personal contact by using protective equipment.
Contain and absorb small quantities with vermiculite or other absorbent material. Wipe up. Collect residues in a flammable waste container.

See
DOT
ERG

Large Spills: Pollutant - contain spillage. Clear area of personnel and move upwind.
Contact fire department and tell them location and nature of hazard.

May be violently or explosively reactive. Wear breathing apparatus plus protective gloves. Prevent, by any means available, spillage from entering drains or waterways. Consider evacuation.
No smoking, bare lights or ignition sources. Increase ventilation.
Stop leak if safe to do so. Water spray or fog may be used to disperse/absorb vapor. Contain spill with sand, earth or vermiculite.
Use only spark-free shovels and explosion proof equipment.
Collect recoverable product into labeled containers for recycling.
Absorb remaining product with sand, earth or vermiculite.
Collect solid residues and seal in labeled drums for disposal.
Wash area and prevent runoff into drains.
If contamination of drains or waterways occurs, advise emergency services.

Regulatory Requirements: Follow applicable OSHA regulations (29 CFR 1910.120).

Section 7 - Handling and Storage

Handling Precautions: Avoid all personal contact, including inhalation.
Wear protective clothing when risk of exposure occurs.
Use in a well-ventilated area. Prevent concentration in hollows and sumps.
DO NOT enter confined spaces until atmosphere has been checked.
Avoid smoking, bare lights, heat or ignition sources.
When handling, DO NOT eat, drink or smoke.
Vapor may ignite on pumping or pouring due to static electricity.
DO NOT use plastic buckets. Ground and secure metal containers when dispensing or pouring product. Use spark-free tools when handling.
Avoid contact with incompatible materials.
Keep containers securely sealed. Avoid physical damage to containers.
Always wash hands with soap and water after handling.
Work clothes should be laundered separately.
Use good occupational work practices. Observe manufacturer's storing and handling recommendations. Atmosphere should be regularly checked against established exposure standards to ensure safe working conditions.

Recommended Storage Methods: Metal can; metal drum. Packing as recommended by manufacturer.
Check all containers are clearly labeled and free from leaks.

Storage Requirements: Store in original containers in approved flame-proof area.
No smoking, bare lights, heat or ignition sources.
DO NOT store in pits, depressions, basements or areas where vapors may be trapped. Keep containers securely sealed.
Store away from incompatible materials in a cool, dry well ventilated area.
Protect containers against physical damage and check regularly for leaks.
Observe manufacturer's storing and handling recommendations.

Regulatory Requirements: Follow applicable OSHA regulations.

Section 8 - Exposure Controls / Personal Protection

Engineering Controls: Use in a well-ventilated area. Local exhaust ventilation usually required.
If risk of overexposure exists, wear NIOSH-approved respirator.
Correct fit is essential to obtain adequate protection. NIOSH-approved self contained breathing apparatus (SCBA) may be required in some situations.
Provide adequate ventilation in warehouse or closed storage area.

Personal Protective Clothing/Equipment:

Eyes: Chemical goggles. Full face shield.
Contact lenses pose a special hazard; soft lenses may absorb irritants and all lenses concentrate them.

Hands/Feet: Nitrile gloves; Neoprene gloves.
Safety footwear.
Do NOT use this product to clean the skin.

Respiratory Protection:
Exposure Range >1 to 10 ppm: Air Purifying, Negative Pressure, Half Mask
Exposure Range >10 to 100 ppm: Air Purifying, Negative Pressure, Full Face
Exposure Range >100 to 1000 ppm: Supplied Air, Constant Flow/Pressure Demand, Full Face
Exposure Range >1000 to unlimited ppm: Self-contained Breathing Apparatus, Pressure Demand, Full Face
Cartridge Color: black
Note: must change cartridge at beginning of each shift

Other: Overalls. Eyewash unit. Barrier cream. Skin cleansing cream.

Glove Selection Index:

PE/EVAL/PE	Best selection
PVA	Best selection
TEFLON	Best selection

VITON	Best selection
VITON/NEOPRENE	Best selection
NITRILE+PVC	Poor to dangerous choice for other than short-term immersion
BUTYL	Poor to dangerous choice for other than short-term immersion
NITRILE	Poor to dangerous choice for other than short-term immersion
NEOPRENE	Poor to dangerous choice for other than short-term immersion
PVC	Poor to dangerous choice for other than short-term immersion
NATURAL RUBBER	Poor to dangerous choice for other than short-term immersion
BUTYL/NEOPRENE	Poor to dangerous choice for other than short-term immersion

Section 9 - Physical and Chemical Properties

Appearance/General Info: Clear, highly flammable liquid; floats on water. Characteristic aromatic odor. Highly volatile. Mixes with alcohol, chloroform, ether, carbon disulfide, carbon tetrachloride, glacial acetic acid, acetone and oils.

Physical State: Liquid
Odor Threshold: 4.68 ppm
Vapor Pressure (kPa): 9.95 at 20 °C
Vapor Density (Air=1): 2.77
Formula Weight: 78.12
Specific Gravity (H_2O=1, at 4 °C): 0.879 at 20 °C
Evaporation Rate: Fast

pH: Not applicable
pH (1% Solution): Not applicable.
Boiling Point: 80.1 °C (176 °F)
Freezing/Melting Point: 5.5 °C (41.9 °F)
Volatile Component (% Vol): 100
Water Solubility: 0.18 g/100 g of water at 25 °C

Section 10 - Stability and Reactivity

Stability/Polymerization/Conditions to Avoid: Product is considered stable. Hazardous polymerization will not occur.
Storage Incompatibilities: Avoid reaction with oxidizing agents.

Section 11 - Toxicological Information

Toxicity
Oral (man) LD_{Lo}: 50 mg/kg
Oral (rat) LD_{50}: 930 mg/kg
Inhalation (rat) LC_{50}: 10000 ppm/7h
Inhalation (human) LC_{Lo}: 2000 ppm/5m
Inhalation (man) TC_{Lo}: 150 ppm/1y - I
Inhalation (human) TC_{Lo}: 100 ppm
Reproductive effector in rats

Irritation
Skin (rabbit): 20 mg/24 hr - mod
Eye (rabbit): 2 mg/24 hr - SEVERE

See *RTECS* CY 1400000, for additional data.

Section 12 - Ecological Information

Environmental Fate: If released to soil, it will be subject to rapid volatilization near the surface and that which does not evaporate will be highly to very highly mobile in the soil and may leach to groundwater. It may be subject to biodegradation based on reported biodegradation of 24% and 47% of the initial 20 ppm in a base-rich para-brownish soil in 1 and 10 weeks, respectively. It may be subject to biodegradation in shallow, aerobic groundwaters, but probably not under anaerobic conditions. If released to water, it will be subject to rapid volatilization; the half-life for evaporation in a wind-wave tank with a moderate wind speed of 7.09 m/sec was 5.23 hours; the estimated half-life for volatilization from a model river one meter deep flowing 1 m/sec with a wind velocity of 3 m/sec is estimated to be 2.7 hours at 20 °C. It will not be expected to significantly adsorb to sediment, bioconcentrate in aquatic organisms or hydrolyze. It may be subject to biodegradation based on a reported biodegradation half-life of 16 days in an aerobic river die-away test. In a marine ecosystem biodegradation occurred in 2 days after an acclimation period of 2 days and 2 weeks in the summer and spring, respectively, whereas no degradation occurred in winter. According to one experiment, it has a half-life of 17 days due to photodegradation which could contribute to removal in situations of cold water, poor nutrients, or other conditions less conductive to microbial degradation. If released to the atmosphere, it will exist predominantly in the vapor phase. Gas-phase will not be subject to direct photolysis but it will react with photochemically produced hydroxyl radicals with a half-life of 13.4 days calculated using an experimental rate constant for the reaction. The reaction time in polluted atmospheres which contain nitrogen oxides or sulfur dioxide is accelerated with the half-life being reported as 4-6 hours. Products of photooxidation include phenol, nitrophenols, nitrobenzene, formic acid, and peroxyacetyl nitrate. It is fairly soluble in water and is removed from the atmosphere in rain.

Ecotoxicity: LC$_{50}$ Clawed toad (3-4 wk after hatching) 190 mg/l/48 hr /Conditions of bioassay not specified; LC$_{50}$ Morone saxatilis (bass) 5.8 to 10.9 ppm/96 hr /Conditions of bioassay not specified; LC$_{50}$ Poecilia reticulata (guppy) 63 ppm/14 days /Conditions of bioassay not specified; LC$_{50}$ Salmo trutta (brown trout yearlings) 12 mg/l/1 hr (static bioassay); LD$_{50}$ Lepomis macrochirus (bluegill sunfish) 20 mg/l/24 to 48 hr /Conditions of bioassay not specified; LC$_{100}$ Tetrahymena pyriformis (ciliate) 12.8 mmole/l/24 hr /Conditions of bioassay not specified; LC$_{50}$ Cancer magister (crab larvae) stage 1, 108 ppm/96 hr /Conditions of bioassay not specified; LC$_{50}$ Crangon franciscorum (shrimp) 20 ppm/96 hr /Conditions of bioassay not specified

Henry's Law Constant: 5.3 x10^{-3}

BCF: eels 3.5

Biochemical Oxygen Demand (BOD): 1.2 lb/lb, 10 days

Octanol/Water Partition Coefficient: log K_{ow} = 2.13

Soil Sorption Partition Coefficient: K_{oc} = woodburn silt loam 31 to 143

Section 13 - Disposal Considerations

Disposal: Consult manufacturer for recycling options and recycle where possible.
Follow applicable federal, state, and local regulations.
Incinerate residue at an approved site.
Recycle containers where possible, or dispose of in an authorized landfill.

Section 14 - Transport Information

DOT Hazardous Materials Table Data (49 CFR 172.101):

Shipping Name and Description: Benzene
ID: UN1114
Hazard Class: 3 - Flammable and combustible liquid
Packing Group: II - Medium Danger
Symbols:
Label Codes: 3 - Flammable Liquid
Special Provisions: IB2, T4, TP1
Packaging: **Exceptions:** 150 **Non-bulk:** 202 **Bulk:** 242
Quantity Limitations: **Passenger aircraft/rail:** 5 L **Cargo aircraft only:** 60 L
Vessel Stowage: **Location:** B **Other:** 40

Section 15 - Regulatory Information

EPA Regulations:
- **RCRA 40 CFR:** Listed U019 Toxic Waste, Ignitable Waste
- **CERCLA 40 CFR 302.4:** Listed per CWA Section 311(b)(4), per RCRA Section 3001, per CWA Section 307(a), per CAA Section 112 10 lb (4.535 kg)
- **SARA 40 CFR 372.65:** Listed
- **SARA EHS 40 CFR 355:** Not listed
- **TSCA:** Listed

Section 16 - Other Information

Disclaimer: Judgments as to the suitability of information herein for the purchaser’s purposes are necessarily the purchaser’s responsibility. Although reasonable care has been taken in the preparation of such information, Genium Group, Inc. extends no warranties, makes no representations, and assumes no responsibility as to the accuracy or suitability of such information for application to the purchaser’s intended purpose or for consequences of its use.

Issue Date: 2006-06

cis-Acetylene Dichloride
DIC4550

Section 1 - Chemical Product and Company Identification 61

Material Name: cis-Acetylene Dichloride **CAS Number:** 156-59-2
Chemical Formula: $C_2H_2Cl_2$
Structural Chemical Formula: CHCl=CHCl
EINECS Number: 205-859-7
ACX Number: X1007815-0
Synonyms: ACETALYNE DICHLORIDE; CIS-ACETYLENE DICHLORIDE; CIS-1,2-DICHLORETHYLENE; CIS-1,2-DICHLOROETHENE; (Z)-1,2-DICHLOROETHYLENE; 1,2-CIS-DICHLOROETHYLENE; CIS-1,2-DICHLOROETHYLENE; CIS-DICHLOROETHYLENE; ETHENE,1,2-DICHLORO-,(Z)-; ETHYLENE,1,2-DICHLORO-,(Z)-
General Use: solvent for waxes, resins, fats, phenol, camphor, acetyl cellulose, organic materials and heat-sensitive substances such as caffeine; in rubber manufacture, as a refrigerant, as an additive to dye and lacquer solutions, in retarding fermentation, in organic synthesis, in medicines, in dye extraction, in chlorination reactions and in the manufacture of artificial pearls; a constituent of perfumes and thermoplastics

Section 2 - Composition / Information on Ingredients

Name	CAS	%
cis-acetylene dichloride	156-59-2	>98

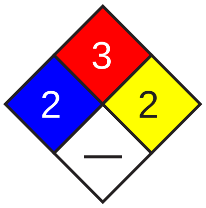
OSHA PEL
TWA: 200 ppm; 790 mg/m^3.

NIOSH REL

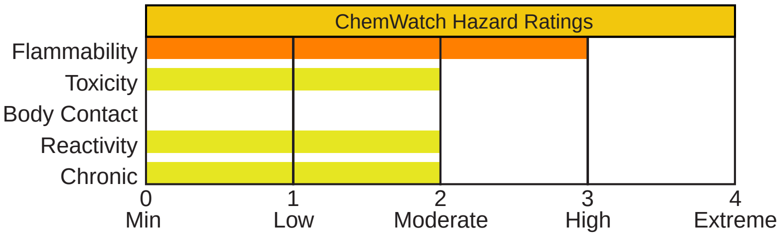
DFG (Germany) MAK
TWA: 200 ppm; PEAK: 400 ppm.

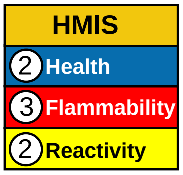
ACGIH TLV
TWA: 200 ppm.

Section 3 - Hazards Identification



Fire Diamond





ANSI Signal Word
Warning!


Flammable

☆☆☆☆☆ Emergency Overview ☆☆☆☆☆

Colorless liquid; sweetish odor. Irritating to eyes/skin/respiratory tract. Harmful. Other Acute Effects: narcotic effect. Flammable.

Potential Health Effects

Target Organs: eyes, skin, respiratory system, nervous system, liver, kidneys
Primary Entry Routes: inhalation of vapor, skin/eye contact

Acute Effects

Inhalation: There is a single report of an industrial poisoning, a fatality caused by the inhalation of a vapor in a small enclosure. Acute intoxication by halogenated aliphatic hydrocarbons appears to take place over two stages. Signs of a reversible narcosis are evident in the first stage and in the second stage signs of injury to organs may become evident, a single organ alone is (almost) never involved. Depression of the central nervous system is the most outstanding effect of most halogenated aliphatic hydrocarbons. Inebriation and excitation, passing into narcosis, is a typical reaction. In severe acute exposures there is always a danger of death from respiratory failure or cardiac arrest due to a tendency to make the heart more susceptible to catecholamines (adrenalin). The most important effects of exposure are narcosis and irritation of the central nervous system. Liver responses may occur after repeated narcotic doses and involves fatty liver degeneration. Vapor exposure may produce central nervous system depression or in milder exposures, nausea, vomiting, weakness, tremor and epigastric cramps. Recovery is usually rapid.

Eye: The vapor when concentrated has pronounced eye irritation effect; this gives some warning of high vapor concentrations. If eye irritation occurs seek to reduce exposure with available control measures, or evacuate area. Exposure to the trans isomer at 2200 ppm caused burning of the eyes, vertigo, nausea. Reversible corneal clouding has been described in exposures to acetylene dichloride.

Skin: The liquid may produce skin discomfort following prolonged contact. Defatting and/ or drying of the skin may lead to dermatitis.

Ingestion: The liquid is discomforting to the gastrointestinal tract and toxic if swallowed. Considered an unlikely route of entry in commercial/industrial environments.

Carcinogenicity: NTP - Not listed; IARC - Not listed; OSHA - Not listed; NIOSH - Not listed; ACGIH - Not listed; EPA - Class D, Not classifiable as to human carcinogenicity; MAK - Not listed.

Chronic Effects: The material may accumulate in the human body and progressively cause tissue damage.

Section 4 - First Aid Measures

See DOT ERG

Inhalation: • If fumes or combustion products are inhaled, remove to fresh air.
- Lay patient down. Keep warm and rested.
- If breathing is shallow or has stopped, ensure clear airway and apply resuscitation, preferably with a demand valve resuscitator, bag-valve mask device, or pocket mask as trained. Perform CPR if necessary.
- Transport to hospital or doctor.

Eye Contact: • Immediately hold the eyes open and flush continuously for at least 15 minutes with fresh running water.
- Ensure complete irrigation of the eye by keeping eyelids apart and away from eye and moving the eyelids by occasionally lifting the upper and lower lids.
- Transport to hospital or doctor without delay.
- Removal of contact lenses after an eye injury should only be undertaken by skilled personnel.

Skin Contact: • Immediately remove all contaminated clothing, including footwear (after rinsing with water).
- Wash affected areas thoroughly with water (and soap if available).
- Seek medical attention in event of irritation.

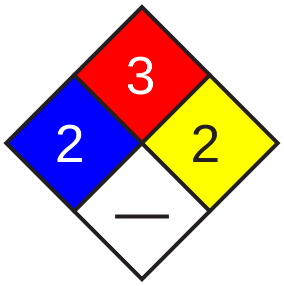
Ingestion: Contact a Poison Control Center. Do NOT induce vomiting. Give a glass of water.

After first aid, get appropriate in-plant, paramedic, or community medical support.

Note to Physicians: Treatment should follow that practiced in carbon tetrachloride exposures:
- Acute exposures to carbon tetrachloride present, initially, with CNS depression followed by hepatic and renal dysfunction.
- Respiratory depression and cardiac dysrhythmias are an immediate threat to life.
- Since a major fraction of absorbed carbon tetrachloride is exhaled in first hour, good tidal volumes should be maintained in severely poisoned patients; hyperventilation may be an additional therapeutic modality.
- Ipecac syrup, lavage, activated charcoal or catharsis may all be used in the first 4 hours.
- Since reactive metabolites may cause hepatorenal toxicity, administration of N-acetyl-L-cysteine may reduce complications. Experience with this therapy is limited.

Section 5 - Fire-Fighting Measures

See DOT ERG



Fire Diamond

Flash Point: 2.2 to 3.9 °C Closed Cup
Autoignition Temperature: 460 °C
LEL: 9.7% v/v
UEL: 12.8% v/v
Extinguishing Media: Foam. Dry chemical powder. BCF (where regulations permit). Carbon dioxide. Water spray or fog - Large fires only.
General Fire Hazards/Hazardous Combustion Products: • Liquid and vapor are highly flammable.
• Severe fire hazard when exposed to heat, flame and/or oxidizers.
• Vapor forms an explosive mixture with air.
• Severe explosion hazard, in the form of vapor, when exposed to flame or spark.
• Vapor may travel a considerable distance to source of ignition.
• Heating may cause expansion/decomposition with violent rupture of containers.
• On combustion, may emit toxic fumes of carbon monoxide (CO). Other combustion products include hydrogen chloride and phosgene.
Fire Incompatibility: Avoid contamination with oxidizing agents i.e., nitrates, oxidizing acids, chlorine bleaches, pool chlorine etc. as ignition may result.
Fire-Fighting Instructions: • Contact fire department and tell them location and nature of hazard.
• May be violently or explosively reactive.
• Wear breathing apparatus plus protective gloves.
• Prevent, by any means available, spillage from entering drains or waterways.
• Consider evacuation (or protect in place).
• Fight fire from a safe distance, with adequate cover.
• If safe, switch off electrical equipment until vapor fire hazard removed.
• Use water delivered as a fine spray to control the fire and cool adjacent area.
• Avoid spraying water onto liquid pools.
• Do not approach containers suspected to be hot.
• Cool fire-exposed containers with water spray from a protected location.
• If safe to do so, remove containers from path of fire.

Section 6 - Accidental Release Measures

See DOT ERG

Small Spills: • Remove all ignition sources.
• Clean up all spills immediately.
• Avoid breathing vapors and contact with skin and eyes.
• Control personal contact by using protective equipment.
• Contain and absorb small quantities with vermiculite or other absorbent material.
• Wipe up.
• Collect residues in a flammable waste container.
Large Spills: • Clear area of personnel and move upwind.
• Contact fire department and tell them location and nature of hazard.
• May be violently or explosively reactive.
• Wear breathing apparatus plus protective gloves.
• Prevent, by any means available, spillage from entering drains or waterways.
• No smoking, bare lights or ignition sources.
• Increase ventilation.
• Stop leak if safe to do so.
• Water spray or fog may be used to disperse/absorb vapor.
• Contain spill with sand, earth or vermiculite.
• Use only spark-free shovels and explosion proof equipment.
• Collect recoverable product into labeled containers for recycling.
• Absorb remaining product with sand, earth or vermiculite.
• Collect solid residues and seal in labeled drums for disposal.
• Wash area and prevent runoff into drains.
• If contamination of drains or waterways occurs, advise emergency services.
Regulatory Requirements: Follow applicable OSHA regulations (29 CFR 1910.120).

Section 7 - Handling and Storage

Handling Precautions: • Avoid all personal contact, including inhalation.
• Wear protective clothing when risk of exposure occurs.
• Use in a well-ventilated area.
• Prevent concentration in hollows and sumps.

- DO NOT enter confined spaces until atmosphere has been checked.
- Avoid smoking, bare lights or ignition sources.
- Avoid contact with incompatible materials.
- When handling, DO NOT eat, drink or smoke.
- Keep containers securely sealed when not in use.
- Avoid physical damage to containers.
- Always wash hands with soap and water after handling.
- Work clothes should be laundered separately.
- Follow good occupational work practices.
- Observe manufacturer's storage and handling recommendations.
- Atmosphere should be regularly checked against established exposure standards to ensure safe working conditions.

Recommended Storage Methods: Check that containers are clearly labeled. Packaging as recommended by manufacturer. DO NOT use aluminum or galvanized containers.

Regulatory Requirements: Follow applicable OSHA regulations.

Section 8 - Exposure Controls / Personal Protection

Engineering Controls: Local exhaust ventilation usually required. If risk of overexposure exists, wear NIOSH-approved respirator. Provide adequate ventilation in warehouse or closed storage area.

Personal Protective Clothing/Equipment:

Eyes: Safety glasses with side shields or, as required, chemical goggles. Contact lenses pose a special hazard; soft lenses may absorb irritants and all lenses concentrate them.

Hands/Feet: Butyl rubber gloves. Neoprene gloves.

Respiratory Protection: Respirator protection may be required. Consult your supervisor.

Other: • Overalls.

- Barrier cream.
- Eyewash unit.

Glove Selection Index:

VITON .... Poor to dangerous choice for other than short-term immersion

Section 9 - Physical and Chemical Properties

Appearance/General Info: Colorless liquid with pleasant chloroform-like odor.

Physical State: colorless liquid

Odor Threshold: 0.085 ppm

Vapor Pressure (kPa): 200 mm Hg at 25 °C

Vapor Density (Air=1): 3.34

Formula Weight: 96.94

Specific Gravity (H_2O=1, at 4 °C): 1.2837 at 20 °C/4 °C

Boiling Point: 60.3 °C (141 °F) at 760 mm Hg

Freezing/Melting Point: -80.5 °C (-112.9 °F)

Water Solubility: 1 to 5 mg/mL at 16 °C

Section 10 - Stability and Reactivity

Stability/Polymerization/Conditions to Avoid: Product is considered stable. Hazardous polymerization will not occur.

Storage Incompatibilities: Avoid reaction with oxidizing agents. Acetylene dichloride in contact with solid caustic alkalies or their concentrated solutions will form chloracetylene which ignites in air. Haloalkenes are highly reactive.

Section 11 - Toxicological Information

Toxicity

Inhalation (mouse) LC_{Lo}: 65000 mg/m^3/2 hr

Rat liver cell mutagen in vitro

Irritation

Nil reported

See *RTECS* KV9420000, for additional data.

Section 12 - Ecological Information

Environmental Fate: If released on soil, it should evaporate and/or leach into the groundwater where very slow biodegradation should occur. If released into water, it will be lost mainly through volatilization (half life 3 hr in a model river). Biodegradation, adsorption to sediment, and bioconcentration in aquatic organisms should not be significant. In the atmosphere it will be lost by reaction with photochemically produced hydroxyl radicals (half life 8 days) and scavenged by rain. Because it is relatively long lived in the atmosphere, considerable dispersal from source areas should occur.

Ecotoxicity: LC_{50} Lepomis machrochirus (bluegill) 135,000 ug/l/96 hr in a static unmeasured bioassay

Henry's Law Constant: estimated at 0.00337

BCF: calculated at 15
Octanol/Water Partition Coefficient: log $K_{ow} = 1.86$
Soil Sorption Partition Coefficient: $K_{oc} = 49$

Section 13 - Disposal Considerations

Disposal: • Consult manufacturer for recycling options and recycle where possible.
- Follow applicable local, state, and federal regulations.
- Incinerate residue at an approved site.
- Recycle containers if possible, or dispose of in an authorized landfill.

Section 14 - Transport Information

DOT Hazardous Materials Table Data (49 CFR 172.101):

Shipping Name and Description: 1,2-Dichloroethylene
ID: UN1150
Hazard Class: 3 - Flammable and combustible liquid
Packing Group: II - Medium Danger
Symbols:
Label Codes: 3 - Flammable Liquid
Special Provisions: IB2, T7, TP2
Packaging: **Exceptions:** 150 **Non-bulk:** 202 **Bulk:** 242
Quantity Limitations: **Passenger aircraft/rail:** 5 L **Cargo aircraft only:** 60 L
Vessel Stowage: **Location:** B **Other:**

Section 15 - Regulatory Information

EPA Regulations:
RCRA 40 CFR: Not listed
CERCLA 40 CFR 302.4: Not listed
SARA 40 CFR 372.65: Not listed
SARA EHS 40 CFR 355: Not listed
TSCA: Listed

Section 16 - Other Information

Disclaimer: Judgments as to the suitability of information herein for the purchaser's purposes are necessarily the purchaser's responsibility. Although reasonable care has been taken in the preparation of such information, Genium Group, Inc. extends no warranties, makes no representations, and assumes no responsibility as to the accuracy or suitability of such information for application to the purchaser's intended purpose or for consequences of its use.

Section 1 - Chemical Product and Company Identification

Material Name: Ethylene Dichloride **CAS Number:** 107-06-2

Chemical Formula: $C_2H_4Cl_2$

Structural Chemical Formula: ClH_2CCH_2Cl

EINECS Number: 203-458-1

ACX Number: X1000031-6

Synonyms: AETHYLENCHLORID; 1,2-BICHLOROETHANE; BICHLORURE D'ETHYLENE; BORER SOL; BROCIDE; CHLORURE D'ETHYLENE; CLORURO DI ETHENE; 1,2-DCE; DESTRUXOL BORER-SOL; 1,2-DICHLOORETHAAN; 1,2-DICHLOR-AETHAN; DICHLOREMULSION; 1,2-DICHLORETHANE; DI-CHLOR-MULSION; DICHLOR-MULSION; 1,2-DICHLOROETHANE; ALPHA,BETA-DICHLOROETHANE; BETA-DICHLOROETHANE; DICHLORO-1,2-ETHANE; SYM-DICHLOROETHANE; DICHLOROETHYLENE; 1,2-DICLOROETANO; DUTCH LIQUID; DUTCH OIL; EDC; ENT 1,656; ETHANE DICHLORIDE; ETHANE,1,2-DICHLORO-; ETHYLEENDICHLORIDE; ETHYLENE CHLORIDE; 1,2-ETHYLENE DICHLORIDE; ETHYLENE DICHLORIDE; FREON 150; GLYCOL DICHLORIDE; NU-G00511; RY DICHLORO-1,2-ETHANE

Derivation: Prepared from reaction of acetone and hydrochloric acid, or ethylene and chloride gas.

General Use: Used primarily as an intermediate in the manufacture of vinyl chloride; also used as a degreaser, as a scavenger in leaded gasoline, in paint removers, in wetting and penetrating agents, in ore floatation processes, as a fumigant, and as a solvent for fats, oils, waxes and gums.

Section 2 - Composition / Information on Ingredients

Name	CAS	%
Ethylene dichloride	107-06-2	ca 100% vol

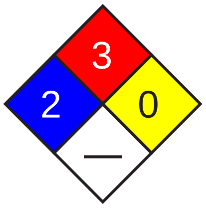
OSHA PEL
TWA: 50 ppm; Ceiling: 100 ppm; 200 ppm, 5-minute maximum peak in any 3 hours.

ACGIH TLV
TWA: 10 ppm.

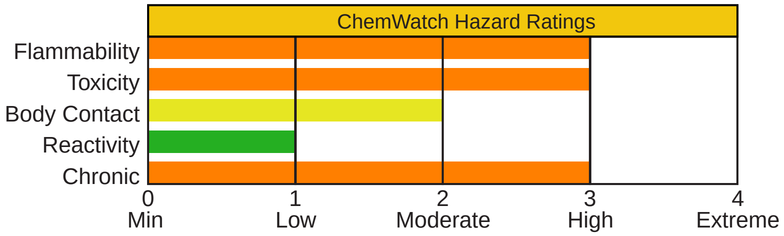
NIOSH REL
TWA: 1 ppm (4 mg/m^3); STEL: 2 ppm (8 mg/m^3) (Chloroethanes).

IDLH Level
50 ppm.

Section 3 - Hazards Identification



Fire Diamond



HMIS
2 Health
3 Flammability
0 Reactivity

ANSI Signal Word

Warning!

Flammable

☆☆☆☆☆ Emergency Overview ☆☆☆☆☆

Clear, colorless liquid; sweet chloroform-like odor. Irritating to respiratory tract. Other Acute Effects: intoxication, CNS depression, vomiting, dizziness, diarrhea, liver/kidney damage, cardiac arrhythmia, coma. death. Possible human carcinogen. Highly flammable. Reacts violently with amines and finely divided alkali metals.

Potential Health Effects

Target Organs: Liver, kidneys, heart, gastrointestinal tract, respiratory tract, skin, CNS, eyes; in animal testing, adrenal glands; cancer sites: forestomach, mammary gland, and circulatory system

Primary Entry Routes: Inhalation, skin and/or eye contact/absorption

Acute Effects Note: Nursing infants of mothers exposed to ethylene dichloride are at risk.

Inhalation: Inhalation may result in respiratory tract irritation, pulmonary edema, dizziness, vomiting, coma and delayed death.

Eye: At high concentrations vapors are irritating. Contact with liquid may cause pain, irritation, lacrimation, and, if not rapidly removed, permanent clouding of the cornea.

Skin: Skin contact with this defatting agent can cause drying and chapping. Prolonged contact with the skin, as when held tightly on skin with clothing, produces severe irritation, moderate edema, and necrosis. Absorption can result in acute systemic effects; only large doses produce serious poisoning.

Ingestion: CNS depression, gastrointestinal upset, mental confusion, dizziness, nausea, and vomiting may result from ingestion and may pose an aspiration hazard. Deaths have occurred from ingestion of 8-200 mL.

Carcinogenicity: NTP - Class 2B, Reasonably anticipated to be a carcinogen, sufficient evidence of carcinogenicity from studies in experimental animals; IARC - Group 2B, Possibly carcinogenic to humans; OSHA - Not listed; NIOSH - Listed as carcinogen; ACGIH - Class A4, Not classifiable as a human carcinogen; EPA - Class B2, Probable human carcinogen based on animal studies; MAK - Class A2, Unmistakably carcinogenic in animal experimentation only.

Medical Conditions Aggravated by Long-Term Exposure: Liver and kidney disease, cardiovascular and CNS disorders; conditions requiring the use of insulin or anti-coagulants may be aggravated by exposure to ethylene dichloride.

Chronic Effects: Long term exposure can result in hepatotoxicity (liver damage), nephrotoxiciy (kidney damage), weight loss, low blood pressure, jaundice, oliguria (reduced urine excretion), anemia, CNS depression, insomnia, nausea, vomiting, pulmonary congestion, and adrenal gland damage. Animal studies suggest that immunologic suppression may occur. Repeated skin contact may produce dermatitis with rough, red, dry, cracking skin.

Section 4 - First Aid Measures

See DOT ERG

Inhalation: Remove exposed person to fresh air, monitor for respiratory distress, and support breathing as needed.

Eye Contact: *Do not* allow victim to rub or keep eyes tightly shut. Gently lift eyelids and flush immediately and continuously with flooding amounts of water until transported to an emergency medical facility. Consult a physician or ophthalmologist immediately.

Skin Contact: *Quickly* remove contaminated clothing. Rinse with flooding amounts of water for at least 15 min. Wash exposed area with soap and water. For reddened or blistered skin, consult a physician.

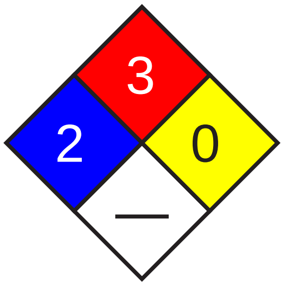
Ingestion: Never give anything by mouth to an unconscious or convulsing person. Contact a poison control center. Unless advised otherwise, have the *conscious and alert* person drink 1 to 2 glasses of water. *Do not* induce vomiting. Consult physician immediately.

After first aid, get appropriate in-plant, paramedic, or community medical support.

Note to Physicians: Implement medical surveillance procedures for workers with potential for exposure. Monitor prothrombin time, serum glucose, electrolytes, liver function, and renal function in severe cases, and arterial blood gases and chest x-ray if respiratory tract irritation is present. Treat overexposure symptomatically and supportively.

Section 5 - Fire-Fighting Measures

See DOT ERG



Fire Diamond

Flash Point: 56 °F (13 °C) CC; 64°F (18 °C) OC

Autoignition Temperature: 775 °F (413 °C)

LEL: 6.2% v/v

UEL: 15.9% v/v

Flammability Classification: OSHA Class IB Flammable Liquid

Extinguishing Media: To extinguish fires involving this flammable liquid, use carbon dioxide, dry chemical, alcohol-resistant foam, water spray or fog. Water may be an ineffective extinguishing medium, but a water spray can be used to cool fire-exposed containers, and flush spills away from ignition sources.

General Fire Hazards/Hazardous Combustion Products: Include carbon oxides, acetylene, vinyl chloride, hydrogen chloride, and phosgene gas. In still air or confined spaces the heavier-than-air vapors of ethylene dichloride may travel along low-lying surfaces to distant ignition sources and flashback. Forms dense soot on burning. Vapors may form explosive mixtures with air.

Fire-Fighting Instructions: Do not release runoff from fire control methods to sewers or waterways. Remove containers from fire hazard area if feasible. Otherwise, cool fire-exposed containers until well after the fire is extinguished. Structural protective clothing is permeable, remain clear of smoke, water fallout, and water runoff. Because fire may produce toxic thermal decomposition products, wear a self-contained breathing apparatus (SCBA) with a full facepiece operated in pressure-demand or positive-pressure mode.

Section 6 - Accidental Release Measures

See DOT ERG

Spill/Leak Procedures: Notify safety personnel, isolate area and deny entry. Remove sources of ignition, and provide maximum explosion-proof ventilation. Stay upwind and have cleanup personnel protect against inhalation and contact. Use appropriate foam to blanket release and suppress vapors.

Small Spills: Absorb ethylene dichloride with vermiculite, earth, sand or similar material.

Large Spills: For large spills, dike far ahead of liquid spill for later disposal. Do not release into sewers or waterways. Ground all tools. Use nonsparking equipment.

Regulatory Requirements: Follow applicable OSHA regulations (29 CFR 1910.120).

Section 7 - Handling and Storage

Handling Precautions: Avoid inhalation of vapors, contact with skin and eyes. Use only with ventilation sufficient to maintain airborne concentrations at nonhazardous levels (see Sec. 2). Wear protective gloves, goggles, and clothing (see Sec. 2). Keep away from heat and ignition sources. Ground and bond containers during transfers to prevent static sparks.

Never eat, drink, or smoke in work areas. Practice good personal hygiene after using this material, especially before eating, drinking, smoking, using the toilet, or applying cosmetics.

Recommended Storage Methods: Store in tightly closed container in well-ventilated area, away from heat, ignition sources and incompatibles (see Sec. 10). Ethylene dichloride is normally packaged under nitrogen gas.

Regulatory Requirements: Follow applicable OSHA regulations.

Section 8 - Exposure Controls / Personal Protection

Engineering Controls: Where possible, transfer ethylene dichloride from drums or other storage containers to process containers in a closed system. Minimize ignition sources in surrounding low-lying areas where ethylene chloride vapors may collect. Electrically ground and bond all containers and equipment. Install Class I, Group D electrical equipment. Provide general or local explosion-proof exhaust ventilation systems to maintain airborne concentrations as low as possible. Local exhaust ventilation is preferred because it prevents contaminant dispersion into the work area by controlling it at its source.

Administrative Controls: Advise employees of potential health hazards associated with occupational exposure to ethylene dichloride. Consider preplacement and periodic medical exams with emphasis on the skin, eyes, respiratory tract, CNS, cardiovascular system, and liver and kidney function.

Personal Protective Clothing/Equipment: Wear chemically protective gloves, boots, aprons, and gauntlets of Barricade^{TM}, Viton^{TM}, Teflon^{TM}, or Responder^{TM} (Breakthrough Time (BT) >8 hr), if possible, or alternatively, polyvinyl alcohol, 4H^{TM} (PE/EVAL) (BT >4 hr) to prevent prolonged or repeated skin contact. Butyl rubber, natural rubber, polyethylene, Neoprene, nitrile rubber, and polyvinyl chloride (BT < 1 hr) will rapidly degrade in the presence of ethylene dichloride, and are not recommended for protective clothing. Wear splash-proof chemical goggles and faceshield, per OSHA eye- and face-protection regulations (29 CFR 1910.133). Contact lenses are not eye protective devices. Appropriate eye protection must be worn instead of, or in conjunction with contact lenses.

Respiratory Protection: Seek professional advice prior to respirator selection and use. Follow OSHA respirator regulations (29 CFR 1910.134) and, if necessary, wear a MSHA/NIOSH-approved respirator. Select respirator based on its suitability to provide adequate worker protection for given working conditions, level of airborne contamination, and presence of sufficient oxygen. At concentrations above the NIOSH RELs, use any SCBA with full facepiece operated in pressure-demand or other positive-pressure mode, or any supplied-air respirator with full facepiece operated in pressure-demand or other positive pressure mode in combination with auxiliary SCBA operated in pressure-demand or other positive-pressure mode. For emergency or nonroutine operations (cleaning spills, reactor vessels, or storage tanks), wear an SCBA. *Warning! Air-purifying respirators do not protect workers in oxygen-deficient atmospheres.* If respirators are used, OSHA requires a written respiratory protection program that includes at least: medical certification, training, fit-testing, periodic environmental monitoring, maintenance, inspection, cleaning, and convenient, sanitary storage areas.

Other: Separate contaminated work clothes from street clothes. Launder before reuse. Remove this material from your shoes and clean personal protective equipment. Make emergency eyewash stations, safety/quick-drench showers, and washing facilities available in work area.

Section 9 - Physical and Chemical Properties

Appearance/General Info: Clear, colorless with a sweet chloroform- like odor characteristic of chlorinated hydrocarbons.

Physical State: Liquid

Odor Threshold: 24 to 440 mg/m^3

Vapor Pressure (kPa): 87 mm Hg at 77 °F (25 °C)

Formula Weight: 98.96

Specific Gravity (H_2O=1, at 4 °C): 1.26 at 69 °F (20 °C)

Refractive Index: 1.445 at 69 °F (20 °C)

Boiling Point: 182.3 °F (83.5 °C)

Freezing/Melting Point: -31.9 °F (-35.5 °C)

Viscosity: 0.84 cP at 68 °F (20 °C)
Surface Tension: 32.2 dynes/cm
Ionization Potential (eV): 11.05 eV

Water Solubility: 8.7 g/L at 68 °F (20 °C)
Other Solubilities: Alcohol, chloroform, ether, acetone, carbon tetrachloride

Section 10 - Stability and Reactivity

Stability/Polymerization/Conditions to Avoid: Ethylene dichloride is stable at room temperature in closed containers under normal storage and handling conditions. However, over time, it slowly decomposes, becomes acidic and darkens in color. Hazardous polymerization cannot occur. Heat and incompatibles.
Storage Incompatibilities: Violent reactions can result from contact with liquid ammonia; nitrogen tetraoxide; chlorine; dimethylaminopropylamine; finely divided metals including aluminum, potassium, and magnesium; other alkalis, amines, strong oxidizers, strong acids, strong bases, and reducing agents. Ethylene dichloride can corrode steel, iron and other metals.
Hazardous Decomposition Products: Thermal oxidative decomposition of ethylene dichloride produces carbon oxides (CO_x), acetylene, vinyl chloride, hydrogen chloride (HCl), and phosgene gas.

Section 11 - Toxicological Information

Acute Oral Effects:
Rat, oral, LD_{50}: 670 mg/kg.
Human, LD_{Lo}: 286 mg/kg produced toxic effects including ulceration or bleeding from stomach, nausea or vomiting, and fatty liver degeneration.
Human, TD_{Lo}: 428 mg/kg produced somnolence, cough, nausea or vomiting.
Human, TD_{Lo}: 892 mg/kg produced hypermotility, diarrhea, nausea or vomiting, and liver effects.
Acute Inhalation Effects:
Human, inhalation, TC_{Lo}: 4000 ppm/1 hr produced flaccid paralysis without anesthesia, coma, and nausea or vomiting.
Rat, inhalation, TC_{50}: 1000 ppm/7 hr produced coma, cyanosis, and body temperature decrease.
Acute Skin Effects:
Rabbit, skin, LD_{50}: 2800 mg/kg produced lacrimation, general anesthesia, and ataxia.
Irritation Effects:
Rabbit, ocular, 63 mg, 24 hr, caused severe irritation.
Other Effects:
Multiple Dose Toxicity Effects: Rat, inhalation, 1500 ppm/7 hr/5 days administered intermittently produced respiratory depression, changes in kidney, ureter or bladder tubules, and death.
Guinea pig, inhalation, 100 ppm/226 day, 7 hr/day, caused body weight loss, and increased liver weight.
Mouse, oral, 4.89 mg/kg/14 day, caused a 30% decrease in leukocyte number and suppressed humoral immune response.
Reproductive Effects: Rat, intrauterine, 1.5 ppm, day 7- term, resulted in increased rates of fetal heart defects.
Genetic Effects: Rat, oral, 150 mg/kg, resulted in DNA damage.
Tumorigenicity - Rat, inhalation, 5 ppm/7 hr/78 weeks, administered intermittently, resulted in production of tumors (skin and appendages) and leukemia.
Rat, oral, 47 mg/kg/day/78 weeks, caused increase in hemangiosarcomas of the circulatory system, squamous cell carcinomas of the forestomach, mammary gland adenocarcinomas and fibroadenomas.

See *RTECS* KI0525000, for additional data.

Section 12 - Ecological Information

Environmental Fate: Because of its moderately high vapor pressure, ethylene dichloride will readily evaporate from surface water (estimated half-life several hours to 10 days), as well as soil. It is not known to bioaccumulate. In the atmosphere, ethylene dichloride may be transported over long distances, and will degrade primarily by photo-oxidation (half-life: 1 month). In groundwater, this chemical does not readily degrade. Ethylene dichloride does not readily sorb and is highly mobile in the soil column.
Ecotoxicity: Stonefly (*Pteronarcys*), LC_{50}=100 mg/L/96 hr; rainbow trout (*Salmo gairdneri*), LC_{50}=225 mg/L/96 hr; bluegill (*Lepomis macrochirs*), LC_{50}=1430 mg/L/96 hr; fathead minnow (*Pimephales promelas*), LC_{50}=136 mg/L/96 hr.
Henry's Law Constant: 1.10×10^{-3} atm-m^3/mole
Octanol/Water Partition Coefficient: $\log K_{ow} = 1.48$

Section 13 - Disposal Considerations

Disposal: Contact your supplier or a licensed contractor for detailed recommendations. Follow applicable Federal, state, and local regulations.

Section 14 - Transport Information

DOT Hazardous Materials Table Data (49 CFR 172.101):

Shipping Name and Description: Ethylene dichloride
ID: UN1184
Hazard Class: 3 - Flammable and combustible liquid
Packing Group: II - Medium Danger
Symbols:
Label Codes: 3 - Flammable Liquid, 6.1 - Poison *or* Poison Inhalation Hazard *if inhalation hazard, Zone A or B*
Special Provisions: IB2, T7, TP1
Packaging: **Exceptions:** None **Non-bulk:** 202 **Bulk:** 243
Quantity Limitations: **Passenger aircraft/rail:** 1 L **Cargo aircraft only:** 60 L
Vessel Stowage: **Location:** B **Other:** 40

Section 15 - Regulatory Information

EPA Regulations:
- **RCRA 40 CFR:** Listed U077 Toxic Waste
- **CERCLA 40 CFR 302.4:** Listed per CWA Section 311(b)(4), per RCRA Section 3001, per CWA Section 307(a) 100 lb (45.35 kg)
- **SARA 40 CFR 372.65:** Listed
- **SARA EHS 40 CFR 355:** Not listed
- **TSCA:** Listed

Section 16 - Other Information

Disclaimer: Judgments as to the suitability of information herein for the purchaser’s purposes are necessarily the purchaser’s responsibility. Although reasonable care has been taken in the preparation of such information, Genium Group, Inc. extends no warranties, makes no representations, and assumes no responsibility as to the accuracy or suitability of such information for application to the purchaser’s intended purpose or for consequences of its use.

Material Safety Data Sheet Collection
Genium group inc.
1171 RiverFront Center, Amsterdam, NY 12010
(518) 842-4111

Issue Date: 2006-06

Hydrochloric Acid
HYD2200

Section 1 - Chemical Product and Company Identification

Material Name: Hydrochloric Acid

CAS Number: 7647-01-0

Chemical Formula: ClH

Structural Chemical Formula: HCl

EINECS Number: 231-595-7

ACX Number: X1002202-3

Synonyms: 4-D BOWL SANITIZER; ACIDE CHLORHYDRIQUE; ACIDO CLORHIDRICO; ACIDO CLORIDRICO; ANHYDROUS HYDROCHLORIC ACID; ANHYDROUS HYDROGEN CHLORIDE; AQUEOUS HYDROGEN CHLORIDE; BOWL CLEANER; CHLOORWATERSTOF; CHLOROHYDRIC ACID; CHLOROWODOR; CHLORURE D'HYDROGENE; CHLORURE D'HYDROGENE ANHYDRE; CHLORURO DE HIDROGENO; CHLORWASSERSTOFF; CLORURO DE HIDROGENO ANHIDRO; EMULSION BOWL CLEANER; EPA PESTICIDE CHEMICAL CODE 045901; HYDROCHLORIC ACID; HYDROCHLORIC ACID GAS; HYDROCHLORIDE; HYDROGEN CHLORIDE; HYDROGEN CHLORIDE (HCL); HYGEIA CREME MAGIC BOWL CLEANER; MURIATIC ACID; MURIATIC ACID); NOW SOUTH SAFTI-SOL BRAND CONCENTRATED BOWL CLEANSE WITHMAGIC ACTIO; PERCLEEN BOWL AND URINAL CLEANER; SPIRITS OF SALT; VARLEY'S OCEAN BLUE SCENTED TOILET BOWL CLEANER; VARLEY POLY-PAK BOWL CREME; WHITE EMULSION BOWL CLEANER; WUEST BOWL CLEANER SUPER CONCENTRATED

General Use: Hydrogen chloride is used to produce pharmaceutical hydrochlorides; vinyl chloride from acetylene; alkyl chlorides from olefins and arsenious chloride from arsenious oxide; electronic grade for etching semiconductor crystals. Used in the chlorination of rubber; in organic reactions involving isomerization, polymerization and alkylation; as a catalyst and condensing agent; for making chlorine where economical; in the separation of cotton from wool and cotton de-linting; as flux in the babbitt type of metal alloy; etching semi-conductor crystals.
Hydrochloric acid is used for pickling and heavy duty cleaning of metal parts; rust and scale removal. The production of chlorides; neutralizing bases; a laboratory reagent. For hydrolyzing starch and proteins in preparations for food. As a catalyst and solvent in organic synthesis. As "spirits of salts" for cleaning of lime and masonry from new brickwork. As flux or flux component for soldering; manufacture of "killed spirits".

Section 2 - Composition / Information on Ingredients

Name	CAS	%
hydrogen chloride	7647-01-0	> 99.0

OSHA PEL
Ceiling: 5 ppm, 7 mg/m^3.

NIOSH REL
Ceiling: 5 ppm (7 mg/m^3).

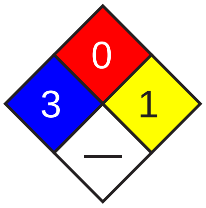
DFG (Germany) MAK
TWA: 5 ppm; PEAK: 5 ppm.

ACGIH TLV
Ceiling: 2 ppm.

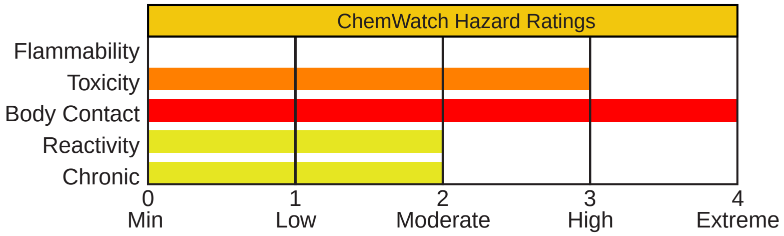
IDLH Level
50 ppm.

EU OEL
TWA: 5 ppm; STEL: 10 ppm.

Section 3 - Hazards Identification



Fire Diamond

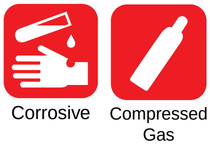


HMIS

2	Health
0	Flammability
0	Reactivity

ANSI Signal Word

Danger!



☆☆☆☆☆ Emergency Overview ☆☆☆☆☆

Colorless gas; characteristic suffocating, pungent odor. Corrosive. Stored as compressed gas which may cause frostbite. Chronic Effects: erosion of teeth.

Potential Health Effects

Target Organs: eyes, skin, respiratory system, liver (in animals)

Primary Entry Routes: inhalation, skin contact, eye contact

Acute Effects

Inhalation: The vapor is extremely discomforting to the upper respiratory tract, may cause severe mucous membrane damage and may be harmful if inhaled.

Inhalation of quantities of liquid mist may be extremely hazardous, even lethal due to spasm, extreme irritation of larynx and bronchi, chemical pneumonitis and pulmonary edema.

A single severe exposure may cause coughing and choking; bleeding of nose, inflammation and occasionally ulceration of the nose, throat and larynx. Fluid on the lungs followed by generalized lung damage may follow.

Breathing of vapor may aggravate asthma and inflammatory or fibrotic pulmonary disease.

High concentrations cause necrosis of the tracheal and bronchial epithelium, pulmonary edema, atelectasis and emphysema and damage to the pulmonary blood vessels and liver.

Inhalation hazard is increased at higher temperatures.

The vapor from heated material is extremely discomforting to the upper respiratory tract and lungs if inhaled.

Continued severe exposure can result in pulmonary edema and corrosion of tissues in the nose and throat.

Eye: Hydrogen Chloride: The vapor is extremely discomforting to the eyes and is capable of causing pain and severe conjunctivitis. Corneal injury may develop, with possible permanent impairment of vision, if not promptly and adequately treated.

The material may be irritating to the eye, with prolonged contact causing inflammation. Repeated or prolonged exposure to irritants may produce conjunctivitis.

Hydrochloric Acid: Eye contact is extremely painful and may cause rapid corneal damage. The liquid is extremely corrosive to the eyes and is capable of causing severe damage with loss of sight.

The vapor is highly discomforting and may be corrosive to the eyes. The vapor from heated material is extremely discomforting to the eyes.

Skin: The material is corrosive to the skin and may cause chemical burns.

Toxic effects may result from skin absorption. Bare unprotected skin should not be exposed to this material. The material may accentuate any pre-existing skin condition.

The vapor is discomforting to the skin.

Ingestion: Considered an unlikely route of entry in commercial/industrial environments.

The liquid is extremely corrosive if swallowed and is capable of causing burns to mouth, throat, esophagus, with extreme discomfort, pain and may be fatal if swallowed in quantity. Ingestion may result in nausea, abdominal irritation, pain and vomiting.

Carcinogenicity: NTP - Not listed; IARC - Group 3, Not classifiable as to carcinogenicity to humans; OSHA - Not listed; NIOSH - Not listed; ACGIH - Not listed; EPA - Not listed; MAK - Not listed.

Chronic Effects: Chronic exposure may cause discoloration or erosion of the teeth, bleeding of the nose and gums; and ulceration of the nasal mucous membranes.

Repeated exposures of animals to concentrations of about 34 ppm produced no immediate toxic effects.

Workers exposed to hydrochloric acid suffered from gastritis and a number of cases of chronic bronchitis have also been reported.

Repeated or prolonged exposure to dilute solutions may cause dermatitis. Repeated exposure to low vapor concentrations can cause skin tenderness, bleeding of the nose and gums, chronic bronchitis, gastritis.

Section 4 - First Aid Measures

See DOT ERG

Inhalation: Remove to fresh air.

Lay patient down. Keep warm and rested.

If breathing is shallow or has stopped, ensure clear airway and apply resuscitation. Transport to hospital or doctor.

Eye Contact: Immediately hold the eyes open and flush continuously for at least 15 minutes with fresh running water. Ensure irrigation under eyelids by occasionally lifting the upper and lower lids.

Transport to hospital or doctor without delay. Removal of contact lenses after an eye injury should only be undertaken by skilled personnel.

Skin Contact: Immediately flush body and clothes with large amounts of water, using safety shower if available.

Quickly remove all contaminated clothing, including footwear.

Wash affected areas with water (and soap if available) for at least 15 minutes. Transport to hospital or doctor.

Ingestion: Contact a Poison Control Center. Rinse mouth out with plenty of water. Do NOT induce vomiting. Give a glass of water.

After first aid, get appropriate in-plant, paramedic, or community medical support.

Note to Physicians: For acute or short-term repeated exposures to strong acids:

1.Airway problems may arise from laryngeal edema and inhalation exposure.

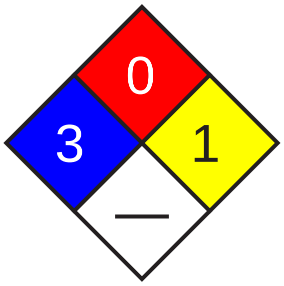
Treat with 100% oxygen initially.

2.Respiratory distress may require cricothyroidotomy if endotracheal intubation is contraindicated by excessive swelling.

3. Intravenous lines should be established immediately in all cases where there is evidence of circulatory compromise.
4.Strong acids produce a coagulation necrosis characterized by formation of a coagulum (eschar) as a result of the desiccating action of the acid on proteins in specific tissues.
INGESTION:
1.Immediate dilution (milk or water) within 30 minutes post-ingestion is recommended.
2.Do not attempt to neutralize the acid since exothermic reaction may extend the corrosive injury.
3.Be careful to avoid further vomiting since re-exposure of the mucosa to the acid is harmful. Limit fluids to one or two glasses in an adult.
4.Charcoal has no place in acid management.
5.Some authors suggest the use of lavage within 1 hour of ingestion.
SKIN:
1.Skin lesions require copious saline irrigation. Treat chemical burns as thermal burns with non-adherent gauze and wrapping.
2.Deep second-degree burns may benefit from topical silver sulfadiazine.
EYE:
1.Eye injuries require retraction of the eyelids to ensure thorough irrigation of the conjunctival cul-de-sacs. Irrigation should last at least 20-30 minutes. Do not use neutralizing agents or any other additives. Several liters of saline are required.
2.Cycloplegic drops (1% cyclopentolate for short-term use or 5% homatropine for longer term use), antibiotic drops, vasoconstrictive agents, or artificial tears may be indicated dependent on the severity of the injury.
3.Steroid eye drops should only be administered with the approval of a consulting ophthalmologist.

Section 5 - Fire-Fighting Measures

See DOT ERG



Fire Diamond

Flash Point: Nonflammable
Autoignition Temperature: Not applicable
LEL: Not applicable
UEL: Not applicable
Extinguishing Media: Water spray or fog; foam; Bromochlorodifluoromethane (BCF) (where regulations permit); Dry agent; Carbon dioxide.
General Fire Hazards/Hazardous Combustion Products: Noncombustible liquid. Will not burn, but heat produces highly toxic fumes/vapors.
Heating may cause expansion or decomposition leading to violent rupture of containers.
Decomposes on heating and produces toxic fumes of hydrogen chloride. Decomposition may produce toxic fumes of chlorine.
Reacts with metals producing flammable/explosive hydrogen gas. Contact with moisture or water may generate heat causing ignition. Reacts vigorously with alkalis. Moderate fire hazard when in contact with reducing agents.
Fire Incompatibility: Reacts with metals producing flammable/explosive hydrogen gas.
Avoid reactions with metals, metal oxides, hydroxides, amines, carbonates, alkaline materials, acetic anhydride, cyanides, sulphides, sulphites, phosphides, acetylides, borides, carbides, silicides, vinyl acetate, formaldehyde and potassium permanganate, unsaturated organics, metal acetylides, sulphuric acid.
Note: Compatibility with plastics should be confirmed prior to use.
Fire-Fighting Instructions: Contact fire department and tell them location and nature of hazard.
Wear full body protective clothing with breathing apparatus. Prevent, by any means available, spillage from entering drains or waterways. Consider evacuation. Cool fire-exposed containers with water spray from a protected location. If safe to do so, remove containers from path of fire. Equipment should be thoroughly decontaminated after use. Water spray or fog may be used to disperse vapor. Do not approach cylinders suspected to be hot. If safe to do so, stop flow of gas.

Section 6 - Accidental Release Measures

See DOT ERG

Small Spills: DO NOT touch the spill material. Clean up all spills immediately. Wear fully protective PVC clothing and breathing apparatus. Contain and absorb spill with sand, earth, inert material or vermiculite. Use soda ash or slaked lime to neutralize. Collect residues and place in labeled plastic containers with vented lids. Clear area of personnel and move upwind. Avoid breathing vapors and contact with skin and eyes. Do not exert excessive pressure on valve; do not attempt to operate damaged valve. Water spray or fog may be used to disperse vapor.
Large Spills: Contact fire department and tell them location and nature of hazard. Clear area of personnel and move upwind. Wear full body protective clothing with breathing apparatus. Prevent, by any means available, spillage from entering drains or waterways. Consider evacuation. Stop leak if safe to do so. Remove leaking cylinders to a safe place if possible. Release pressure under safe, controlled conditions by opening the valve. Do not exert excessive pressure on valve; do not attempt to operate damaged valve. Shut off all possible sources of ignition and increase ventilation. Water spray or fog may be used to disperse vapor. Use soda ash or slaked lime to neutralize.
Collect and seal in labeled drums for disposal. Wash spill area with large quantities of water. If contamination of

drains or waterways occurs, advise emergency services. After clean-up operations, decontaminate and launder all protective clothing and equipment before storing and reusing. DO NOT touch the spill material. Contain and absorb spill with sand, earth, inert material or vermiculite.

DO NOT USE WATER OR NEUTRALIZING AGENTS INDISCRIMINATELY ON LARGE SPILLS.

Regulatory Requirements: Follow applicable OSHA regulations (29 CFR 1910.120).

Section 7 - Handling and Storage

Handling Precautions: Avoid generating and breathing mist and vapor, breathing vapors and contact with skin and eyes.

Avoid physical damage to containers. Use in a well-ventilated area. Wear protective clothing and gloves when handling containers. Handle and open container with care.

WARNING: To avoid violent reaction, ALWAYS add material to water and NEVER water to material. When handling, DO NOT eat, drink or smoke. Always wash hands with soap and water after handling. Work clothes should be laundered separately. Use good occupational work practices. Observe manufacturer's storing and handling recommendations.

Atmosphere should be regularly checked against established exposure standards to ensure safe working conditions are maintained.

Local exhaust ventilation may be required for safe working, i.e. to keep exposures below required standards; otherwise, PPE is required.

Keep dry. Reacts violently with water.

Transport containers on a trolley. Avoid sources of heat. DO NOT transfer gas from one cylinder to another.

Recommended Storage Methods: Packaging as recommended by manufacturer. Check that containers are clearly labeled.

Cylinder. Ensure the use of equipment rated for cylinder pressure. Ensure the use of compatible materials of construction. Valve protection cap to be in place until cylinder is secured, connected. Cylinder must be properly secured either in use or in storage. Cylinder valve must be closed when not in use or when empty. Segregate full from empty cylinders. WARNING: Suckback into cylinder may result in rupture. Use back-flow preventive device in piping.

Hydrochloric acid: Packs of 2.5 litres or less require a child-resistant closure. Glass container or Plastic carboy or Polylined drum.

Regulatory Requirements: Follow applicable OSHA regulations.

Section 8 - Exposure Controls / Personal Protection

Engineering Controls: If risk of overexposure exists, wear air supplied breathing apparatus. Provide adequate ventilation in warehouse or closed storage areas. Use in a well-ventilated area. Local exhaust ventilation may be required for safe working, i. e. , to keep exposures below required standards; otherwise, PPE is required.

If risk of inhalation or overexposure exists, wear NIOSH-approved respirator or work in fume hood. Hydrogen chloride vapors will not be adequately absorbed by organic vapor respirators.

Personal Protective Clothing/Equipment:

Eyes: Chemical goggles. Full face shield.

DO NOT wear contact lenses. Contact lenses pose a special hazard; soft contact lenses may absorb irritants and all lenses concentrate them.

Hands/Feet: Neoprene gloves; rubber gloves. Nitrile gloves.

Safety footwear. Rubber boots.

Hydrochloric acid: Barrier cream and Neoprene gloves or Elbow length PVC gloves. Nitrile gloves.

PVC boots or PVC safety gumboots.

Respiratory Protection:

Exposure Range >5 to <50 ppm: Air Purifying, Negative Pressure, Half Mask
Exposure Range 50 to unlimited ppm: Self-contained Breathing Apparatus, Pressure Demand, Full Face
Cartridge Color: white

Other: Ensure there is ready access to a safety shower; Eyewash unit.

Acid-resistant overalls. Full protective suit. Operators should be trained in procedures for safe use of this material.

Glove Selection Index:

BUTYL	Best selection
BUTYL/NEOPRENE	Best selection
HYPALON	Best selection
NEOPRENE	Best selection
NEOPRENE/NATURAL	Best selection
NITRILE+PVC	Best selection
PE/EVAL/PE	Best selection
SARANEX-23	Best selection
VITON/NEOPRENE	Best selection
PVC	Best selection

NITRILE.................................. Best selection
NATURAL RUBBER............... Satisfactory; may degrade after 4 hours continuous immersion
NATURAL+NEOPRENE......... Satisfactory; may degrade after 4 hours continuous immersion
NAT+NEOPR+NITRILE .... Poor to dangerous choice for other than short-term immersion

Section 9 - Physical and Chemical Properties

Appearance/General Info: Hydrogen chloride: Colorless, corrosive gas. Pungent suffocating odor. White fumes in moist air. Soluble in methanol, ethanol, ether and benzene.
Hydrochloric acid: Clear to light yellow (orange tint for inhibited grades) fuming corrosive liquid with sharp, suffocating odor.

Physical State: Hydrogen chloride: Compressed gas; Hydrochloric acid: Liquid
Odor Threshold: 0.26 to 0.3 ppm
Vapor Pressure (kPa): < 24.8 at 25 °C
Vapor Density (Air=1): 1.268 at 20 °C
Formula Weight: 36.461
Specific Gravity (H_2O=1, at 4 °C): < 1.19 at 20 °C
Evaporation Rate: Slow
pH: Hydrochloric acid: < 1
Boiling Point: -85 °C (-121 °F)
Freezing/Melting Point: -114.44 °C (-173.992 °F)
Volatile Component (% Vol): 100
Decomposition Temperature (°C): Not applicable
Water Solubility: 56.1 g/100 cc hot water at 60 °C

Section 10 - Stability and Reactivity

Stability/Polymerization/Conditions to Avoid: Decomposes in the presence of moisture to produce corrosive acid. May generate sufficient heat to ignite combustible materials. Presence of heat source and direct sunlight (ultra-violet radiation). Product is considered stable under normal handling conditions. Hazardous polymerization will not occur.

Storage Incompatibilities: Hydrogen chloride: Segregate from most common metals and their alloys, alkalis, unsaturated organics, fluorine, metal carbides, metal acetylides, potassium permanganate and sulfuric acid. Compatibility with plastics should be confirmed prior to use.
Hydrochloric acid: Segregate from alkalies, oxidizing agents and chemicals readily decomposed by acids, i.e. cyanides, sulfides, carbonates. Avoid storage with metals, metal oxides, hydroxides, amines, carbonates, alkaline materials, acetic anhydride, cyanides, sulphides, sulphites, phosphides, acetylides, borides, carbides, silicides, vinyl acetate, formaldehyde and potassium permanganate. Reacts with zinc, brass, galvanized iron, aluminum, copper and copper alloys.

Section 11 - Toxicological Information

Toxicity
Inhalation (human) LC_{Lo}: 1300 ppm/30 m
Inhalation (human) LC_{Lo}: 3000 ppm/5 m
Inhalation (rat) LC_{50}: 3124 ppm/60 m
Inhalation (rat) LC_{50}: 4701 ppm/30 m
Oral (rat) LD_{50}: 900 mg/kg

Irritation
Eye (rabbit): 5 mg/30 s - mild

See *RTECS* MW 4025000, for additional data.

Section 12 - Ecological Information

Environmental Fate: No data found.

Ecotoxicity: TL_m Gambusia affinis (mosquito fish) 282 ppm/96 hr (fresh water) /Conditions of bioassay not specified; Lethal Lepomis macrochirus (bluegill sunfish) 3.6 mg/l/48 hr /Conditions of bioassay not specified; LC_{50} Cockle 330 to 1,000 mg/l/48 hr /Conditions of bioassay not specified; LC_{50} Carassius auratus (goldfish) 178 mg/l (1 to 2 hr survival time) /Conditions of bioassay not specified; LC_{50} Shore crab 240 mg/l/48 hr /Conditions of bioassay not specified; LC_{50} Shrimp 100 to 330 ppm/48 hr (salt water) /Conditions of bioassay not specified; LC_{100} Trout 10 mg/l 24 hr /Conditions of bioassay not specified

Biochemical Oxygen Demand (BOD): none

Section 13 - Disposal Considerations

Disposal: Recycle wherever possible. Consult manufacturer for recycling options. Treat and neutralize at an effluent treatment plant. Bury residue in an authorized landfill. Decontaminate empty containers with a lime slurry. Return empty containers to supplier or bury empty containers at an authorized landfill.
Return empty cylinders to supplier.

Section 14 - Transport Information

DOT Hazardous Materials Table Data (49 CFR 172.101):

Note: This material has multiple possible HMT entries. Choose the appropriate one based on state and condition of specific material when shipped.

Shipping Name and Description: Hydrogen chloride, anhydrous
ID: UN1050
Hazard Class: 2.3 - Poisonous gas
Packing Group:
Symbols:
Label Codes: 2.3 - Poison Gas, 8 - Corrosive
Special Provisions: 3
Packaging: **Exceptions:** None **Non-bulk:** 304 **Bulk:** None
Quantity Limitations: **Passenger aircraft/rail:** Forbidden **Cargo aircraft only:** Forbidden
Vessel Stowage: **Location:** D **Other:** 40

Shipping Name and Description: Hydrochloric acid
ID: UN1789
Hazard Class: 8 - Corrosive material
Packing Group: II - Medium Danger
Symbols:
Label Codes: 8 - Corrosive
Special Provisions: A3, A6, B3, B15, IB2, N41, T8, TP2, TP12
Packaging: **Exceptions:** 154 **Non-bulk:** 202 **Bulk:** 242
Quantity Limitations: **Passenger aircraft/rail:** 1 L **Cargo aircraft only:** 30 L
Vessel Stowage: **Location:** C **Other:**

Shipping Name and Description: Hydrochloric acid
ID: UN1789
Hazard Class: 8 - Corrosive material
Packing Group: III - Minor Danger
Symbols:
Label Codes: 8 - Corrosive
Special Provisions: IB3, T4, TP1, TP12
Packaging: **Exceptions:** 154 **Non-bulk:** 203 **Bulk:** 241
Quantity Limitations: **Passenger aircraft/rail:** 5 L **Cargo aircraft only:** 60 L
Vessel Stowage: **Location:** C **Other:**

Section 15 - Regulatory Information

EPA Regulations:
- **RCRA 40 CFR:** Not listed
- **CERCLA 40 CFR 302.4:** Listed per CWA Section 311(b)(4) 5000 lb (2268 kg)
- **SARA 40 CFR 372.65:** Listed
- **SARA EHS 40 CFR 355:** Listed
  - **RQ:** 5000 lb
  - **TPQ:** 500 lb
- **TSCA:** Listed

Section 16 - Other Information

Disclaimer: Judgments as to the suitability of information herein for the purchaser's purposes are necessarily the purchaser's responsibility. Although reasonable care has been taken in the preparation of such information, Genium Group, Inc. extends no warranties, makes no representations, and assumes no responsibility as to the accuracy or suitability of such information for application to the purchaser's intended purpose or for consequences of its use.

Material Safety Data Sheet

24 Hour Assistance:
1-847-367-7700
Rust-Oleum Corp.
www.rustoleum.com

Section 1 - Chemical Product / Company Information

Product Name:	PRO LSPR 6PK MARK FLUORESCENT ORANGE	Revision Date:	08/13/2009
Identification Number:	2554838		
Product Use/Class:	Topcoat/Aerosols		
Supplier:	Rust-Oleum Corporation 11 Hawthorn Parkway Vernon Hills, IL 60061 USA	Manufacturer:	Rust-Oleum Corporation 11 Hawthorn Parkway Vernon Hills, IL 60061 USA
Preparer:	Regulatory Department		

Section 2 - Composition / Information On Ingredients

Chemical Name	CAS Number	Weight % Less Than	ACGIH TLV-TWA	ACGIH TLV-STEL	OSHA PEL-TWA	OSHA PEL CEILING
Liquefied Petroleum Gas	68476-86-8	30.0	N.E.	N.E.	N.E.	N.E.
Aliphatic Hydrocarbon	64742-89-8	20.0	N.E.	N.E.	300 ppm	N.E.
Toluene	108-88-3	15.0	20 ppm	N.E.	200 ppm	300 ppm
Magnesium Silicate	14807-96-6	10.0	2 mg/m3	N.E.	0.1 mg/m3 (Respirable)	N.E.
Hydrotreated Light Distillate	64742-47-8	5.0	200 mg/m3	N.E.	N.E.	N.E.
Naphtha	8032-32-4	5.0	N.E.	N.E.	N.E.	N.E.
Xylene	1330-20-7	5.0	100 ppm	150 ppm	100 ppm	N.E.
Ethylbenzene	100-41-4	1.0	100 ppm	125 ppm	100 ppm	N.E.
Quartz (Crystalline Silica)	14808-60-7	1.0	0.025 mg/m3	N.E.	2.4 mppcf (Respirable)	N.E.

Section 3 - Hazards Identification

*** Emergency Overview ***: Contents Under Pressure. Harmful if inhaled. May affect the brain or nervous system causing dizziness, headache or nausea. Vapors may cause flash fire or explosion. Harmful if swallowed. Extremely flammable liquid and vapor.

Effects Of Overexposure - Eye Contact: Causes eye irritation.

Effects Of Overexposure - Skin Contact: Prolonged or repeated contact may cause skin irritation. Substance may cause slight skin irritation.

Effects Of Overexposure - Inhalation: High vapor concentrations are irritating to the eyes, nose, throat and lungs. Avoid breathing vapors or mists. High gas, vapor, mist or dust concentrations may be harmful if inhaled. Harmful if inhaled.

Effects Of Overexposure - Ingestion: Aspiration hazard if swallowed; can enter lungs and cause damage. Substance may be harmful if swallowed.

Effects Of Overexposure - Chronic Hazards: IARC lists Ethylbenzene as a possible human carcinogen (group 2B).

May cause central nervous system disorder (e.g., narcosis involving a loss of coordination, weakness, fatigue, mental confusion, and blurred vision) and/or damage. Reports have associated repeated and prolonged occupational overexposure to solvents with permanent brain and nervous system damage. Overexposure to xylene in laboratory animals has been associated with liver abnormalities, kidney, lung, spleen, eye and blood damage as well as reproductive disorders. Effects in humans, due to chronic overexposure, have included liver, cardiac abnormalities and nervous system damage. Overexposure to toluene in laboratory animals has been associated with liver abnormalities, kidney, lung and spleen damage. Effects in humans have included liver and cardiac abnormalities.

Contains crystalline silica as silicon dioxide. Excessive inhalation of respirable crystalline silica dust may cause lung disease, silicosis or lung cancer. Significant exposure is not anticipated during brush or trowel application or drying. Risk of overexposure depends on the duration and level of exposure to dust from repeated sanding of surfaces, mechanical abrasion or spray mist and actual concentration of crystalline silica in the formula. Crystalline silica is listed as Group 1 "carcinogenic to humans" by the International Agency for Research on Cancer (IARC), and Group 2 "reasonably anticipated to be a carcinogen" by the National Toxicology Program (NTP).

Primary Route(s) Of Entry: Skin Contact, Skin Absorption, Inhalation, Ingestion, Eye Contact

Section 4 - First Aid Measures

First Aid - Eye Contact: Immediately flush eyes with plenty of water for at least 15 minutes holding eyelids open. Get medical attention. Do NOT allow rubbing of eyes or keeping eyes closed.

First Aid - Skin Contact: Wash with soap and water. Get medical attention if irritation develops or persists.

First Aid - Inhalation: If you experience difficulty in breathing, leave the area to obtain fresh air. If continued difficulty is experienced, get medical assistance immediately.

First Aid - Ingestion: Aspiration hazard: Do not induce vomiting or give anything by mouth because this material can enter the lungs and cause severe lung damage. Get immediate medical attention.

Section 5 - Fire Fighting Measures

Flash Point: -156 F (Setaflash)	LOWER EXPLOSIVE LIMIT: 0.9 % UPPER EXPLOSIVE LIMIT : 32.5 %

Extinguishing Media: Alcohol Film Forming Foam, Carbon Dioxide, Dry Chemical, Water Fog

Unusual Fire And Explosion Hazards: Water spray may be ineffective. Closed containers may explode when exposed to extreme heat. Vapors may form explosive mixtures with air. Vapors can travel to a source of ignition and flash back. FLASH POINT IS LESS THAN 20 °. F. - EXTREMELY FLAMMABLE LIQUID AND VAPOR! Keep containers tightly closed. Isolate from heat, electrical equipment, sparks and open flame. Perforation of the pressurized container may cause bursting of the can.

Special Firefighting Procedures: Evacuate area and fight fire from a safe distance.

Section 6 - Accidental Release Measures

Steps To Be Taken If Material Is Released Or Spilled: Contain spilled liquid with sand or earth. DO NOT use combustible materials such as sawdust. Dispose of according to local, state (provincial) and federal regulations. Do not incinerate closed containers. Remove all sources of ignition, ventilate area and remove with inert absorbent and non-sparking tools.

Section 7 - Handling And Storage

Handling: Wash thoroughly after handling. Follow all MSDS/label precautions even after container is emptied because it may retain product residues. Use only in a well-ventilated area. Avoid breathing vapor or mist. Wash hands before eating.

Storage: Contents under pressure. Do not expose to heat or store above 120 ° F. Do not store above 120 ° F. Store large quantities in buildings designed and protected for storage of NFPA Class I flammable liquids. Keep containers tightly closed. Isolate from heat, electrical equipment, sparks and open flame.

Section 8 - Exposure Controls / Personal Protection

Engineering Controls: Prevent build-up of vapors by opening all doors and windows to achieve cross-ventilation. Use process enclosures, local exhaust ventilation, or other engineering controls to control airborne levels below recommended exposure limits. Use explosion-proof ventilation equipment.

Respiratory Protection: A respiratory protection program that meets OSHA 1910.134 and ANSI Z88.2 requirements must be followed whenever workplace conditions warrant a respirator's use. A NIOSH/MSHA approved air purifying respirator with an organic vapor cartridge or canister may be permissible under certain circumstances where airborne concentrations are expected to exceed exposure limits.

Protection provided by air purifying respirators is limited. Use a positive pressure air supplied respirator if there is any potential for an uncontrolled release, exposure levels are not known, or in any other circumstances where air purifying respirators may not provide adequate protection.

Skin Protection: Nitrile or Neoprene gloves may afford adequate skin protection. Use impervious gloves to prevent skin contact and absorption of this material through the skin.

Eye Protection: Use safety eyewear designed to protect against splash of liquids.

Other protective equipment: Refer to safety supervisor or industrial hygienist for further information regarding personal protective equipment and its application.

Hygienic Practices: Wash thoroughly with soap and water before eating, drinking or smoking.

Section 9 - Physical And Chemical Properties

Boiling Range:	-34 - 468 F	Vapor Density:	Heavier than Air
Odor:	Solvent Like	Odor Threshold:	N.E.
Appearance:	Aerosolized Mist	Evaporation Rate:	Faster than Ether
Solubility in H2O:	None		
Freeze Point:	N.D.	Specific Gravity:	0.874
Vapor Pressure:	N.D.	PH:	N.A.
Physical State:	Liquid		

(See section 16 for abbreviation legend)

Section 10 - Stability And Reactivity

Conditions To Avoid: Avoid temperatures above 120 ° F. Avoid all possible sources of ignition.

Incompatibility: Incompatible with strong oxidizing agents, strong acids and strong alkalies.

Hazardous Decomposition: When heated to decomposition, it emits acrid smoke and irritating fumes. By open flame, carbon monoxide and carbon dioxide.

Hazardous Polymerization: Will not occur under normal conditions.

Stability: This product is stable under normal storage conditions.

Section 11 - Toxicological Information

Product LD50: N.D. Product LC50: N.D.

Chemical Name	**LD50**	**LC50**
Liquefied Petroleum Gas	N.E.	N.E.
Aliphatic Hydrocarbon	>5000 mg/kg (Rat, Oral)	N.E.
Toluene	636 mg/kg (Rat, Oral)	>26700 ppm (Rat, Inhalation, 1Hr)
Magnesium Silicate	N.E.	TCLo: 11 mg/m3 (Inhalation)
Hydrotreated Light Distillate	>3160 mg/kg (Skin)	N.E.
Naphtha	>5000 mg/kg (Rat, Oral)	N.E.
Xylene	4300 mg/kg (Rat, Oral)	5000 ppm (Rat, Inhalation, 4Hr)
Ethylbenzene	3500 mg/kg (Rat, Oral)	N.E.
Quartz (Crystalline Silica)	N.E.	N.E.

Section 12 - Ecological Information

Ecological Information: Product is a mixture of listed components.

Section 13 - Disposal Information

Disposal Information: Dispose of material in accordance to local, state and federal regulations and ordinances. Do not allow to enter storm drains or sewer systems.

Section 14 - Transportation Information

DOT Proper Shipping Name:	ORM-D, Consumer Commodity	Packing Group:	N.A.
DOT Technical Name:	N.A.	Hazard Subclass:	N.A.
DOT Hazard Class:	2.1	Resp. Guide Page:	126
DOT UN/NA Number:	UN1950		

Section 15 - Regulatory Information

CERCLA - SARA Hazard Category

This product has been reviewed according to the EPA "Hazard Categories" promulgated under Sections 311and 312 of the Superfund Amendment and Reauthorization Act of 1986 (SARA Title III) and is considered, under applicable definitions, to meet the following categories:

IMMEDIATE HEALTH HAZARD, CHRONIC HEALTH HAZARD, FIRE HAZARD, PRESSURIZED GAS HAZARD

SARA Section 313:

Listed below are the substances (if any) contained in this product that are subject to the reporting requirements of Section 313 of Title III of the Superfund Amendment and Reauthorization Act of 1986 and 40 CFR part 372:

Chemical Name	CAS Number
Toluene	108-88-3
Xylene	1330-20-7
Ethylbenzene	100-41-4

Toxic Substances Control Act:

Listed below are the substances (if any) contained in this product that are subject to the reporting requirements of TSCA 12(B) if exported from the United States:

It is the policy of Rust-Oleum Corporation to use only TSCA compliant materials in its products.

U.S. State Regulations: As follows -

New Jersey Right-to-Know:

The following materials are non-hazardous, but are among the top five components in this product.

Chemical Name	CAS Number
Calcium Carbonate	1317-65-3

Pennsylvania Right-to-Know:

The following non-hazardous ingredients are present in the product at greater than 3%.

Chemical Name	CAS Number
Calcium Carbonate	1317-65-3
Polymer Anchored Orange Dye Dispersion	MIXTURE
Modified Alkyd	PROPRIETARY

California Proposition 65:

WARNING! This product contains a chemical(s) known by the State of California to cause cancer.

WARNING! This product contains a chemical(s) known to the State of California to cause birth defects or other reproductive harm.

International Regulations: As follows -

CANADIAN WHMIS:

This MSDS has been prepared in compliance with Controlled Product Regulations except for the use of the 16 headings.

CANADIAN WHMIS CLASS: AB5 D2A D2B

Section 16 - Other Information

HMIS Ratings:

Health: 2* Flammability: 4 Reactivity: 0 Personal Protection: X

REASON FOR REVISION: Regulatory Update

Legend: N.A. - Not Applicable, N.E. - Not Established, N.D. - Not Determined

Rust-Oleum Corporation believes, to the best of its knowledge, information and belief, the information contained herein to be accurate and reliable as of the date of this material safety data sheet. However, because the conditions of handling, use, and storage of these materials are beyond our control, we assume no responsibility or liability for personal injury or property damage incurred by the use of these materials. Rust-Oleum Corporation makes no warranty, expressed or implied, regarding the accuracy or reliability of the data or results obtained from their use. All materials may present unknown hazards and should be used with caution. The information and recommendations in this material safety data sheet are offered for the users' consideration and examination. It is the responsibility of the user to determine the final suitability of this information and to comply with all applicable international, federal, state, and local laws and regulations.

Material Safety Data Sheet Collection

Genium group inc.

1171 RiverFront Center, Amsterdam, NY 12010
(518) 842-4111

Issue Date: 2006-06

Methyl tert-Butyl Ether
BUT5410

Section 1 - Chemical Product and Company Identification 61

Material Name: Methyl tert-Butyl Ether **CAS Number:** 1634-04-4

Chemical Formula: $C_5H_{12}O$

Structural Chemical Formula: $(CH_3)_3COCH_3(CH_3)_3COCH_3$

EINECS Number: 216-653-1

ACX Number: X1001502-4

Synonyms: T-BUTYL METHYL ETHER; TERT-BUTYL METHYL ETHER; ETHER,TERT-BUTYL METHYL; 2-METHOXY-2-METHYL PROPANE; 2-METHOXY-2-METHYLPROPANE; METHYL TERT-BUTYL ETHER; METHYL 1,1-DIMETHYLETHYL ETHER; METHYL-TERT-BUTYL ETHER; METHYL TERTIARY BUTYL ETHER; 2-METHYL-2-METHOXYPROPANE; MTBE; PROPANE,2-METHOXY-2-METHYL-; PROPANE,2-METHOXY-2-METHYL-(9CI)

General Use: Octane booster for unleaded petrol (up to 7% by volume), manufacture of isobutene; solvent for a number of applications, including pesticide analysis.

Section 2 - Composition / Information on Ingredients

Name	**CAS**	**%**
methyl tert-butyl ether	1634-04-4	>95
commercial material may contain C_5 hydrocarbons <5%		

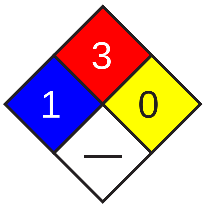
OSHA PEL

NIOSH REL

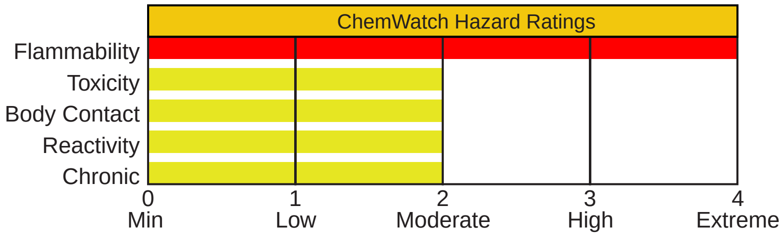
DFG (Germany) MAK
TWA: 50 ppm; PEAK: 75 ppm.

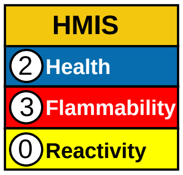
ACGIH TLV
TWA: 50 ppm.

Section 3 - Hazards Identification



Fire Diamond





ANSI Signal Word

Danger!

Flammable

☆☆☆☆☆ Emergency Overview ☆☆☆☆☆

Clear, colorless liquid; slight hydrocarbon/mint or terpene-like odor. Irritating to eyes/skin/respiratory tract. Other Acute Effects: CNS and respiratory depression, aspiration pneumonitis. Chronic Effects: nasal/tracheal inflammation. Flammable.

Potential Health Effects

Target Organs: upper respiratory system, central nervous system (CNS)

Primary Entry Routes: inhalation, ingestion

Acute Effects

Inhalation: The vapor is harmful and discomforting to the upper respiratory tract. Acute effects from inhalation of high vapor concentrations may be chest and nasal irritation with coughing, sneezing, headache and even nausea. If exposure to highly concentrated vapor atmosphere is prolonged this may lead to narcosis, unconsciousness, even coma, and unless resuscitated, death. Inhalation of the lower alkyl ethers may produce intoxication, blurred vision, headache, dizziness, excitation, pharyngitis, and irritation of the nose and throat. Convulsions, respiratory distress or paralysis, asphyxia, pneumonitis, and unconsciousness are all serious manifestations of poisoning; liver and kidney damage may occur. Fatalities have been reported. Ethers produce narcosis following inhalation.

Rats exposed to 8000 ppm for 6 hours showed changes in motor activity with the pattern and time course of effects being indicative of transient central nervous system depression. Transient increases in motor activity, on the other hand, were observed in male rats exposed at 800 and 4000 ppm. This finding may reflect an exposure-related stimulant effect or an exaggerated response following recovery from an anesthetic effect. Rats exposed to 1000 ppm, 6 hours/day, 5 days/week for 9 exposures showed lachrymation, conjunctival swelling and corneal changes. A reduced reaction to auditory stimuli was noted at 3000 ppm. There were no other macroscopic pathology findings other than chronic inflammatory changes in the nasal mucosa and trachea.

Eye: The liquid is extremely discomforting to the eyes.

Eye contact with alkyl ethers (vapors or liquid) may produce irritation, redness and lachrymation.

Skin: The liquid is discomforting to the skin and is capable of causing skin reactions which may lead to dermatitis.

Bare unprotected skin should not be exposed to this material. Considered to be harmful if it is absorbed by the skin.

Alkyl ethers may defat and dehydrate the skin producing dermatoses.

Absorption may produce headache, dizziness, and central nervous system depression.

Ingestion: Considered an unlikely route of entry in commercial/industrial environments. The liquid is extremely discomforting and may be fatal if swallowed. Ingestion may result in nausea, pain, vomiting. Vomit entering the lungs by aspiration may cause potentially lethal chemical pneumonitis. Ingestion of alkyl ethers may produce symptoms to those produced following inhalation.

Carcinogenicity: NTP - Not listed; IARC - Group 2B, Possibly carcinogenic to humans; OSHA - Not listed; NIOSH - Not listed; ACGIH - Class A3, Animal carcinogen; EPA - Not listed; MAK - Not listed.

Chronic Effects: Prolonged or repeated exposure may cause kidney damage.

Rats exposed to 800, 4000 or 8000 ppm, 6 hours/day, 5 days/week, for 13 weeks showed statistically significant increases in the mean absolute and relative weights of liver, kidneys, and adrenal gland in the two higher exposure groups, while mild hematological changes were seen in all animals.

Chronic exposure to alkyl ethers may result in loss of appetite, excessive thirst, fatigue, and weight loss.

Section 4 - First Aid Measures

See DOT ERG

Inhalation: Remove to fresh air. Lay patient down. Keep warm and rested. If available, administer medical oxygen by trained personnel. If breathing is shallow or has stopped, ensure clear airway and apply resuscitation. Transport to hospital or doctor without delay.

Eye Contact: Immediately hold the eyes open and wash continuously for at least 15 minutes with fresh running water. Ensure irrigation under eyelids by occasionally lifting the upper and lower lids. Transport to hospital or doctor without delay. Removal of contact lenses after an eye injury should only be undertaken by skilled personnel.

Skin Contact: Immediately flush body and clothes with large amounts of water, using safety shower if available. Quickly remove all contaminated clothing, including footwear. Wash affected areas with water (and soap if available) for at least 15 minutes. Transport to hospital or doctor.

Ingestion: DO NOT induce vomiting.

Observe the patient carefully. Never give liquid to a person showing signs of being sleepy or with reduced awareness; i.e. becoming unconscious. Give water (or milk) to rinse out mouth. Then provide liquid slowly and as much as casualty can comfortably drink. Transport to hospital or doctor without delay.

After first aid, get appropriate in-plant, paramedic, or community medical support.

Note to Physicians: Treat symptomatically.

Section 5 - Fire-Fighting Measures

Flash Point: -26 °C Closed Cup

Autoignition Temperature: 435 °C

LEL: 1.5% v/v

Extinguishing Media: Foam. Dry chemical powder. BCF (where regulations permit). Carbon dioxide.

Water spray or fog - Large fires only.

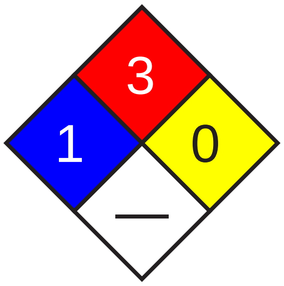
See DOT ERG

General Fire Hazards/Hazardous Combustion Products: Liquid and vapor are highly flammable. Severe fire hazard when exposed to heat, flame and/or oxidizers. Vapor forms an explosive mixture with air.

Severe explosion hazard, in the form of vapor, when exposed to flame or spark. Vapor may travel a considerable distance to source of ignition.

Heating may cause expansion/decomposition with violent rupture of containers.

On combustion, may emit toxic fumes of carbon monoxide (CO). May emit poisonous fumes.



Fire Diamond

Fire Incompatibility: Avoid contact with oxidizing agents and strong acids.

Fire-Fighting Instructions: Contact fire department and tell them location and nature of hazard.

May be violently or explosively reactive. Wear breathing apparatus plus protective gloves. Prevent, by any means available, spillage from entering drains or waterways. Consider evacuation.

Fight fire from a safe distance, with adequate cover.

If safe, switch off electrical equipment until vapor fire hazard removed.
Use water delivered as a fine spray to control the fire and cool adjacent area. Avoid spraying water onto liquid pools.
Do not approach containers suspected to be hot. Cool fire exposed containers with water spray from a protective location. If safe to do so, remove containers from path of fire.

Section 6 - Accidental Release Measures

Small Spills: Remove all ignition sources. Clean up all spills immediately.
Avoid breathing vapors and contact with skin and eyes.
Control personal contact by using protective equipment.
Contain and absorb small quantities with vermiculite or other absorbent material. Wipe up. Collect residues in a flammable waste container.

See DOT ERG

Large Spills: Clear area of personnel and move upwind.
Contact fire department and tell them location and nature of hazard.
May be violently or explosively reactive. Wear breathing apparatus plus protective gloves. Prevent, by any means available, spillage from entering drains or waterways. Consider evacuation.
No smoking, bare lights or ignition sources. Increase ventilation.
Stop leak if safe to do so. Water spray or fog may be used to disperse/absorb vapor. Contain spill with sand, earth or vermiculite.
Use only spark-free shovels and explosion proof equipment.
Collect recoverable product into labeled containers for recycling.
Absorb remaining product with sand, earth or vermiculite.
Collect solid residues and seal in labeled drums for disposal.
Wash area and prevent runoff into drains.
If contamination of drains or waterways occurs, advise emergency services.

Regulatory Requirements: Follow applicable OSHA regulations (29 CFR 1910.120).

Section 7 - Handling and Storage

Handling Precautions: Do not allow clothing wet with material to stay in contact with skin.
Avoid all personal contact, including inhalation. Wear protective clothing when risk of exposure occurs. Use in a well-ventilated area. Prevent concentration in hollows and sumps.
DO NOT enter confined spaces until atmosphere has been checked.
Avoid smoking, bare lights, heat or ignition sources. When handling, DO NOT eat, drink or smoke.
Vapor may ignite on pumping or pouring due to static electricity.
Ground and secure containers when dispensing or pouring product. Use spark-free tools when handling.
Avoid contact with incompatible materials.
Keep containers securely sealed. Avoid physical damage to containers.
Always wash hands with soap and water after handling. Work clothes should be laundered separately.
Use good occupational work practice. Observe manufacturer's storing and handling recommendations. Atmosphere should be regularly checked against established exposure standards to ensure safe working conditions.

Recommended Storage Methods: Metal can; Metal drum; Metal safety cans. Packing as supplied by manufacturer.
Plastic containers may only be used if approved for flammable liquid.
Check that containers are clearly labeled and free from leaks.

Storage Requirements: Store below 38 deg. C.
Rotate all stock to prevent aging. Use on FIFO (First In-First Out) basis.
Store in original containers in approved flame-proof area.
No smoking, bare lights, heat or ignition sources.
DO NOT store in pits, depressions, basements or areas where vapors may be trapped. Keep containers securely sealed.
Store away from incompatible materials in a cool, dry well ventilated area.
Protect containers against physical damage and check regularly for leaks.
Observe manufacturer's storing and handling recommendations.

Regulatory Requirements: Follow applicable OSHA regulations.

Section 8 - Exposure Controls / Personal Protection

Engineering Controls: Use in a well-ventilated area.
General exhaust is adequate under normal operating conditions.
Local exhaust ventilation may be required in specific circumstances.
If risk of overexposure exists, wear NIOSH approved respirator.
Correct fit is essential to obtain adequate protection.
Provide adequate ventilation in warehouse or closed storage areas.
Ground and secure containers when dispensing or pouring.
Avoid generation of static electricity. Ground all lines and equipment.

Vapor may ignite on pumping or pouring due to static electricity. Ground and secure containers when dispensing or pouring product.

Personal Protective Clothing/Equipment:

Eyes: Safety glasses. Safety glasses with side shields. Chemical goggles.
Contact lenses pose a special hazard; soft lenses may absorb irritants and all lenses concentrate them.

Hands/Feet: Nitrile gloves Safety footwear.

Respiratory Protection:
Exposure Range >40 to 2000 ppm: Supplied Air, Constant Flow/Pressure Demand, Half Mask
Exposure Range >2000 to 40,000 ppm: Supplied Air, Constant Flow/Pressure Demand, Full Face
Exposure Range >40,000 to unlimited ppm: Self-contained Breathing Apparatus, Pressure Demand, Full Face
Note: odor threshold unknown

Other: Eyewash unit. Ensure there is ready access to a safety shower.
Impervious apron.

Section 9 - Physical and Chemical Properties

Appearance/General Info: Colorless, highly volatile, highly flammable liquid; ethereal odor.

Physical State: Liquid
Vapor Pressure (kPa): 27.5 at 20 °C
Vapor Density (Air=1): 3.1
Formula Weight: 88.15
Specific Gravity (H_2O=1, at 4 °C): 0.74
Evaporation Rate: Fast
pH: Not applicable

pH (1% Solution): Not applicable
Boiling Point: 55.2 °C (131 °F)
Freezing/Melting Point: -109 °C (-164.2 °F)
Volatile Component (% Vol): 100
Water Solubility: Solubility of water in methyl-tbutyl ether 2 g/100 g

Section 10 - Stability and Reactivity

Stability/Polymerization/Conditions to Avoid: WARNING: Long standing in contact with air and light may result in the formation of potentially explosive peroxides.
Product is considered stable. Hazardous polymerization will not occur.

Storage Incompatibilities: May form explosive peroxides on standing or following concentration by distillation. Review of stocks and testing for peroxide content by given tested procedures at 3-monthly intervals is recommended, together with safe disposal of peroxidic samples.
[Peroxide containing residues can often be rendered innocuous by pouring into an excess of sodium carbonate solution] Avoid storage with oxidizers and strong acids.

Section 11 - Toxicological Information

Toxicity
Oral (rat) LD_{50}: 4000 mg/kg
Inhalation (rat) LC_{50}: 23576 ppm/4 H

Irritation
Nil reported

See *RTECS* KN5250000, for additional data.

Section 12 - Ecological Information

Environmental Fate: If released to soil, it will be subject to volatilization. It will be expected to exhibit very high mobility in soil and, therefore, it may leach to groundwater. It will not be expected to hydrolyze in soil. If released to water, it will not be expected to significantly adsorb to sediment or suspended particulate matter, bioconcentrate in aquatic organisms, hydrolyze, directly photolyze, or photooxidize via reaction with photochemically produced hydroxyl radicals in the water, based upon estimated physical-chemical properties or analogies to other structurally related aliphatic ethers. In surface water it will be subject to rapid volatilization with estimated half-lives of 4.1 hr and 2.0 days for volatilization from a river one meter deep flowing 1 m/sec with a wind velocity of 3 m/sec and a model pond, respectively. It may be resistant to biodegradation in environmental media based upon screening test data from a study using activated sludge inocula. Many ethers are known to be resistant to biodegradation. If released to the atmosphere, it will be expected to exist almost entirely in the vapor phase based on its vapor pressure. It will be susceptible to photooxidation via vapor phase reaction with photochemically produced hydroxyl radicals with an estimated half-life of 5.6 days for this process. Direct photolysis will not be an important removal process since aliphatic ethers do not adsorb light at wavelengths >290 nm.

Ecotoxicity: No data found.

Henry's Law Constant: 5.87×10^{-4}

BCF: carp 1.5

Soil Sorption Partition Coefficient: K_{oc} = estimated at 11.2

Section 13 - Disposal Considerations

Disposal: Consult manufacturer for recycling options and recycle where possible. Follow applicable federal, state, and local regulations. Incinerate residue at an approved site. Recycle containers where possible, or dispose of in an authorized landfill.

BEWARE: Empty solvent, paint, lacquer and flammable liquid drums present a severe explosion hazard if cut by flame torch or welded. Even when thoroughly cleaned or reconditioned the drum seams may retain sufficient solvent to generate an explosive atmosphere in the drum.

Section 14 - Transport Information

DOT Hazardous Materials Table Data (49 CFR 172.101):

Shipping Name and Description: Methyl tert-butyl ether
ID: UN2398
Hazard Class: 3 - Flammable and combustible liquid
Packing Group: II - Medium Danger
Symbols:
Label Codes: 3 - Flammable Liquid
Special Provisions: IB2, T7, TP1
Packaging: **Exceptions:** 150 **Non-bulk:** 202 **Bulk:** 242
Quantity Limitations: **Passenger aircraft/rail:** 5 L **Cargo aircraft only:** 60 L
Vessel Stowage: **Location:** E **Other:**

Section 15 - Regulatory Information

EPA Regulations:
- **RCRA 40 CFR:** Not listed
- **CERCLA 40 CFR 302.4:** Listed per CAA Section 112 1000 lb (453.5 kg)
- **SARA 40 CFR 372.65:** Listed
- **SARA EHS 40 CFR 355:** Not listed
- **TSCA:** Listed

Section 16 - Other Information

Disclaimer: Judgments as to the suitability of information herein for the purchaser's purposes are necessarily the purchaser's responsibility. Although reasonable care has been taken in the preparation of such information, Genium Group, Inc. extends no warranties, makes no representations, and assumes no responsibility as to the accuracy or suitability of such information for application to the purchaser's intended purpose or for consequences of its use.

Material Safety Data Sheet Collection
Genium group inc.
1171 RiverFront Center, Amsterdam, NY 12010
(518) 842-4111

Issue Date: 2006-06

Nitric Acid
NIT1080

Section 1 - Chemical Product and Company Identification 61

Material Name: Nitric Acid **CAS Number:** 7697-37-2
Chemical Formula: HNO_3
Structural Chemical Formula: HNO_3
EINECS Number: 231-714-2
ACX Number: X1002177-5
Synonyms: ACIDE NITRIQUE; ACIDO NITRICO; AQUA FORTIS; AZOTIC ACID; AZOTOWY KWAS; ENGRAVER'S ACID; ENGRAVERS ACID; HYDROGEN NITRATE; KYSELINA DUSICNE; NITAL; NITRIC ACID; NITRIC ACID OTHER THAN RED FUMING WITH >70% NITRIC ACID; NITRIC ACID OTHER THAN RED FUMING WITH NOT >70% NITRICACID; NITROUS FUMES; NITRYL HYDROXIDE; RED FUMING NITRIC ACID (RFNA); SALPETERSAURE; SALPETERZUUROPLOSSINGEN; WHITE FUMING NITRIC ACID (WFNA)
General Use: Manufacture of organic and inorganic nitrates and nitro compounds for fertilizers, dye intermediates and many organic chemicals.
Used for etching and cleaning metals.
Operators should be trained in procedures for safe use of this material.

Section 2 - Composition / Information on Ingredients

Name	CAS	%
nitric acid	7697-37-2	>95

OSHA PEL
TWA: 2 ppm; 5 mg/m^3.

ACGIH TLV
TWA: 2 ppm; STEL: 4 ppm.

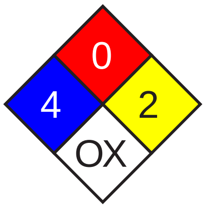
EU OEL
STEL: 2.6 mg/m^3 (1 ppm).

NIOSH REL
TWA: 2 ppm (5 mg/m^3); STEL: 4 ppm (10 mg/m^3).

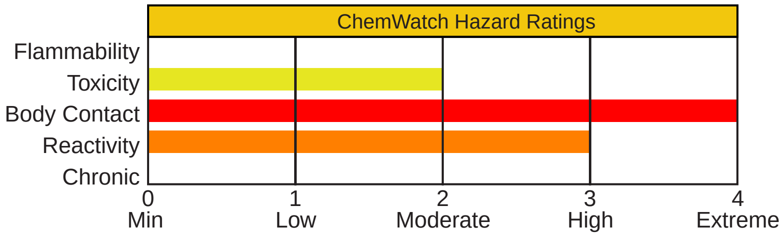
IDLH Level
25 ppm.

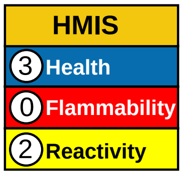
DFG (Germany) MAK
TWA: 2 ppm; PEAK: 2 ppm.

Section 3 - Hazards Identification



Fire Diamond





ANSI Signal Word
Danger!


Corrosive

☆☆☆☆☆ Emergency Overview ☆☆☆☆☆

Clear to yellow fuming liquid; acrid, suffocating odor. Corrosive. Other Acute Effects: lung damage. Chronic Effects: tooth erosion, bronchitis. Strong oxidizer.

Potential Health Effects

Target Organs: eyes, skin, respiratory system, teeth
Primary Entry Routes: inhalation, ingestion, skin contact, eye contact
Acute Effects
Inhalation: The vapor is extremely discomforting and corrosive to the upper respiratory tract and lungs and the material presents a hazard from a single acute exposure or from repeated exposures over long periods.
Inhalation hazard is increased at higher temperatures.
Reactions may occur following a single acute exposure or may only appear after repeated exposures.

Reactions may not occur on exposure but response may be delayed with symptoms only appearing many hours later.

The material may produce respiratory tract irritation which produces an inflammatory response involving the recruitment and activation of many cell types, mainly derived from the vascular system. Unlike most organs the lung can respond to a chemical insult or agent by first trying to remove or neutralize the irritant and then repairing the damage. The repair process, which initially developed to protect mammalian lungs from foreign matter and antigens, may however, cause further damage the lungs when activated by hazardous chemicals. The result is often the impairment of gas exchange, the primary function of the lungs.

Inhalation of nitric acid mist or fumes at 2 to 25 ppm over an 8 hour period may cause pulmonary irritation and symptoms of lung damage.

Only several minutes of exposure to concentrated atmosphere i.e. 200 ppm may cause severe pulmonary damage and even fatality. Death may be delayed for several days.

Exposure to nitric acid fumes (with concurrent inhalation of nitrogen dioxide and nitric oxide) may elicit prompt irritation of the upper respiratory tract leading to coughing, gagging, chest pain, dyspnea, cyanosis if concentrations are sufficiently high and duration of exposure sufficiently long, pulmonary edema.

Eye: The liquid is extremely corrosive to the eyes and contact may cause rapid tissue destruction and is capable of causing severe damage with loss of sight.

The vapor is extremely discomforting to the eyes and is capable of causing pain and severe conjunctivitis.

Corneal injury may develop, with possible permanent impairment of vision, if not promptly and adequately treated.

The material may produce moderate eye irritation leading to inflammation.

Repeated or prolonged exposure to irritants may produce conjunctivitis.

Eye contact with concentrated acid may give no pain, whilst diluted solution causes intense pain and both can cause permanent eye damage or blindness. Burns may result in shrinkage of the eyeball, symblepharon (adhesions between tarsal and bulbar conjunctivae), permanent corneal opacification, and visual impairment leading to blindness.

Skin: The liquid is extremely corrosive to the skin and contact may cause tissue destruction with severe burns.

Bare unprotected skin should not be exposed to this material.

The vapor is highly discomforting to the skin.

The material may cause skin irritation after prolonged or repeated exposure and may produce a contact dermatitis (nonallergic). This form of dermatitis is often characterized by skin redness (erythema) and swelling (edema) which may progress to vesiculation, scaling and thickening of the epidermis. Histologically there may be intercellular edema of the spongy layer (spongiosis) and intracellular edema of the epidermis.

Skin contact causes yellow discoloration of the skin, blisters and scars that may not heal. The skin may be stained bright-yellow or yellowish brown due to the formation of xanthoproteic acid. Dilute solutions may harden the epithelium without producing overt corrosion.

Ingestion: Considered an unlikely route of entry in commercial/industrial environments.

The material is extremely corrosive if swallowed and is capable of causing burns to mouth, throat, esophagus, with extreme discomfort, pain and may be fatal.

Even a small amount causes severe corrosion of the stomach, burning pain, vomiting and shock, possibly causing non-healing scarring of the gastrointestinal tract and stomach. Death may be delayed 12 hours to 14 days or to several months. Such late fatalities are attributed to a chemical lobular pneumonitis secondary to aspiration. Survivors show stricture of the gastric mucosa and subsequent pernicious anemia.

Carcinogenicity: NTP - Not listed; IARC - Not listed; OSHA - Not listed; NIOSH - Not listed; ACGIH - Not listed; EPA - Not listed; MAK - Not listed.

Chronic Effects: Prolonged or repeated overexposure to low concentrations of vapor may cause chronic bronchitis, corrosion of teeth, even chemical pneumonitis.

Section 4 - First Aid Measures

See DOT ERG

Inhalation: Remove to fresh air.

Lay patient down. Keep warm and rested.

If available, administer medical oxygen by trained personnel.

If breathing is shallow or has stopped, ensure clear airway and apply resuscitation. Transport to hospital or doctor, without delay.

Eye Contact: Immediately hold the eyes open and flush continuously for at least 15 minutes with fresh running water. Ensure irrigation under eyelids by occasionally lifting the upper and lower lids.

Transport to hospital or doctor without delay. Removal of contact lenses after an eye injury should only be undertaken by skilled personnel.

Immediately transport to hospital or doctor. DO NOT delay.

Skin Contact: Immediately flush body and clothes with large amounts of water, using safety shower if available.

Quickly remove all contaminated clothing, including footwear.

Wash affected areas with water (and soap if available) for at least 15 minutes. Transport to hospital or doctor. DO NOT delay.

Ingestion: Contact a Poison Control Center.

Do NOT induce vomiting. Give a glass of water.

Immediately transport to hospital or doctor. DO NOT delay.

After first aid, get appropriate in-plant, paramedic, or community medical support.

Note to Physicians: For acute or short-term repeated exposures to strong acids:

1.Airway problems may arise from laryngeal edema and inhalation exposure.
Treat with 100% oxygen initially.
2.Respiratory distress may require cricothyroidotomy if endotracheal intubation is contraindicated by excessive swelling.
3. Intravenous lines should be established immediately in all cases where there is evidence of circulatory compromise.
4.Strong acids produce a coagulation necrosis characterized by formation of a coagulum (eschar) as a result of the desiccating action of the acid on proteins in specific tissues.

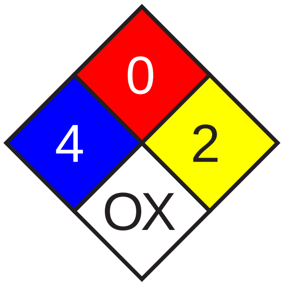
INGESTION:
1.Immediate dilution (milk or water) within 30 minutes post-ingestion is recommended.
2.Do not attempt to neutralize the acid since exothermic reaction may extend the corrosive injury.
3.Be careful to avoid further vomiting since re-exposure of the mucosa to the acid is harmful. Limit fluids to one or two glasses in an adult.
4.Charcoal has no place in acid management.
5.Some authors suggest the use of lavage within 1 hour of ingestion.

SKIN:
1.Skin lesions require copious saline irrigation. Treat chemical burns as thermal burns with non-adherent gauze and wrapping.
2.Deep second-degree burns may benefit from topical silver sulfadiazine.

EYE:
1.Eye injuries require retraction of the eyelids to ensure thorough irrigation of the conjunctival cul-de-sacs. Irrigation should last at least 20-30 minutes. Do not use neutralizing agents or any other additives. Several liters of saline are required.
2.Cycloplegic drops (1% cyclopentolate for short-term use or 5% homatropine for longer term use), antibiotic drops, vasoconstrictive agents, or artificial tears may be indicated dependent on the severity of the injury.
3.Steroid eye drops should only be administered with the approval of a consulting ophthalmologist.

Section 5 - Fire-Fighting Measures

See DOT ERG



Fire Diamond

Flash Point: Nonflammable
Autoignition Temperature: Not applicable
LEL: Not applicable
UEL: Not applicable

Extinguishing Media: Water spray or fog; foam, dry chemical powder, or BCF (where regulations permit).
Carbon dioxide.

General Fire Hazards/Hazardous Combustion Products: Will not burn but increases intensity of fire.
Heating may cause expansion or decomposition leading to violent rupture of containers.
Heat affected containers remain hazardous.
Contact with combustibles such as wood, paper, oil or finely divided metal may cause ignition, combustion or violent decomposition.
May emit irritating, poisonous or corrosive fumes.
Decomposes on heating and produces toxic fumes of nitrogen oxides (NO_x) and nitric acid.

Fire Incompatibility: Oxidizing agents as a class are not necessarily combustible themselves, but can increase the risk and intensity of fire in many other substances.
Reacts vigorously with water and alkali.
Avoid reaction with organic materials/compounds, powdered metals, reducing agents and hydrogen sulfide (H_2S) as ignition may result.
Reacts with metals producing flammable/explosive hydrogen gas.

Fire-Fighting Instructions: Contact fire department and tell them location and nature of hazard.
May be violently or explosively reactive. Wear full body protective clothing with breathing apparatus. Prevent, by any means available, spillage from entering drains or waterways. Consider evacuation.
Fight fire from a safe distance, with adequate cover.
Extinguishers should be used only by trained personnel.
Use water delivered as a fine spray to control fire and cool adjacent area.
Avoid spraying water onto liquid pools.
Do not approach containers suspected to be hot.
Cool fire-exposed containers with water spray from a protected location.
If safe to do so, remove containers from path of fire.
If fire gets out of control withdraw personnel and warn against entry.
Equipment should be thoroughly decontaminated after use.

Section 6 - Accidental Release Measures

See
DOT
ERG

Small Spills: Dangerous levels of nitrogen oxides may form during spills of nitric acid.
Wear fully protective PVC clothing and breathing apparatus.
Clean up all spills immediately. No smoking, bare lights, ignition sources.
Avoid all contact with any organic matter including fuel, solvents, sawdust, paper or cloth and other incompatible materials, as ignition may result.
Avoid breathing dust or vapors and all contact with skin and eyes.
Control personal contact by using protective equipment.
Contain and absorb spill with dry sand, earth, inert material or vermiculite. DO NOT use sawdust as fire may result.
Scoop up solid residues and seal in labeled drums for disposal.
Neutralize/decontaminate area.
Use soda ash or slaked lime to neutralize.

Large Spills: DO NOT touch the spill material. Restrict access to area.
Clear area of personnel and move upwind. Contact fire department and tell them location and nature of hazard.
May be violently or explosively reactive. Wear full body protective clothing with breathing apparatus. Prevent, by any means available, spillage from entering drains or waterways. Consider evacuation.
No smoking, flames or ignition sources. Increase ventilation.
Contain spill with sand, earth or other clean, inert materials.
NEVER use organic absorbents such as sawdust, paper, cloth; as fire may result. Avoid any contamination by organic matter.
Use spark-free and explosion-proof equipment.
Collect any recoverable product into labeled containers for possible recycling. DO NOT mix fresh with recovered material.
Collect residues and seal in labeled drums for disposal.
Wash area and prevent runoff into drains. Decontaminate equipment and launder all protective clothing before storage and reuse.
If contamination of drains or waterways occurs advise emergency services.
DO NOT USE WATER OR NEUTRALIZING AGENTS INDISCRIMINATELY ON LARGE SPILLS.

Regulatory Requirements: Follow applicable OSHA regulations (29 CFR 1910.120).

Section 7 - Handling and Storage

Handling Precautions: Avoid generating and breathing mist. Do not allow clothing wet with material to stay in contact with skin.
Avoid all personal contact, including inhalation.
Wear protective clothing when risk of exposure occurs.
Use in a well-ventilated area.
WARNING: To avoid violent reaction, ALWAYS add material to water and NEVER water to material.
Avoid smoking, bare lights or ignition sources.
Avoid contact with incompatible materials.
When handling, DO NOT eat, drink or smoke.
Keep containers securely sealed when not in use. Avoid physical damage to containers. Always wash hands with soap and water after handling. Work clothes should be laundered separately.
Launder contaminated clothing before reuse.
Use good occupational work practices. Observe manufacturer's storing and handling recommendations. Atmosphere should be regularly checked against established exposure standards to ensure safe working conditions are maintained.

Recommended Storage Methods: Stainless steel drum. Check that containers are clearly labeled.
Packaging as recommended by manufacturer.

Regulatory Requirements: Follow applicable OSHA regulations.

Section 8 - Exposure Controls / Personal Protection

Engineering Controls: Use in a well-ventilated area.
Local exhaust ventilation may be required for safe working, i. e. , to keep exposures below required standards; otherwise, PPE is required.
If risk of overexposure exists, wear NIOSH-approved respirator.
Correct fit is essential to obtain adequate protection.
In confined spaces where there is inadequate ventilation, wear full-face air supplied breathing apparatus.

Personal Protective Clothing/Equipment:

Eyes: Chemical goggles. Full face shield.
DO NOT wear contact lenses. Contact lenses pose a special hazard; soft contact lenses may absorb irritants and all lenses concentrate them.

Hands/Feet: Bare unprotected skin should not be exposed to this material. Impervious, gauntlet length gloves i.e., butyl rubber gloves or Neoprene rubber gloves or wear chemical protective gloves, e.g. PVC.

Wear safety footwear or safety gumboots, e.g. Rubber.

Respiratory Protection:

Exposure Range >2 to <25 ppm: Supplied Air, Constant Flow/Pressure Demand, Half Mask

Exposure Range 25 to unlimited ppm: Self-contained Breathing Apparatus, Pressure Demand, Full Face

Other: Operators should be trained in procedures for safe use of this material.

Acid-resistant overalls or Rubber apron or PVC apron.

Ensure there is ready access to an emergency shower.

Ensure that there is ready access to eye wash unit.

Ensure that there is ready access to breathing apparatus.

Glove Selection Index:

BUTYL	Best selection
HYPALON	Best selection
NEOPRENE	Best selection
NEOPRENE/NATURAL	Best selection
PE/EVAL/PE	Best selection
SARANEX-23	Best selection
NATURAL RUBBER	Satisfactory; may degrade after 4 hours continuous immersion
NATURAL+NEOPRENE	Satisfactory; may degrade after 4 hours continuous immersion
PVC	Poor to dangerous choice for other than short-term immersion
NITRILE+PVC	Poor to dangerous choice for other than short-term immersion

Section 9 - Physical and Chemical Properties

Appearance/General Info: Clear, colorless to slightly yellow liquid. Sharp strong odor.
CAUTION: exothermic dilution hazard.
HIGHLY CORROSIVE. Corrosive to most metals. Powerful oxidizing agent.
Darkens to brownish color on aging and exposure to light.

Physical State: Liquid
Odor Threshold: 0.75 to 2.50 mg/m^3
Vapor Pressure (kPa): 8.26
Vapor Density (Air=1): 1.5
Formula Weight: 63.02
Specific Gravity (H_2O=1, at 4 °C): 1.3-1.42

pH (1% Solution): 1
Boiling Point: 83 °C (181 °F) at 760 mm Hg
Freezing/Melting Point: -42 °C (-43.6 °F)
Volatile Component (% Vol): 100 (nominal)
Decomposition Temperature (°C): Not applicable
Water Solubility: Soluble in all proportions

pH: < 1

Section 10 - Stability and Reactivity

Stability/Polymerization/Conditions to Avoid: Presence of heat source and direct sunlight. Storage in unsealed containers. Hazardous polymerization will not occur.

Storage Incompatibilities: Segregate from reducing agents, finely divided combustible materials, combustible materials, sawdust, metals and powdered metals.

Avoid contamination of water, foodstuffs, feed or seed.

Segregate from alkalies, oxidizing agents and chemicals readily decomposed by acids, i.e. cyanides, sulfides, carbonates.

Section 11 - Toxicological Information

Toxicity

Oral (human) LD_{Lo}: 430 mg/kg
Inhalation (rat) LC_{50}: 2500 ppm/1 hr
Unreported (man) LD_{Lo}: 110 mg/kg

Irritation

Nil reported

See *RTECS* QU 5775000, for additional data.

Section 12 - Ecological Information

Environmental Fate: No data found.

Ecotoxicity: LC_{50} Starfish 100-300 mg/l/48 hr /Aerated water conditions; LC_{50} Shore crab 180 mg/l/48 hr /Static, aerated water conditions; LC_{50} Cockle 330-1000 mg/l/48 hr /Aerated water conditions

BCF: no food chain concentration potential

Biochemical Oxygen Demand (BOD): none

Section 13 - Disposal Considerations

Disposal: Recycle wherever possible. Special hazards may exist - specialist advice may be required.
Consult manufacturer for recycling options.
Follow applicable federal, state, and local regulations.
Treat and neutralize at an approved treatment plant.
Decontaminate empty containers. Observe all label safeguards until containers are cleaned and destroyed.
Puncture containers to prevent reuse and bury at an authorized landfill.

Section 14 - Transport Information

DOT Hazardous Materials Table Data (49 CFR 172.101):

Note: This material has multiple possible HMT entries. Choose the appropriate one based on state and condition of specific material when shipped.

Shipping Name and Description: Nitric acid *other than red fuming, with more than 70 percent nitric acid*

ID: UN2031
Hazard Class: 8 - Corrosive material
Packing Group: I - Great Danger
Symbols:
Label Codes: 8 - Corrosive, 5.1 - Oxidizer
Special Provisions: B47, B53, T10, TP2, TP12, TP13
Packaging: **Exceptions:** None **Non-bulk:** 158 **Bulk:** 243
Quantity Limitations: **Passenger aircraft/rail:** Forbidden **Cargo aircraft only:** 2.5 L
Vessel Stowage: **Location:** D **Other:** 44, 66, 89, 90, 110, 111

Shipping Name and Description: Nitric acid *other than red fuming, with not more than 70 percent nitric acid*

ID: UN2031
Hazard Class: 8 - Corrosive material
Packing Group: II - Medium Danger
Symbols:
Label Codes: 8 - Corrosive
Special Provisions: B2, B47, B53, IB2, T8, TP2, TP12
Packaging: **Exceptions:** None **Non-bulk:** 158 **Bulk:** 242
Quantity Limitations: **Passenger aircraft/rail:** Forbidden **Cargo aircraft only:** 30 L
Vessel Stowage: **Location:** D **Other:**

Shipping Name and Description: Nitric acid, red fuming

ID: UN2032
Hazard Class: 8 - Corrosive material
Packing Group: I - Great Danger
Symbols: + - Override definitions
Label Codes: 8 - Corrosive, 5.1 - Oxidizer, 6.1 - Poison *or* Poison Inhalation Hazard *if inhalation hazard, Zone A or B*
Special Provisions: 2, B9, B32, B74, T20, TP2, TP12, TP13, TP38, TP45
Packaging: **Exceptions:** None **Non-bulk:** 227 **Bulk:** 244
Quantity Limitations: **Passenger aircraft/rail:** Forbidden **Cargo aircraft only:** Forbidden
Vessel Stowage: **Location:** D **Other:**

Section 15 - Regulatory Information

EPA Regulations:
- **RCRA 40 CFR:** Not listed
- **CERCLA 40 CFR 302.4:** Listed per CWA Section 311(b)(4) 1000 lb (453.5 kg)
- **SARA 40 CFR 372.65:** Listed
- **SARA EHS 40 CFR 355:** Listed
  - **RQ:** 1000 lb
  - **TPQ:** 1000 lb
- **TSCA:** Listed

Section 16 - Other Information

Issue Date: 2006-06

Sodium Hydroxide
SOD4950

Section 1 - Chemical Product and Company Identification

Material Name: Sodium Hydroxide **CAS Number:** 1310-73-2
Chemical Formula: HNaO
Structural Chemical Formula: NaOH
EINECS Number: 215-185-5
ACX Number: X1000118-8
Synonyms: CAUSTIC SODA; CAUSTIC SODA,BEAD; CAUSTIC SODA,DRY; CAUSTIC SODA,FLAKE; CAUSTIC SODA,GRANULAR; CAUSTIC SODA,SOLID; HYDROXYDE DE SODIUM; LEWIS-RED DEVIL LYE; LYE; NATRIUMHYDROXID; NATRIUMHYDROXYDE; SODA LYE; SODA,CAUSTIC; SODA,HYDRATE; SODIO(IDROSSIDO DI); SODIUM HYDRATE; SODIUM HYDROXIDE; SODIUM HYDROXIDE,BEAD; SODIUM HYDROXIDE,DRY; SODIUM HYDROXIDE,FLAKE; SODIUM HYDROXIDE,GRANULAR; SODIUM HYDROXIDE,SOLID; SODIUM(HYDROXYDE DE); WHITE CAUSTIC
General Use: Component of alkali cleaners. Manufacture of soap, pulp and paper; rayon. Chemical manufacture. Neutralizing agent in petroleum refining; manufacture of aluminum, detergents, textile processing, refining of vegetable oils. Laboratory reagent, for organic fusion, etching of metal. Used for regenerating ion exchange resins, lye peeling of fruits and vegetables in the food industry.

Section 2 - Composition / Information on Ingredients

Name	CAS	%
sodium hydroxide	1310-73-2	>98

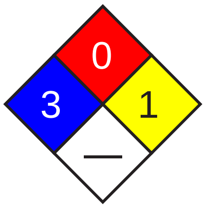
OSHA PEL
TWA: 2 mg/m^3.

NIOSH REL
Ceiling: 2 mg/m^3.

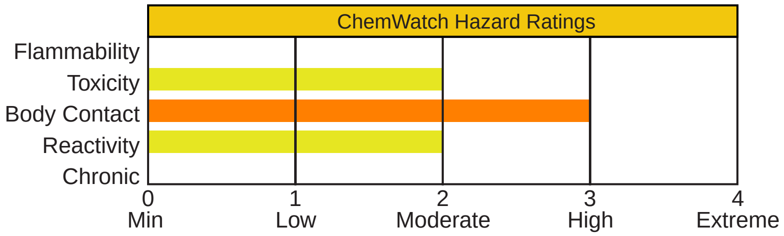
ACGIH TLV
Ceiling: 2 mg/m^3.

IDLH Level
10 mg/m^3.

Section 3 - Hazards Identification



Fire Diamond



HMIS
3 Health
1 Flammability
0 Reactivity

ANSI Signal Word

Danger!

Corrosive

☆☆☆☆☆ Emergency Overview ☆☆☆☆☆

White, odorless, hydroscopic flakes, lumps, or pellets. Corrosive, causes severe burns to eyes/skin/respiratory tract. Chronic Effects: dermatitis. Reacts with water.

Potential Health Effects

Target Organs: eyes, digestive system, respiratory system, skin
Primary Entry Routes: ingestion, inhalation, skin contact, eye contact
Acute Effects
Inhalation: Generated dust may be highly discomforting and corrosive to the upper respiratory tract if inhaled and is capable of causing severe burns to the upper respiratory tract.

The material may produce respiratory tract irritation which produces an inflammatory response involving the recruitment and activation of many cell types, mainly derived from the vascular system. Unlike most organs the lung can respond to a chemical insult or agent by first trying to remove or neutralize the irritant and then repairing the damage. The repair process, which initially developed to protect mammalian lungs from foreign matter and antigens, may however, cause further damage the lungs when activated by hazardous chemicals. The result is often the impairment of gas exchange, the primary function of the lungs.

Severe acute dust inhalation exposure may be fatal due to spasm, inflammation and edema of the larynx and bronchi, chemical pneumonitis and severe pulmonary edema.

Symptoms of overexposure include burning sensation, coughing, wheezing, laryngitis, shortness of breath, headache, nausea and vomiting.

Eye: The solid/dust is extremely corrosive to the eyes and is capable of causing severe damage with loss of sight.

The material may produce severe irritation to the eye causing pronounced inflammation. Repeated or prolonged exposure to irritants may produce conjunctivitis.

Skin: The solid/dust is highly discomforting and extremely corrosive to the skin and is capable of causing severe burns and ulceration.

Bare unprotected skin should not be exposed to this material. The material may produce severe skin irritation after prolonged or repeated exposure, and may produce a contact dermatitis (nonallergic). This form of dermatitis is often characterized by skin redness (erythema) and swelling (edema) which may progress to vesiculation, scaling and thickening of the epidermis. Histologically there may be intercellular edema of the spongy layer (spongiosis) and intracellular edema of the epidermis.

Burns are not immediately painful; onset of pain may be delayed minutes or hours; thus care should be taken to avoid contamination of gloves and boots. A 5% aqueous solution applied to the skin of rabbits for 4 hours produced severe necrosis. Instillation of a 1% solution into the conjunctival sac failed to produce ocular or conjunctival injury in rabbits provided the eye was promptly irrigated with copious amounts of water.

Ingestion: Considered an unlikely route of entry in commercial/industrial environments.

The solid is extremely corrosive to the gastrointestinal tract and may be fatal if swallowed.

Ingestion may result in severe burns to the mouth, throat and stomach, pain, nausea and vomiting, swelling of the larynx and subsequent suffocation, perforation of the gastrointestinal tract.

A 1% aqueous solution (pH 13.4) failed to cause gastric, esophageal or other damage in rabbits.

Carcinogenicity: NTP - Not listed; IARC - Not listed; OSHA - Not listed; NIOSH - Not listed; ACGIH - Not listed; EPA - Not listed; MAK - Not listed.

Chronic Effects: Prolonged contact is unlikely, given the severity of response, but repeated exposures may produce severe ulceration.

Section 4 - First Aid Measures

Inhalation: If dust is inhaled, remove to fresh air. Encourage patient to blow nose to ensure clear breathing passages. Ask patient to rinse mouth with water but to not drink water. Seek immediate medical attention.

See
DOT
ERG

Eye Contact: DO NOT delay. Immediately hold the eyes open and wash continuously for at least 15 minutes with fresh running water. Ensure irrigation under eyelids by occasionally lifting the upper and lower lids. Transport to hospital or doctor without delay. Removal of contact lenses after an eye injury should only be undertaken by skilled personnel.

Skin Contact: DO NOT delay. Immediately flush body and clothes with large amounts of water, using safety shower if available. Quickly remove all contaminated clothing, including footwear. Wash affected areas with water (and soap if available) for at least 15 minutes. Transport to hospital or doctor.

In case of burns: Quickly immerse affected area in cold running water for 10 to 15 minutes. Bandage lightly with a sterile dressing. Treat for shock if required. Lay patient down. Keep warm and rested. Transport to hospital or doctor.

Ingestion: DO NOT delay. Contact a Poison Control Center. If swallowed, do NOT induce vomiting. Give a glass of water.

After first aid, get appropriate in-plant, paramedic, or community medical support.

Note to Physicians: For acute or short-term repeated exposures to highly alkaline materials:

1. Respiratory stress is uncommon but presents occasionally because of soft tissue edema.
2. Unless endotracheal intubation can be accomplished under direct vision, cricothyroidotomy or tracheotomy may be necessary.
3. Oxygen is given as indicated.
4. The presence of shock suggests perforation and mandates an intravenous line and fluid administration.
5. Alkali corrosives damage occurs by liquefaction necrosis whereby the saponification of fats and solubilization of proteins allow deep penetration into the tissue. Alkalis continue to cause damage after exposure.

INGESTION:

1. Milk and water are the preferred diluents. No more than 2 glasses of water should be given to an adult.
2. Neutralizing agents should never be given since exothermic heat reaction may compound injury.

* Catharsis and emesis are absolutely contra-indicated.

* Activated charcoal does not absorb alkali.
* Gastric lavage should not be used.
Supportive care involves the following.

1. Withhold oral feedings initially.
2. If endoscopy confirms transmucosal injury start steroids only within the first 48 hours.
3. Carefully evaluate the amount of tissue necrosis before assessing the need for surgical intervention.
4. Patients should be instructed to seek medical attention whenever they develop difficulty in swallowing (dysphagia).

SKIN AND EYE: Injury should be irrigated for 20-30 minutes. Eye injuries require saline.

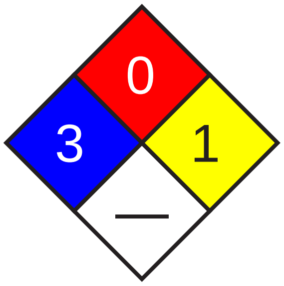
Section 5 - Fire-Fighting Measures

Extinguishing Media: Use extinguishing media suitable for surrounding area.

General Fire Hazards/Hazardous Combustion Products: Noncombustible.
Not considered to be a significant fire risk, however containers may burn.
Solid in contact with water or moisture reacts violently, and solutions are highly alkaline and may cause severe skin burns.

Fire Incompatibility: Avoid reaction with strong oxidizers, strong acids, organic materials/compounds.
In presence of moisture, the material is corrosive to aluminum, zinc and tin producing highly flammable hydrogen gas.

See DOT ERG



Fire Diamond

Fire-Fighting Instructions: Contact fire department and tell them location and nature of hazard.
Wear full body protective clothing with breathing apparatus. Prevent, by any means available, spillage from entering drains or waterways.
Use fire fighting procedures suitable for surrounding area.
Do not approach containers suspected to be hot.
Cool fire exposed containers with water spray from a protected location.
If safe to do so, remove containers from path of fire.
Avoid spraying water onto liquid pools.
Equipment should be thoroughly decontaminated after use.

Section 6 - Accidental Release Measures

Small Spills: DO NOT touch the spill material. Slippery when spilt.
Clean up all spills immediately.
Control personal contact by using protective equipment.
Use dry clean up procedures and avoid generating dust.
Place in suitable containers for disposal.

See DOT ERG

Large Spills: DO NOT touch the spill material. Slippery when spilt.
Keep dry. Reacts violently with water.
Clear area of personnel and move upwind.
Contact fire department and tell them location and nature of hazard.
Wear full body protective clothing with breathing apparatus. Prevent, by any means available, spillage from entering drains or waterways.
Shut off all possible sources of ignition and increase ventilation.
Stop leak if safe to do so.
Use dry clean up procedures and avoid generating dust. Collect recoverable product into labeled containers for recycling. Collect residues and seal in labeled drums for disposal.
Wash area down with large quantity of water and prevent runoff into drains.
If contamination of drains or waterways occurs, advise emergency services.
After clean up operations, decontaminate and launder all protective clothing and equipment before storing and reusing.

Regulatory Requirements: Follow applicable OSHA regulations (29 CFR 1910.120).

Section 7 - Handling and Storage

Handling Precautions: Avoid generating and breathing dust. Avoid contact with skin and eyes.
Wear protective clothing when risk of exposure occurs.
Use in a well-ventilated area.
Local exhaust ventilation may be required for safe working, i.e. to keep exposures below required standards, otherwise PPE is required.
Handle and open container with care.
Keep dry. Reacts violently with water.
WARNING: Contact with water generates heat.

Avoid contact with incompatible materials.
Avoid physical damage to containers.
Keep containers securely sealed when not in use.
Always wash hands with soap and water after handling. Work clothes should be laundered separately. Launder contaminated clothing before reuse. Use good occupational work practice. Observe manufacturer's storing and handling recommendations.
Atmosphere should be regularly checked against established exposure standards to ensure safe working conditions are maintained.

Recommended Storage Methods: Plastic bag or Packaging as recommended by manufacturer. Glass container.
Polyethylene or polypropylene container or Polylined drum.
DO NOT use aluminum, galvanized or tin-plated containers.
Check that containers are clearly labeled.

Storage Requirements: Keep dry. Reacts violently with water.
Store in original containers. Keep containers securely sealed. Store in a cool, dry, well-ventilated area. Store away from incompatible materials and foodstuff containers. Protect containers against physical damage and check regularly for leaks.
Observe manufacturer's storing and handling recommendations.
DO NOT use aluminum, galvanized or tin-plated containers.

Regulatory Requirements: Follow applicable OSHA regulations.

Section 8 - Exposure Controls / Personal Protection

Engineering Controls: Use in a well-ventilated area DO NOT handle directly. Wear gloves and use scoop/tongs/tools.
If risk of overexposure exists, wear NIOSH approved respirator.
If conditions are such that worker exposure potential is high, wear full-face air-supplied breathing apparatus and full protective suit.

Personal Protective Clothing/Equipment:

Eyes: Safety glasses with side shields Chemical goggles. Full face shield.
Contact lenses pose a special hazard; soft lenses may absorb irritants and all lenses concentrate them.

Hands/Feet: DO NOT handle directly. Wear gloves and use scoop/tongs/tools.
Elbow length PVC gloves or Butyl rubber gloves or Neoprene rubber gloves.
Safety footwear.

Respiratory Protection:
Exposure Range >2 to <10 mg/m^3: Air Purifying, Negative Pressure, Half Mask
Exposure Range 10 to unlimited mg/m^3: Self-contained Breathing Apparatus, Pressure Demand, Full Face
Cartridge Color: dust/mist filter (use P100 or consult supervisor for appropriate dust/mist filter)

Other: Overalls. PVC apron. PVC protective suit may be required if exposure severe.
Eyewash unit. Ensure there is ready access to a safety shower.

Section 9 - Physical and Chemical Properties

Appearance/General Info: White hygroscopic, odorless, pellets, flakes, sticks or solid cast mass. Explosive boiling and spitting will occur if added to hot water. Reacts violently with acids. CAUSTIC alkali. Soluble in alcohol, ether, glycerol.

Physical State: Divided solid
Vapor Pressure (kPa): Negligible
Formula Weight: 40
Specific Gravity (H_2O=1, at 4 °C): 2.12 at 20 °C
pH: Not applicable
pH (1% Solution): 12.7
Boiling Point: 1390 °C (2534 °F)
Freezing/Melting Point: 318.4 °C (605.12 °F)
Water Solubility: 1 g dissolves in 0.9 ml water

Section 10 - Stability and Reactivity

Stability/Polymerization/Conditions to Avoid: Vigorously exotherms when mixed with water. In the presence of moisture, highly corrosive to aluminum, zinc and tin.
HIGHLY reactive: with ammonium salts evolves ammonia gas. Rapidly picks up moisture from the air and with carbon dioxide in air forms sodium carbonate.
Presence of incompatible materials and storage in unsealed containers.
Product is considered stable under normal handling conditions.
Hazardous polymerization will not occur.

Storage Incompatibilities: Keep dry. Reacts violently with water.
Segregate from water, strong oxidizers, strong acids, organic materials, ammonium compounds, nitro compounds and trichlorethylene.

Section 11 - Toxicological Information

Toxicity
No data reported

Irritation
Skin (rabbit): 500 mg/24h SEVERE
Eye (rabbit): 0.05 mg/24h SEVERE
Eye(rabbit):1 mg/24h SEVERE
Eye(rabbit):1 mg/30s rinsed-SEVERE

See *RTECS* WB 4900000, for additional data.

Section 12 - Ecological Information

Environmental Fate: No data found.
Ecotoxicity: LC_{100} Cyprinus carpio 180 ppm/24 hr at 25 deg; TL_m mosquito fish 125 ppm/96 hr (freshwater) ; TL_m Bluegill 99 mg/L/48 hr (tap water)
Biochemical Oxygen Demand (BOD): none
Octanol/Water Partition Coefficient: log K_{ow} = too low to be measured

Section 13 - Disposal Considerations

Disposal: Recycle wherever possible or consult manufacturer for recycling options.
Follow applicable federal, state, and local regulations.
Treat and neutralize with dilute acid at an effluent treatment plant.
Recycle containers, otherwise dispose of in an authorized landfill.

Section 14 - Transport Information

DOT Hazardous Materials Table Data (49 CFR 172.101):

Shipping Name and Description: Sodium hydroxide, solid
ID: UN1823
Hazard Class: 8 - Corrosive material
Packing Group: II - Medium Danger
Symbols:
Label Codes: 8 - Corrosive
Special Provisions: IB8, IP2, IP4
Packaging: **Exceptions:** 154 **Non-bulk:** 212 **Bulk:** 240
Quantity Limitations: **Passenger aircraft/rail:** 15 kg **Cargo aircraft only:** 50 kg
Vessel Stowage: **Location:** A **Other:**

Section 15 - Regulatory Information

EPA Regulations:
RCRA 40 CFR: Not listed
CERCLA 40 CFR 302.4: Listed per CWA Section 311(b)(4) 1000 lb (453.5 kg)
SARA 40 CFR 372.65: Not listed
SARA EHS 40 CFR 355: Not listed
TSCA: Listed

Section 16 - Other Information

Disclaimer: Judgments as to the suitability of information herein for the purchaser's purposes are necessarily the purchaser's responsibility. Although reasonable care has been taken in the preparation of such information, Genium Group, Inc. extends no warranties, makes no representations, and assumes no responsibility as to the accuracy or suitability of such information for application to the purchaser's intended purpose or for consequences of its use.

Material Safety Data Sheet Collection

Genium group inc.

1171 RiverFront Center, Amsterdam, NY 12010
(518) 842-4111

Issue Date: 2006-06

Sulfuric Acid
SUL7960

Section 1 - Chemical Product and Company Identification 61

Material Name: Sulfuric Acid **CAS Number:** 7664-93-9

Chemical Formula: H_2O_4S

Structural Chemical Formula: H_2SO_4

EINECS Number: 231-639-5

ACX Number: X1002217-4

Synonyms: ACIDE SULFURIQUE; ACIDO SOLFORICO; ACIDO SULFURICO; BATTERY ACID; BOV; DIHYDROGEN SULFATE; DIPPING ACID; ELECTROLYTE ACID; EPA PESTICIDE CHEMICAL CODE 078001; HYDROGEN SULFATE; MATTLING ACID; OIL OF VITRIOL; SCHWEFELSAEURELOESUNGEN; SULFURIC ACID; SULFURIC ACID (AQUEOUS); SULFURIC ACID,SPENT; SULPHURIC ACID; VITRIOL BROWN OIL; ZWAVELZUUROPLOSSINGEN

General Use: The manufacture of superphosphate fertilizer, inorganic and petro-chemicals, explosives and pigments.
Component of heavy duty metal cleaners, pickles.
In manufacture of rayon, cellulose film.
As battery electrolyte and also in electroplating processes.

Section 2 - Composition / Information on Ingredients

Name	CAS	%
sulfuric acid	7664-93-9	>51
water	7732-18-5	remainder

OSHA PEL
TWA: 1 mg/m^3.

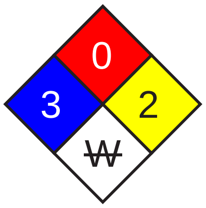
ACGIH TLV
TWA: 1 mg/m^3; STEL: 3 mg/m^3; A2 = as contained in strong inorganic acid mists.

NIOSH REL
TWA: 1 mg/m^3.

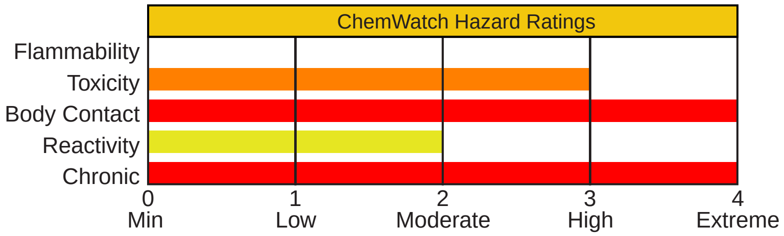
IDLH Level
15 mg/m^3.

DFG (Germany) MAK
TWA: 0.1 mg/m^3; PEAK: 0.1 mg/m^3; Ceiling: 0.2 mg/m^3; measured as inhalable fraction of the aerosol.

Section 3 - Hazards Identification



Fire Diamond



HMIS
③ Health
⓪ Flammability
② Reactivity

ANSI Signal Word
Danger!

Corrosive

☆☆☆☆☆ Emergency Overview ☆☆☆☆☆

Colorless to dark-brown, oily, odorless liquid. Corrosive. Other Acute Effects: blindness. Chronic Effects: tooth erosion, GI disturbances, dermatitis. Reaction with water produces excessive heat.

Potential Health Effects

Target Organs: respiratory system, eyes, skin, teeth

Primary Entry Routes: inhalation, skin contact, eye contact

Acute Effects

Inhalation: The vapor is extremely discomforting to the upper respiratory tract and is capable of causing severe mucous membrane irritation, upper respiratory tract inflammation.
Exposure to high concentrations causes bronchitis and is characterized by the onset of hemorrhagic pulmonary edema.

Mists are highly irritating to eyes, mucous membranes and respiratory tract and high mist concentrations may lead to pulmonary edema.

Eye: HIGHLY CORROSIVE The liquid is extremely corrosive to the eyes and any contact may cause rapid tissue destruction and is capable of causing severe damage with loss of sight.

The mist is highly corrosive and contact may cause rapid tissue destruction.

The vapor is extremely discomforting to the eyes.

The material may produce severe irritation to the eye causing pronounced inflammation. Repeated or prolonged exposure to irritants may produce conjunctivitis.

Skin: HIGHLY CORROSIVE. The liquid is extremely corrosive to the skin and any contact may cause rapid tissue destruction with severe burns.

The mist is highly discomforting to the skin and may cause deep ulceration to body tissue.

Topical application of a 10% solution to skin on the scapula or waist produces only negligible evidence of irritation.

Ingestion: HIGHLY CORROSIVE and Considered toxic by all exposure routes.

The liquid is extremely corrosive and may rapidly cause severe burns to the gastrointestinal tract and may be fatal if swallowed in quantity.

Considered an unlikely route of entry in commercial/industrial environments.

Carcinogenicity: NTP - Not listed; IARC - Group 1, Carcinogenic to humans; OSHA - Not listed; NIOSH - Not listed; ACGIH - Class A2, Suspected human carcinogen; EPA - Not listed; MAK - Not listed.

Chronic Effects: Repeated minor exposure to mists can cause erosion of teeth and inflammation of the upper respiratory tract leading to chronic bronchitis.

Repeated skin contact with dilute solutions may cause dermatitis.

Lungs of sulfuric acid plant workers appear to be less affected than the lungs of workers exposed to "dust".

There is evidence that the corrosion of tooth enamel occurs at 1 mg/m^3 but that acclimatized workers could tolerate three to four times that level. Forming room workers in a battery factory exposed to 3 to 16 mg/m^3 sulfuric acid mist concentrations exhibited the most serious signs of erosion whilst charging room workers exposed to 0.08 to 2.5 mg/m^3 were affected to a lesser degree.

Workers chronically exposed to sulfuric acid mists may show various skin lesions, tracheobronchitis, stomatitis, conjunctivitis and gastritis.

Increased risk of laryngeal cancer is associted with chronic exposures.

Section 4 - First Aid Measures

Inhalation: Remove to fresh air.

Lay patient down. Keep warm and rested.

If available, administer medical oxygen by trained personnel.

If breathing is shallow or has stopped, ensure clear airway and apply resuscitation. Transport to hospital or doctor, without delay.

See DOT ERG

Eye Contact: Immediately hold the eyes open and flush continuously for at least 15 minutes with fresh running water. Ensure irrigation under eyelids by occasionally lifting the upper and lower lids.

Transport to hospital or doctor without delay. Removal of contact lenses after an eye injury should only be undertaken by skilled personnel.

Skin Contact: Immediately flush body and clothes with large amounts of water, using safety shower if available.

Quickly remove all contaminated clothing, including footwear.

Wash affected areas with water (and soap if available) for at least 15 minutes. Transport to hospital or doctor.

DO NOT attempt to neutralize burns with alkaline solutions.

Ingestion: Rinse mouth out with plenty of water.

Contact a Poison Control Center.

Do NOT induce vomiting. Give a glass of water.

After first aid, get appropriate in-plant, paramedic, or community medical support.

Note to Physicians: For acute or short-term repeated exposures to strong acids:

1.Airway problems may arise from laryngeal edema and inhalation exposure.

Treat with 100% oxygen initially.

2.Respiratory distress may require cricothyroidotomy if endotracheal intubation is contraindicated by excessive swelling.

3. Intravenous lines should be established immediately in all cases where there is evidence of circulatory compromise.

4.Strong acids produce a coagulation necrosis characterized by formation of a coagulum (eschar) as a result of the desiccating action of the acid on proteins in specific tissues.

INGESTION:

1.Immediate dilution (milk or water) within 30 minutes post-ingestion is recommended.

2.Do not attempt to neutralize the acid since exothermic reaction may extend the corrosive injury.

3.Be careful to avoid further vomiting since re-exposure of the mucosa to the acid is harmful. Limit fluids to one or two glasses in an adult.

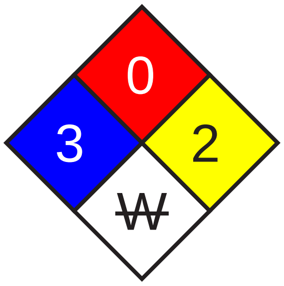
4.Charcoal has no place in acid management.

5.Some authors suggest the use of lavage within 1 hour of ingestion.

SKIN:
1.Skin lesions require copious saline irrigation. Treat chemical burns as thermal burns with non-adherent gauze and wrapping.
2.Deep second-degree burns may benefit from topical silver sulfadiazine.
EYE:
1.Eye injuries require retraction of the eyelids to ensure thorough irrigation of the conjunctival cul-de-sacs. Irrigation should last at least 20-30 minutes. Do not use neutralizing agents or any other additives. Several liters of saline are required.
2.Cycloplegic drops (1% cyclopentolate for short-term use or 5% homatropine for longer term use), antibiotic drops, vasoconstrictive agents, or artificial tears may be indicated dependent on the severity of the injury.
3.Steroid eye drops should only be administered with the approval of a consulting ophthalmologist.

Section 5 - Fire-Fighting Measures

See DOT ERG



Fire Diamond

Flash Point: Nonflammable
Autoignition Temperature: Not applicable
LEL: Not applicable
UEL: Not applicable
Extinguishing Media: Use extinguishing media suitable for surrounding area. Water spray or fog, from a safe distance only.
General Fire Hazards/Hazardous Combustion Products: HIGHLY CORROSIVE.
Noncombustible liquid. Reacts vigorously with water.
Heating may cause expansion or decomposition leading to violent rupture of containers.
Contact with readily oxidizable organic material may cause ignition /fire.
Reacts with metals producing flammable/explosive hydrogen gas.
Decomposes on heating and produces acrid and toxic fumes of sulfur oxides (SO_x).
Fire Incompatibility: Reacts with mild steel, galvanized steel/zinc producing hydrogen gas which may form an explosive mixture with air.
Contact with readily oxidizable organic material may cause ignition /fire.
Avoid any contamination of this material as it is very reactive and any contamination is potentially hazardous.
Fire-Fighting Instructions: Contact fire department and tell them location and nature of hazard.
May be violently or explosively reactive. Wear full body protective clothing with breathing apparatus. Prevent, by any means available, spillage from entering drains or waterways. Consider evacuation.
Use water delivered as a fine spray to control fire and cool adjacent area.
Avoid spraying water onto liquid pools.
Do not approach containers suspected to be hot.
Cool fire-exposed containers with water spray from a protected location.
If safe to do so, remove containers from path of fire.
Equipment should be thoroughly decontaminated after use.

Section 6 - Accidental Release Measures

See DOT ERG

Small Spills: Clean up all spills immediately.
Avoid breathing vapors and contact with skin and eyes.
Control personal contact by using protective equipment.
Contain and absorb spill with sand, earth, inert material or vermiculite.
Wipe up. Place in a suitable labeled container for waste disposal.
Use soda ash or slaked lime to neutralize.
Large Spills: DO NOT touch the spill material. Clear area of personnel and move upwind.
Contact fire department and tell them location and nature of hazard.
May be violently or explosively reactive. Wear full body protective clothing with breathing apparatus. Prevent, by any means available, spillage from entering drains or waterways. Consider evacuation.
Stop leak if safe to do so.
Contain spill with sand, earth or vermiculite.
Collect recoverable product into labeled containers for recycling.
Neutralize/decontaminate residue.
Collect solid residues and seal in labeled drums for disposal.
Wash area and prevent runoff into drains.
After clean-up operations, decontaminate and launder all protective clothing and equipment before storing and reusing.
If contamination of drains or waterways occurs, advise emergency services.
DO NOT USE WATER OR NEUTRALIZING AGENTS INDISCRIMINATELY ON LARGE SPILLS.
Regulatory Requirements: Follow applicable OSHA regulations (29 CFR 1910.120).

Section 7 - Handling and Storage

Handling Precautions: Avoid generating and breathing mist. Avoid all personal contact, including inhalation.
Wear protective clothing when risk of exposure occurs.
Use in a well-ventilated area.
WARNING: To avoid violent reaction, ALWAYS add material to water and NEVER water to material.
Avoid smoking, bare lights or ignition sources.
Avoid contact with incompatible materials.
When handling, DO NOT eat, drink or smoke.
Keep containers securely sealed when not in use. Avoid physical damage to containers. Always wash hands with soap and water after handling. Work clothes should be laundered separately.
Launder contaminated clothing before reuse.
Use good occupational work practices. Observe manufacturer's storing and handling recommendations. Atmosphere should be regularly checked against established exposure standards to ensure safe working conditions are maintained.
Recommended Storage Methods: Glass carboy. Glass container is suitable for laboratory quantities.
Plastic carboy. Polylined drum.
Check that containers are clearly labeled.
Packaging as recommended by manufacturer.
DO NOT use mild steel or galvanized containers.
Regulatory Requirements: Follow applicable OSHA regulations.

Section 8 - Exposure Controls / Personal Protection

Engineering Controls: Use in a well-ventilated area.
General exhaust is adequate under normal operating conditions.
Local exhaust ventilation may be required in special circumstances.
If risk of overexposure exists, wear NIOSH-approved respirator. Correct fit is essential to ensure adequate protection.
Provide adequate ventilation in warehouses and enclosed storage areas.
Personal Protective Clothing/Equipment:
Eyes: Chemical goggles. Full face shield.
Contact lenses pose a special hazard; soft lenses may absorb irritants and all lenses concentrate them.
Hands/Feet: Wear chemical protective gloves, eg. PVC.
Wear safety footwear or safety gumboots, eg. Rubber.
Respiratory Protection:
Exposure Range >1 to 10 mg/m^3: Air Purifying, Negative Pressure, Half Mask
Exposure Range >10 to <15 mg/m^3: Air Purifying, Negative Pressure, Full Face
Exposure Range 15 to unlimited mg/m^3: Self-contained Breathing Apparatus, Pressure Demand, Full Face
Cartridge Color: white with dust/mist prefilter (use P100 or consult supervisor for appropriate dust/mist prefilter)
Other: Overalls. PVC apron. PVC protective suit may be required if exposure severe.
Eyewash unit. Ensure there is ready access to a safety shower.
Glove Selection Index:

NATURAL RUBBER	Best selection
NATURAL+NEOPRENE	Best selection
NEOPRENE	Best selection
NEOPRENE/NATURAL	Best selection
NITRILE	Best selection
PE	Best selection
PVC	Best selection
SARANEX-23	Best selection

Section 9 - Physical and Chemical Properties

Appearance/General Info: Colorless, oily, dense, HIGHLY CORROSIVE liquid. Faint acid odor.
Material is a powerful oxidizing and dehydrating agent causing rapid human tissue destruction on contact.
Concentrated acid is very exothermic (generates heat) when mixed with water.
DANGER: Adding water to acid will cause violent steam explosion, scattering corrosive acid. Always add acid slowly to water.
Mixes with alcohol in all proportions. Available in technical, pure and analytical grades

Physical State: Liquid
Odor Threshold: 1.0 mg/m^3
Vapor Pressure (kPa): 0.133 at 146 °C
Vapor Density (Air=1): 3.40
Formula Weight: 98.07
Specific Gravity (H_2O=1, at 4 °C): 1.6-1.84 at 15 °C
Evaporation Rate: Non Vol. at 38 °C
pH: < 1
pH (1% Solution): 1
Boiling Point: About 290 °C (554 °F)
Freezing/Melting Point: 10.36 °C (50.648 °F)
Decomposition Temperature (°C): 340

Water Solubility: Soluble in water

Section 10 - Stability and Reactivity

Stability/Polymerization/Conditions to Avoid: Product is considered stable. Hazardous polymerization will not occur.
Storage Incompatibilities: Segregate from alkalies, oxidizing agents and chemicals readily decomposed by acids, i.e. cyanides, sulfides, carbonates.
Reacts vigorously with water and alkali.
Contact with readily oxidizable organic material may cause ignition /fire.
Avoid contamination of water, foodstuffs, feed or seed.

Section 11 - Toxicological Information

Toxicity
Oral (rat) LD_{50}: 2140 mg/kg
Inhalation (rat) LC_{50}: 510 mg/m^3/2h
Inhalation (human) TC_{Lo}: 3 mg/m^3/24w

Irritation
Eye (rabbit): 1.38 mg SEVERE
Eye (rabbit): 5 mg/30sec SEVERE

See *RTECS* WS 5600000, for additional data.

Section 12 - Ecological Information

Environmental Fate: No data found.
Ecotoxicity: TL_m Lepomis macrochirus (bluegill) 24.5 ppm/24 hr fresh water /Conditions of bioassay not specified; LC_{50} Flounder 100 to 330 mg/l/48 hr aerated water /Conditions of bioassay not specified; LC_{50} Shrimp 80 to 90 mg/l/48 hr aerated water /Conditions of bioassay not specified; LC_{50} Prawn 42.5 ppm/48 hr salt water /Conditions of bioassay not specified
BCF: no food chain concentration potential
Biochemical Oxygen Demand (BOD): none

Section 13 - Disposal Considerations

Disposal: Recycle wherever possible or consult manufacturer for recycling options.
Follow applicable federal, state, and local regulations.
Treat and neutralize at an effluent treatment plant.
Use soda ash or slaked lime to neutralize.
Recycle containers, otherwise dispose of in an authorized landfill.
Bury residue in an authorized landfill.

Section 14 - Transport Information

DOT Hazardous Materials Table Data (49 CFR 172.101):

Shipping Name and Description: Sulfuric acid *with more than 51 percent acid*
ID: UN1830
Hazard Class: 8 - Corrosive material
Packing Group: II - Medium Danger
Symbols:
Label Codes: 8 - Corrosive
Special Provisions: A3, A7, B3, B83, B84, IB2, N34, T8, TP2, TP12
Packaging: **Exceptions:** 154 **Non-bulk:** 202 **Bulk:** 242
Quantity Limitations: **Passenger aircraft/rail:** 1 L **Cargo aircraft only:** 30 L
Vessel Stowage: **Location:** C **Other:** 14

Section 15 - Regulatory Information

EPA Regulations:
RCRA 40 CFR: Not listed
CERCLA 40 CFR 302.4: Listed per CWA Section 311(b)(4) 1000 lb (453.5 kg)
SARA 40 CFR 372.65: Listed
SARA EHS 40 CFR 355: Listed
RQ: 1000 lb
TPQ: 1000 lb

TSCA: Listed

Section 16 - Other Information

Disclaimer: Judgments as to the suitability of information herein for the purchaser's purposes are necessarily the purchaser's responsibility. Although reasonable care has been taken in the preparation of such information, Genium Group, Inc. extends no warranties, makes no representations, and assumes no responsibility as to the accuracy or suitability of such information for application to the purchaser's intended purpose or for consequences of its use.

Material Safety Data Sheet Collection
Genium group inc.
1171 RiverFront Center, Amsterdam, NY 12010
(518) 842-4111

Issue Date: 2006-06

Trichloroethylene
TRI2710

Section 1 - Chemical Product and Company Identification 61

Material Name: Trichloroethylene **CAS Number:** 79-01-6
Chemical Formula: C_2HCl_3
Structural Chemical Formula: $ClCH{=}CCl_2$
EINECS Number: 201-167-4
ACX Number: X1000039-2
Synonyms: ACETYLENE TRICHLORIDE; ALGYLEN; ANAMENTH; BENZINOL; BLACOSOLV; BLANCOSOLV; CECOLENE; CHLORILEN; 1-CHLORO-2,2-DICHLOROETHYLENE; CHLORYLEA; CHLORYLEA,CHORYLEN,CIRCOSOLV,CRAWHASPOL,DOW-TRI,DUKERON,PER-A-CLOR,TRIAD,TRIAL,TRI-PLUS M,VITRAN; CHLORYLEN; CHORYLEN; CIRCOSOLV; CRAWHASPOL; DENSINFLUAT; 1,1-DICHLORO-2-CHLOROETHYLENE; DOW-TRI; DUKERON; EPA PESTICIDE CHEMICAL CODE 081202; ETHENE,TRICHLORO-; ETHINYL TRICHLORIDE; ETHYLENE TRICHLORIDE; ETHYLENE,TRICHLORO-; FLECK-FLIP; FLOCK FLIP; FLUATE; GEMALGENE; GERMALGENE; LANADIN; LETHURIN; NARCOGEN; NARKOGEN; NARKOSOID; NIALK; NSC 389; PERM-A-CHLOR; PERM-A-CLOR; PETZINOL; PHILEX; TCE; THRETHYLEN; THRETHYLENE; TRETHYLENE; TRI; TRIAD; TRIAL; TRIASOL; TRICHLOORETHEEN; TRICHLOORETHYLEEN,TRI; TRICHLORAETHEN; TRICHLORAETHYLEN,TRI; TRICHLORAN; TRICHLOREN; TRICHLORETHENE; TRICHLORETHYLENE; TRICHLORETHYLENE,TRI; TRICHLOROETHENE; 1,1,2-TRICHLOROETHYLENE; TRICHLOROETHYLENE; TRICHLOROETHYLENE; 1,2,2-TRICHLOROETHYLENE; TRI-CLENE; TRICLENE; TRICLORETENE; TRICLOROETILENE; TRIELENE; TRIELIN; TRIELINA; TRIELINE; TRIKLONE; TRILEN; TRILENE; TRILINE; TRIMAR; TRIOL; TRI-PLUS; TRI-PLUS M; VESTROL; VITRAN; WESTROSOL
General Use: Mainly used for vapor degreasing; solvent in textile and electronics industries; for adhesives, lubricants and consumer products (such as spot removers and rug cleaners).
Until recently, it was used to make hop extracts for beer, decaffeinated coffee and spice extracts.

Section 2 - Composition / Information on Ingredients

Name	CAS	%
trichloroethylene	79-01-6	> 99

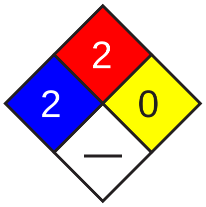
OSHA PEL
TWA: 100 ppm; Ceiling: 200 ppm; 300 ppm, 5-minute maximum peak in any 2 hours.

ACGIH TLV
TWA: 50 ppm; STEL: 100 ppm.

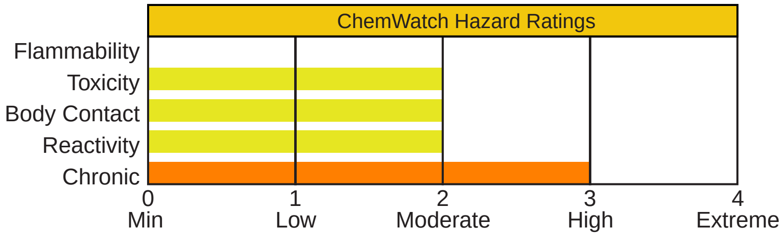
NIOSH REL

IDLH Level
1000 ppm.

Section 3 - Hazards Identification



Fire Diamond



HMIS
2 Health
2 Flammability
0 Reactivity

ANSI Signal Word
Warning!

Flammable

☆☆☆☆☆ Emergency Overview ☆☆☆☆☆

Clear, colorless liquid; sweet odor. Irritating to eyes/skin/respiratory tract. Other Acute Effects: irregular heart beat, drunkenness. Chronic Effects: heart/liver/kidney damage, dermatitis, birth defects, cancer (animal studies). Flammable.

Potential Health Effects

Target Organs: respiratory system, central nervous system (CNS), peripheral nervous system, cardiovascular system, liver, kidneys, skin

Primary Entry Routes: inhalation, skin contact, eye contact, ingestion (rarely)

Acute Effects

Inhalation: The vapor is mildly discomforting to the upper respiratory tract.

Inhalation hazard is increased at higher temperatures.

Anesthetics and narcotic effects (with dulling of senses and odor fatigue) are a consequence of exposure to chlorinated solvents.

Individual response varies widely; odor may not be considered objectionable at levels which quickly induce central nervous system effects.

High vapor concentrations may give a feeling of euphoria. This may result in reduced responses, followed by rapid onset of unconsciousness, possible respiratory arrest and death.

Acute effects from inhalation of high concentrations of vapor are pulmonary irritation, including coughing, with nausea; central nervous system depression - characterized by headache and dizziness, increased reaction time, fatigue and loss of coordination.

If exposure to highly concentrated solvent atmosphere is prolonged this may lead to narcosis, unconsciousness, even coma and possible death.

Evidence of acute human toxicity comes mainly from the use of TCE as an anesthetic, Tachypnea and ventricular arrhythmias are experienced at inhaled concentrations exceeding 15000 ppm. Systemic toxicity is low following anesthesia. Occasional hepatotoxicity (liver dysfunction) has been reported; this is probably due to the breakdown of TCE to dichloroacetylene and phosgene by soda-lime present in some anesthetic devices. The effects of TCE appear to be enhanced in some individuals by simultaneous exposure to caffeine, ethanol and other drugs. "Degreasers Flush" describes a reddening of facial, neck, and back skin and is seen after intake of substantial quantities of ethanol by certain individuals after exposures to TCE.

Eye: The liquid is highly discomforting to the eyes and is capable of causing pain and severe conjunctivitis.

Corneal injury may develop, with possible permanent impairment of vision, if not promptly and adequately treated.

The vapor is discomforting to the eyes.

The material may produce moderate eye irritation leading to inflammation.

Repeated or prolonged exposure to irritants may produce conjunctivitis.

Skin: The liquid is discomforting to the skin and may cause drying of the skin, which may lead to dermatitis.

Toxic effects may result from skin absorption.

Bare unprotected skin should not be exposed to this material. The material may accentuate any pre-existing skin condition.

The material may produce severe skin irritation after prolonged or repeated exposure, and may produce a contact dermatitis (nonallergic).

This form of dermatitis is often characterized by skin redness (erythema) and swelling (edema) which may progress to vesiculation, scaling and thickening of the epidermis.

Histologically there may be intercellular edema of the spongy layer (spongiosis) and intracellular edema of the epidermis.

Repeated exposures may produce severe ulceration.

Localized application may produce pustular eruptions, pruritus and erythema. A permeability coefficient of 1.6×10^{-2} cm/hr has been calculated by the US EPA. Percutaneous absorption is unlikely to contribute significantly to total body burdens unless dermatitis is present.

Ingestion: The liquid is highly discomforting and toxic if swallowed.

Ingestion may result in nausea, abdominal irritation, pain and vomiting.

Considered an unlikely route of entry in commercial/industrial environments.

Carcinogenicity: NTP - Not listed; IARC - Group 3, Not classifiable as to carcinogenicity to humans; OSHA - Not listed; NIOSH - Listed as carcinogen; ACGIH - Class A5, Not suspected as a human carcinogen; EPA - Not listed; MAK - Class B, Justifiably suspected of having carcinogenic potential.

Chronic Effects: Sensitive humans may experience anesthetic effects from short exposures.

Chronic effects of exposure include fatigue, headache, irritability, vomiting, skin flush and intolerance to alcohol.

Liver, kidney, heart and neurological damage may also result from chronic overexposure.

Alcohol intake may increase the toxic effects of the material.

A variety of disturbances have been seen among workers exposed at concentrations ranging from 1 to 335 ppm. These disturbances increased with the length of exposure (to 5 years or more) and where more prominent when exposures exceeded 40 ppm. Increased complaints of alcohol intolerance, tremors, giddiness and anxiety were amongst symptoms recorded. Variation in effects in different occupational settings may be due to different physical workloads.

There appeared to be no increase in the expected rates of congenital defects in children born to women exposed to TCE over a 13 year period.

Epidemiological studies consistently fail to show a link between cancers and TCE exposure. This is significant because of the tens of thousands of exposed workers monitored.

Section 4 - First Aid Measures

See DOT ERG

Inhalation: Remove to fresh air.
Lay patient down. Keep warm and rested.
If available, administer medical oxygen by trained personnel.
If breathing is shallow or has stopped, ensure clear airway and apply resuscitation. Transport to hospital or doctor, without delay.

Eye Contact: Immediately hold the eyes open and flush continuously for at least 15 minutes with fresh running water. Ensure irrigation under eyelids by occasionally lifting the upper and lower lids.
Transport to hospital or doctor without delay. Removal of contact lenses after an eye injury should only be undertaken by skilled personnel.

Skin Contact: Immediately remove all contaminated clothing, including footwear (after rinsing with water).
Wash affected areas thoroughly with water (and soap if available).
Seek medical attention in event of irritation.

Ingestion: Contact a Poison Control Center.
Do NOT induce vomiting. Give a glass of water.
Avoid giving milk or oils.
Avoid giving alcohol.

After first aid, get appropriate in-plant, paramedic, or community medical support.

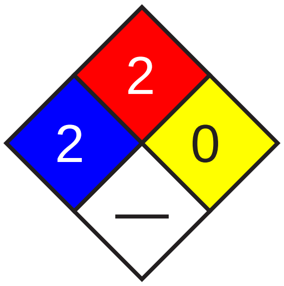
Note to Physicians: Treat symptomatically.
Do not administer sympathomimetic drugs as they may cause ventricular arrhythmias.
Following acute or short-term continued exposures to trichloroethylene:
1.Trichloroethylene concentration in expired air correlates with exposure. 8 hours exposure to 100 ppm produces levels of 25 ppm immediately and 1 ppm 16 hours after exposures.
2.Most mild exposure respond to removal from the source and supportive care.
Serious toxicity most often results from hypoxemia or cardiac dysrhythmias so that oxygen, intubation, intravenous lines and cardiac monitoring should be started initially as the clinical situation dictates.
3.Ipecac syrup should be give to alert patients who ingest more than a minor amount and present within 2 hours.
4.The efficacy of activated charcoal and cathartics is unclear.
5.The metabolites, trichloracetic acid, trichlorethanol and to a lesser degree, chloral hydrate, may be detected in the urine up to 16 days postexposure.
BIOLOGICAL EXPOSURE INDEX - BEI
These represent the determinants observed in specimens collected from a healthy worker exposed at the Exposure Standard (ES or TLV):

Determinant	Index	Sampling Time	Comments
Trichloroacetic acid in urine	10 mg/gm creatinine	End of work-week	NS
Trichloroacetic acid AND Trichloroethanol in urine	300 mg/mg creatinine	End of shift at end of work-week	NS
Free Trichloroethanol in blood	4 mg/L	End of shift at end of work-week	NS
Trichloroethylene in end-exhaled air			SQ
Trichloroethylene in blood			SQ

NS: Non-specific determinant; also seen after exposure to other materials
SQ: Semi-quantitative determinant - Interpretation may be ambiguous; should be used as a screening test or confirmatory test.

Section 5 - Fire-Fighting Measures

See DOT ERG



Fire Diamond

Flash Point: 32.222 °C Closed Cup
Autoignition Temperature: 420 °C
LEL: 8% v/v
UEL: 10.5% v/v
Extinguishing Media: Water spray or fog; foam, dry chemical powder, or BCF (where regulations permit).
Carbon dioxide.
General Fire Hazards/Hazardous Combustion Products: Vapor will burn when in contact with high temperature flame.
May form a flammable/explosive mixture in an oxygen enriched atmosphere. Heating may cause expansion/vaporization with violent rupture of containers. Decomposes on heating and produces corrosive fumes of hydrochloric acid, carbon monoxide and small amounts of toxic phosgene.
Fire Incompatibility: Avoid reaction with strong oxidizing agents (particularly oxygen in gas or liquid form and nitrogen dioxide), strong bases, sodium and sodium-potassium alloys. Powdered metals; magnesium, zinc and aluminum.
Contact with water may result in the slow formation of hydrochloric acid.
Attacks natural rubber.
Fire-Fighting Instructions: Contact fire department and tell them location and nature of hazard.
Wear breathing apparatus plus protective gloves. Prevent, by any means available, spillage from entering drains or waterways.
Use water delivered as a fine spray to control fire and cool adjacent area.
Avoid spraying water onto liquid pools.
Do not approach containers suspected to be hot.
Cool fire-exposed containers with water spray from a protected location.
If safe to do so, remove containers from path of fire.

Section 6 - Accidental Release Measures

See DOT ERG

Small Spills: Remove all ignition sources. Clean up all spills immediately.
Avoid breathing vapors and contact with skin and eyes.
Control personal contact by using protective equipment.
Contain and absorb spill with sand, earth, inert material or vermiculite.
Wipe up. Place in a suitable labeled container for waste disposal.
Large Spills: Clear area of personnel and move upwind.
Wear breathing apparatus plus protective gloves. Prevent, by any means available, spillage from entering drains or waterways.
Increase ventilation.
No smoking or bare lights within area.
Stop leak if safe to do so.
Contain and absorb spill with sand, earth, inert material or vermiculite.
Collect and seal in labeled drums for disposal.
If contamination of drains or waterways occurs, advise emergency services.
After clean-up operations, decontaminate and launder all protective clothing and equipment before storing and reusing.
Regulatory Requirements: Follow applicable OSHA regulations (29 CFR 1910.120).

Section 7 - Handling and Storage

Handling Precautions: Avoid all personal contact, including inhalation.
Wear protective clothing when risk of overexposure occurs.
Use in a well-ventilated area. Prevent concentration in hollows and sumps.
DO NOT enter confined spaces until atmosphere has been checked.
DO NOT allow material to contact humans, exposed food or food utensils.
Avoid smoking, bare lights or ignition sources. When handling, DO NOT eat, drink or smoke. Avoid contact with incompatible materials.
Keep containers securely sealed when not in used. Avoid physical damage to containers. Always wash hands with soap and water after handling. Working clothes should be laundered separately.
Launder contaminated clothing before reuse.
Observe manufacturer's storing/handling recommendations. Atmosphere should be regularly checked against established exposure standards to ensure safe working conditions are maintained.
Recommended Storage Methods: Inhibited grades may be stored in metal drums.
DO NOT use aluminum or galvanized containers. Check that containers are clearly labeled and free from leaks.

Packaging as recommended by manufacturer.

Regulatory Requirements: Follow applicable OSHA regulations.

Section 8 - Exposure Controls / Personal Protection

Engineering Controls: Local exhaust ventilation usually required.

If risk of overexposure exists, wear NIOSH-approved respirator.

Correct fit is essential to obtain adequate protection. NIOSH-approved self contained breathing apparatus (SCBA) may be required in some situations.

Provide adequate ventilation in warehouse or closed storage area.

Personal Protective Clothing/Equipment:

Eyes: Safety glasses with side shields; chemical goggles. Full face shield.

Contact lenses pose a special hazard; soft lenses may absorb irritants and all lenses concentrate them.

Hands/Feet: PVA gloves. Polyethylene gloves.

Viton gloves.

PVC boots.

Respiratory Protection:

Exposure Range >100 to <1000 ppm: Supplied Air, Constant Flow/Pressure Demand, Half Mask

Exposure Range 1000 to unlimited ppm: Self-contained Breathing Apparatus, Pressure Demand, Full Face

Note: odor threshold unknown

Other: Overalls. Eyewash unit. Barrier cream. Skin cleansing cream.

Glove Selection Index:

PE/EVAL/PE	Best selection
PVA	Best selection
TEFLON	Best selection
VITON	Satisfactory; may degrade after 4 hours continuous immersion
VITON/NEOPRENE	Poor to dangerous choice for other than short-term immersion
VITON/NITRILE	Poor to dangerous choice for other than short-term immersion
HYPALON	Poor to dangerous choice for other than short-term immersion
NEOPRENE	Poor to dangerous choice for other than short-term immersion
PVC	Poor to dangerous choice for other than short-term immersion
NITRILE	Poor to dangerous choice for other than short-term immersion

Section 9 - Physical and Chemical Properties

Appearance/General Info: Colorless liquid with a sweetish, chloroform-like odor, miscible with most organic solvents.

Physical State: Liquid
Odor Threshold: 10 mg/l
Vapor Pressure (kPa): 7.87 at 20 °C
Vapor Density (Air=1): 4.54
Formula Weight: 131.38
Specific Gravity (H_2O=1, at 4 °C): 1.47 at 15 °C

pH: Not applicable
pH (1% Solution): Not applicable.
Boiling Point: 87 °C (189 °F)
Freezing/Melting Point: -73 °C (-99.4 °F)
Volatile Component (% Vol): 100
Water Solubility: < 1 mg/mL at 21 °C

Section 10 - Stability and Reactivity

Stability/Polymerization/Conditions to Avoid: Decomposes in the presence of moisture to produce corrosive acid.

Product is considered stable under normal handling conditions. Hazardous polymerization will not occur.

Storage Incompatibilities: Avoid storage with strong oxidizers (particularly oxygen in gas or liquid form and nitrogen dioxide), strong bases, acetone, sodium/sodium-potassium alloys, magnesium, zinc and aluminum.

Avoid contact with water as the slow formation of hydrochloric acid results.

Attacks natural rubber.

Haloalkenes are highly reactive. Some of the more lightly substituted lower members are highly flammable; many members of the group are peroxidizable and polymerizable.

Section 11 - Toxicological Information

Toxicity

Oral (human) LD_{Lo}: 7000 mg/kg
Oral (man) TD_{Lo}: 2143 mg/kg
Oral (rat) LD_{50}: 5650 mg/kg
Inhalation (man) LC_{Lo}: 2900 ppm
Inhalation (human) TD_{Lo}: 812 mg/kg
Inhalation (human) TC_{Lo}: 6900 mg/m^3/10 m
Inhalation (man) TC_{Lo}: 2900 ppm
Inhalation (man) TC_{Lo}: 110 ppm/8h
Inhalation (man) TC_{Lo}: 160 ppm/83 m

Irritation

Skin (rabbit): 500 mg/24h - SEVERE
Eye (rabbit): 20 mg/24h - SEVERE

See *RTECS* KX 4550000, for additional data.

Section 12 - Ecological Information

Environmental Fate: No data found.

Ecotoxicity: LC_{50} Sheepshead minnow 20 mg/l/96 hr. /Conditions of bioassay not specified; LC_{50} Mexican axolotl (3-4 wk after hatching) 48 mg/l/48 hr /Conditions of bioassay not specified; LC_{50} Clawed toad (3-4 wk after hatching) 45 mg/l/48 hr /Conditions of bioassay not specified; LC_{50} Pimephales promelas (fathead minnow) 40.7 mg/l/96 hr (95% confidence limits 31.4-71.8 mg/l) /Flow-through test; EC_{10} Pimephales promelas (fathead minnow) 15.2 mg/l/24 hr; 16.9 mg/l/48 hr; 15.5 mg/l/72 hr; 13.7 mg/l/96 hr; Toxic effect for all concentrations specified: loss of equilibrium. /Flow-through bioassay; Toxicity Threshold (Cell Multiplication Inhibition Test) Scenedesmus quadricauda(green algae) >1000 mg/l /Time not specified, conditions of bioassay not specified; Toxicity Threshold (Cell Multiplication Inhibition Test) Pseudomonas putida (bacteria) 65 mg/l ; LC_{50} Grass shrimp 2 mg/l/96 hr. /Conditions of bioassay not specified

Henry's Law Constant: 1×10^{-2}

BCF: bluegill 17 to 39

Biochemical Oxygen Demand (BOD): 0%, 20 days

Octanol/Water Partition Coefficient: $\log K_{ow} = 2.29$

Soil Sorption Partition Coefficient: $K_{oc} = 2.0$

Section 13 - Disposal Considerations

Disposal: Recycle wherever possible. Consult manufacturer for recycling options.
Follow applicable federal, state, and local regulations.
Reclaim solvent at an approved site.
Evaporate or incinerate residue at an approved site.
Recycle containers if possible, or dispose of in an authorized landfill.

Section 14 - Transport Information

DOT Hazardous Materials Table Data (49 CFR 172.101):

Shipping Name and Description: Trichloroethylene
ID: UN1710
Hazard Class: 6.1 - Poisonous materials
Packing Group: III - Minor Danger
Symbols:
Label Codes: 6.1 - Poison *or* Poison Inhalation Hazard *if inhalation hazard, Zone A or B*
Special Provisions: IB3, N36, T4, TP1
Packaging: **Exceptions:** 153 **Non-bulk:** 203 **Bulk:** 241
Quantity Limitations: **Passenger aircraft/rail:** 60 L **Cargo aircraft only:** 220 L
Vessel Stowage: **Location:** A **Other:** 40

Section 15 - Regulatory Information

EPA Regulations:
RCRA 40 CFR: Listed U228 Toxic Waste
CERCLA 40 CFR 302.4: Listed per CWA Section 311(b)(4), per RCRA Section 3001, per CWA Section 307(a) 100 lb (45.35 kg)

SARA 40 CFR 372.65: Listed
SARA EHS 40 CFR 355: Not listed
TSCA: Listed

Section 16 - Other Information

Disclaimer: Judgments as to the suitability of information herein for the purchaser's purposes are necessarily the purchaser's responsibility. Although reasonable care has been taken in the preparation of such information, Genium Group, Inc. extends no warranties, makes no representations, and assumes no responsibility as to the accuracy or suitability of such information for application to the purchaser's intended purpose or for consequences of its use.

Section 1 - Chemical Product and Company Identification

Material Name: Vinyl Chloride **CAS Number:** 75-01-4
Chemical Formula: C_2H_3Cl
Structural Chemical Formula: $CH_2{=}CHCl$
EINECS Number: 200-831-0
ACX Number: X1003494-5
Synonyms: CHLORETHENE; CHLORETHYLENE; CHLOROETHENE; CHLOROETHYLENE; CHLORURE DE VINYLE; CLORURO DI VINILE; ETHENE,CHLORO-; ETHYLENE MONOCHLORIDE; ETHYLENE,CHLORO-; MONOCHLOROETHENE; MONOCHLOROETHYLENE; MONOCHOROETHENE; MONOVINYL CHLORIDE (MVC); TROVIDUR; VC; VCM; VINILE (CLORURO DI); VINYL C MONOMER; VINYL CHLORIDE; VINYL CHLORIDE MONOMER; VINYL CHLORIDE MONOMER (VCM); VINYL CHLORIDE,INHIBITED; VINYLCHLORID; VINYLE(CHLORURE DE); WINYLU CHLOREK
General Use: Used in the plastics industry; as a refrigerant; in organic syntheses

Section 2 - Composition / Information on Ingredients

Name	CAS	%
vinyl chloride	75-01-4	>98

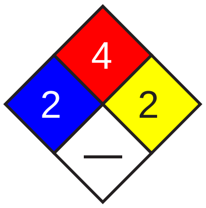
OSHA PEL
TWA: 1 ppm; Ceiling: 5 ppm, 15-minute; 1910.1017.

NIOSH REL

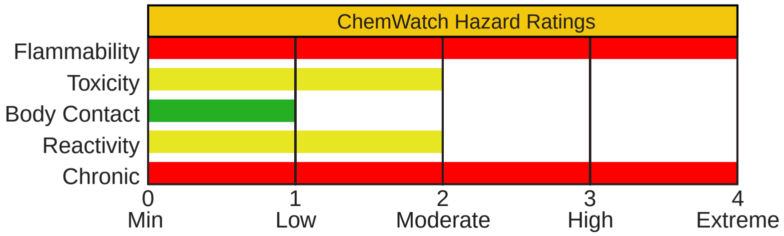
ACGIH TLV
TWA: 1 ppm.

EU OEL
TWA: 7.77 mg/m^3.

Section 3 - Hazards Identification



Fire Diamond



HMIS
3 Health
4 Flammability
2 Reactivity

ANSI Signal Word

Danger!

Flammable Compressed Gas

☆☆☆☆☆ Emergency Overview ☆☆☆☆☆

Colorless gas; pleasant ethereal odor. Compressed gas can cause frostbite. Toxic. Other Acute Effects: CNS depression. Chronic Effects: reproductive effects, skin/blood changes, arthralgias, bone effects (hand), vascular disorder (fingers/toes). Cancer hazard. Flammable

Potential Health Effects

Target Organs: liver, central nervous system (CNS), respiratory system, lymphatic system, bone, connective tissue of the skin
Primary Entry Routes: inhalation, skin contact, eye contact
Acute Effects
Inhalation: The gas is highly discomforting and may be fatal if inhaled.
Acute intoxication by halogenated aliphatic hydrocarbons appears to take place over two stages. Signs of a reversible narcosis are evident in the first stage and in the second stage signs of injury to organs may become evident. A single organ alone is (almost) never involved.

Depression of the central nervous system is the most outstanding effect of most halogenated aliphatic hydrocarbons. Inebriation and excitation, passing into narcosis, is a typical reaction. In severe acute exposures there is always a danger of death from respiratory failure or cardiac arrest due to a tendency to make the heart more susceptible to catecholamines (adrenalin).

A single 5 minute inhalation exposure of 8000-25000 ppm caused nausea, headache and dizziness among volunteers. After cessation of exposure only 3-5% of the parent compound was exhaled unchanged. Metabolism by microsomal cytochrome P-450 results in the production of chloroethylene oxide and 2-chloroacetaldehyde and subsequent urinary elimination as thiodiglycolic acid. Half-life is 4-5 hours.

Vinyl chloride and related vinyl monomers possess narcotic action and produce depending upon concentration, characteristic neurological effects, a state of euphoria, followed by a state of inebriation, similar to ethanol intoxication.

Exposure of mice, rats and guinea pigs at 100,000-300,000 ppm caused concentration-dependent mortality. Pulmonary edema, inflammation, hyperemia, congestion and engorgement were recorded - liver and kidney involvement was surprisingly low. Deaths were due to central arrest in narcosis.

Eye: The vapor is discomforting to the eyes and is capable of causing a mild, temporary redness of the conjunctiva (similar to wind-burn), temporary impairment of vision and/or other transient eye damage/ulceration.

Skin: The vapor is mildly discomforting to the skin.

Toxic effects may result from skin absorption.

Vinyl chloride acts upon the skin and produces a sensation of heat.

Vaporizing liquid causes rapid cooling and contact may cause cold burns, frostbite.

Ingestion: Not normally a hazard due to physical form of product.

Carcinogenicity: NTP - Class 1, Known to be a carcinogen; IARC - Group 1, Carcinogenic to humans; OSHA - Listed as a carcinogen; NIOSH - Listed as carcinogen; ACGIH - Class A3, Animal carcinogen; EPA - Listed; MAK - Class A1, Capable of inducing malignant tumors as shown by experience with humans.

Chronic Effects: Repeated exposure of laboratory animals to vinyl chloride produced little liver or kidney damage. Repeated exposures produce neurological effects in man with somnolence prominent. Dyspeptic disturbances include epigastric pain, swelling, discomfort, heaviness in the right hypochondrium and anorexia. Congestive hepatomegaly may mimic toxic hepatitis without jaundice. Some case become chronic. Allergic dermatitis and schleroderma and Raynaud's syndrome have been observed. Repeated exposure of workers has caused increased liver enzyme concentrations, restricted blood flow, bone degeneration in the fingers, liver and spleen enlargement, nervous system disturbance, CNS depression, decreased respiratory function and emphysema.

A dose-dependent relationship between exposure and the incidence of several tumor types has been established. Exposures to high concentrations have little additional effect because the action of metabolites is responsible for the carcinogenicity rather than the action of the parent molecule. Formation rates of the metabolites are limited and dose-dependent and once the enzyme systems responsible for vinyl chloride activation are saturated, greater doses do not produce a corresponding increase in tumor incidence. Reports of hepatic angiosarcoma and respiratory cancers in vinyl chloride workers have appeared over many years. Cancers of the respiratory system (primarily angiosarcoma), brain as well as lymphomas occur more often than might be expected among men occupationally exposed to vinyl chloride for at least one year.

Section 4 - First Aid Measures

See DOT ERG

Inhalation: Remove to fresh air.

Lay patient down. Keep warm and rested.

If breathing is shallow or has stopped, ensure clear airway and apply resuscitation. Transport to hospital or doctor.

Eye Contact: Immediately hold the eyes open and flush continuously for at least 15 minutes with fresh running water. Ensure irrigation under eyelids by occasionally lifting the upper and lower lids. Transport to hospital or doctor without delay. Removal of contact lenses after an eye injury should only be undertaken by skilled personnel.

Skin Contact: Immediately flush body and clothes with large amounts of water, using safety shower if available.

Quickly remove all contaminated clothing, including footwear.

Wash affected areas with water (and soap if available) for at least 15 minutes. Transport to hospital or doctor.

In case of cold burns (frostbite): Bathe the affected area immediately in cold water for 10 to 15 minutes, immersing if possible and without rubbing.

Do not apply hot water or radiant heat. Apply a clean, dry dressing.

Transport to hospital or doctor.

Ingestion: Not normally a hazard due to physical form of product. DO NOT delay. Immediately transport to hospital or doctor.

After first aid, get appropriate in-plant, paramedic, or community medical support.

Note to Physicians: Treat symptomatically. Do not give adrenalin (epinephrine) or related drugs.

Section 5 - Fire-Fighting Measures

Flash Point: -78 °C Open Cup

Autoignition Temperature: 472 °C

LEL: 3.6% v/v

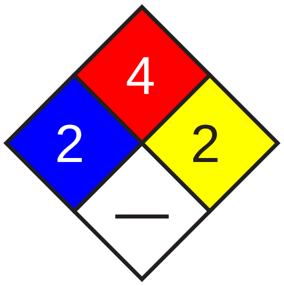
UEL: 33% v/v

Extinguishing Media: Dry chemical powder. DANGER: Deliver media remotely.

For minor fires: Flooding quantities only.

For large fires: Do not attempt to extinguish.

See DOT ERG



Fire Diamond

General Fire Hazards/Hazardous Combustion Products: WARNING: Long standing in contact with air and light may result in the formation of potentially explosive peroxides.

Liquid and vapor are highly flammable.

Dangerous hazard when exposed to heat or flame.

Severe vapor explosion hazard, when exposed to flame or spark.

Heating may cause expansion or decomposition leading to violent rupture of containers.

Decomposition may produce toxic fumes of hydrogen chloride.

Fire Incompatibility: Avoid reaction with copper, aluminum, oxidizing agents and certain catalytic impurities.

Explosion hazard may follow contact with incompatible materials. Avoid contamination with oxidizing agents i.e. nitrates, oxidizing acids, chlorine bleaches, pool chlorine etc. as ignition may result.

Fire-Fighting Instructions: Contact fire department and tell them location and nature of hazard.

Fight fire from a safe distance, with adequate cover.

May be violently or explosively reactive. Wear full body protective clothing with breathing apparatus. Consider evacuation.

If safe to do so, switch off electrical equipment until vapor fire hazard is removed.

If safe to do so, stop flow of gas.

Do not approach cylinders suspected to be hot.

Cool fire-exposed containers with water spray from a protected location.

Use water delivered as a fine spray to control the fire and cool adjacent area.

Given its high vapor density spilled vinyl chloride is slow to disperse and will collect in low lying areas.

Section 6 - Accidental Release Measures

See DOT ERG

Small Spills: Erect warning notices and seal off area.

1. Avoid breathing vapor and any contact with liquid or gas. Protective equipment including respirator should be used.
2. Do NOT enter confined spaces were gas may have accumulated.
3. Shut of all sources of possible ignition and increase ventilation.
4. Clear area of personnel.
5. Stop leak only if safe to so do.
6. Remove leaking cylinders to safe place. Release pressure under safe controlled conditions by opening valve.
7. Keep area clear of personnel until gas has dispersed.

Large Spills: Supply maximum air ventilation (explosion proof equipment) to keep concentration well below lower flammability limit.

1. Clear area of all unprotected personnel and move upwind.
2. Contact fire department and advise them of the location and nature of hazard.
3. May be violently or explosively reactive.
4. Wear full body clothing with breathing apparatus.
5. Consider evacuation.
6. Shut off all possible sources of ignition and increase ventilation.
7. No smoking or bare lights within area.
8. Use extreme caution to prevent violent reaction.
9. Stop leak only if safe to so do.
10. Water spray or fog may be used to disperse vapor.
11. Do NOT enter confined space where gas may have collected.
12. Keep area clear until gas has dispersed.

When leaking containers have been removed or leak has been stopped, hose spill area down with copious quantities of water. Allow any liquid to evaporate prior to wash down.

Regulatory Requirements: Follow applicable OSHA regulations (29 CFR 1910.120).

Section 7 - Handling and Storage

Handling Precautions: Used in closed pressurized systems, fitted with safety relief valve.

Vented gas is flammable, denser than air and will spread. Vent path must not contain ignition sources, pilot lights, bare flames.

Atmospheres must be tested and O.K. before work resumes after leakage.
Obtain a work permit before attempting any repairs.
Do not attempt repair work on lines, vessels under pressure.
Handle and open container with care.
Avoid all personal contact, including inhalation.
Wear protective clothing when risk of exposure occurs.
Use in a well-ventilated area. Prevent concentration in hollows and sumps.
DO NOT enter confined spaces until atmosphere has been checked.
Avoid smoking, bare lights, heat or ignition sources.
When handling, DO NOT eat, drink or smoke.
Vapor may ignite on pumping or pouring due to static electricity.
DO NOT use plastic buckets. Ground and secure metal containers when dispensing or pouring product. Use spark-free tools when handling.
Avoid contact with incompatible materials.
Keep containers securely sealed. Avoid physical damage to containers.
Always wash hands with soap and water after handling.
Work clothes should be laundered separately.
Use good occupational work practices. Observe manufacturer's storing and handling recommendations. Atmosphere should be regularly checked against established exposure standards to ensure safe working conditions.
DO NOT transfer gas from one cylinder to another.

Recommended Storage Methods: Check that containers are clearly labeled.
Cylinder fitted with valve protector cap.
Ensure the use of equipment rated for cylinder pressure.
Ensure the use of compatible materials of construction.
Cylinder valve must be closed when not in use or when empty.
Cylinder must be properly secured either in use or in storage.
WARNING: Suckback into cylinder may result in rupture.
Use back-flow preventive device in piping.
Aerosol pack.
Vacuum insulated container.

Regulatory Requirements: Follow applicable OSHA regulations.

Section 8 - Exposure Controls / Personal Protection

Engineering Controls: Fans and electrical equipment must be explosion-proof to meet TLV requirements. Approved respirators must be available for non-routine and emergency situations.
Areas where gas cylinders are stored/used require discrete, controlled exhaust ventilation.
Local exhaust ventilation usually required.
If risk of overexposure exists, wear NIOSH-approved respirator.
Correct fit is essential to obtain adequate protection. NIOSH-approved self contained breathing apparatus (SCBA) may be required in some situations.
Provide adequate ventilation in warehouse or closed storage area.

Personal Protective Clothing/Equipment:

Eyes: Close fitting gas tight goggles and DO NOT wear contact lenses.
Contact lenses pose a special hazard; soft lenses may absorb irritants and all lenses concentrate them.

Hands/Feet: Neoprene rubber gloves.

Respiratory Protection:
Exposure Range >1 to 50 ppm: Supplied Air, Constant Flow/Pressure Demand, Half Mask
Exposure Range >50 to 1000 ppm: Supplied Air, Constant Flow/Pressure Demand, Full Face
Exposure Range >1000 to unlimited ppm: Self-contained Breathing Apparatus, Pressure Demand, Full Face
Note: poor warning properties

Other: Protective overalls, closely fitted at neck and wrist. Eye-wash unit.
IN CONFINED SPACES:
1. Non-sparking protective boots
2. Static-free clothing.

Glove Selection Index:

VITON	Best selection
NITRILE	Satisfactory; may degrade after 4 hours continuous immersion

Section 9 - Physical and Chemical Properties

Appearance/General Info: A colorless poisonous gas at ambient temperature, with a mild sweet odor in high concentrations. It liquefies readily under increased pressure or at reduced temperatures. Soluble in alcohol, ether, carbon tetrachloride and benzene.

Physical State: Liquefied gas	**Vapor Pressure (kPa):** 343.5 at 20 °C

Vapor Density (Air=1): 2.2
Formula Weight: 62.5
Specific Gravity (H_2O=1, at 4 °C): 0.912 at 20 °C
Evaporation Rate: Not applicable
pH: Not applicable
pH (1% Solution): Not applicable.
Boiling Point: -13.37 °C (8 °F)
Freezing/Melting Point: -153.8 °C (-244.84 °F)
Volatile Component (% Vol): 100
Water Solubility: Slightly soluble

Section 10 - Stability and Reactivity

Stability/Polymerization/Conditions to Avoid: Presence of heat source and direct sunlight (ultra-violet radiation).
Presence of elevated temperatures.
Presence of an ignition source.
Storage in unsealed containers.
Stable under normal storage conditions. Polymerization may occur at elevated temperatures and in the presence of ignition sources.

Storage Incompatibilities: Avoid contamination with oxidizing agents i.e. nitrates, oxidizing acids, chlorine bleaches, pool chlorine etc. as ignition may result.
Avoid peroxides, copper and copper alloys and plastics.
Haloalkenes are highly reactive. Some of the more lightly substituted lower members are highly flammable; many members of the group are peroxidizable and polymerizable.
If peroxidation occurs, vinyl chloride tends to self-polymerize violently and this has resulted in several industrial accidents.
Accidental exposure of the recovered monomer to atmospheric oxygen for a long period resulted in the formation of an unstable polyperoxide which initiated explosion. A 20-30% aqueous solution has been used to destroy the peroxide.
An explosion in a valve in a liquid monomer line appears to have been caused by traces of nitrogen oxides remaining after passivation of the line by nitric acid.

Section 11 - Toxicological Information

Toxicity
Oral (rat) LD_{50}: 500mg/kg
Oral (rat) TD_{Lo}: 3463mg/kg/52w
Inhalation (rat) TC_{Lo}: 1ppm/4h/52w
Inhalation (man) TC_{Lo}: 200ppm/14y
Tumors of the sense organs, vascular system, respiratory system, gastrointestinal system, skin and liver, lymphoma, paternal effects, effects on fertility, fetotoxicity, specific developmental abnormalities involving the musculoskeletal system recorded.

Irritation
Nil reported

See *RTECS* YZ 3200000, for additional data.

Section 12 - Ecological Information

Environmental Fate: If released to soil, it will be subject to rapid volatilization with reported half-lives of 0.2 and 0.5 days for evaporation from soil at 1 and 10 cm incorporation, respectively. Any which does not evaporate will be expected to be highly to very highly mobile in soil and it may leach to the groundwater. It may be subject to biodegradation under anaerobic conditions such as exists in flooded soil and groundwater. If released to water, it will not be expected to hydrolyze, to bioconcentrate in aquatic organisms or to adsorb to sediments. It will be subject to rapid volatilization with an estimated half-life of 0.805 hr for evaporation from a river 1 m deep with a current of 3 m/sec and a wind velocity of 3 m/sec. In waters containing photosensitizers such as humic acid, photodegradation will occur fairly rapidly. Limited existing data indicate that it is resistant to biodegradation in aerobic systems and therefore, it may not be subject to biodegradation in aerobic soils and natural waters. It will not be expected to hydrolyze in soils or natural waters under normal environmental conditions. If released to the atmosphere, it can be expected to exist mainly in the vapor-phase in the ambient atmosphere and to degrade rapidly in air by gas-phase reaction with photochemically produced hydroxyl radicals with an estimated half-life of 1.5 days.
Ecotoxicity: No data found.
Henry's Law Constant: 0.0560
BCF: estimated at 7
Biochemical Oxygen Demand (BOD): none
Octanol/Water Partition Coefficient: log K_{ow} = calculated at 0.6
Soil Sorption Partition Coefficient: K_{oc} = estimated at 56

Section 13 - Disposal Considerations

Disposal: The gas should be burned in a high temperature furnace equipped with an afterburner and scrubber to remove HCl formed.
Follow applicable federal, state, and local regulations.
Return all damaged and empty cylinders and containers to the supplier.

Section 14 - Transport Information

DOT Hazardous Materials Table Data (49 CFR 172.101):

Shipping Name and Description: Vinyl chloride, stabilized
ID: UN1086
Hazard Class: 2.1 - Flammable gas
Packing Group:
Symbols:
Label Codes: 2.1 - Flammable Gas
Special Provisions: 21, B44, T50
Packaging: **Exceptions:** 306 **Non-bulk:** 304 **Bulk:** 314, 315
Quantity Limitations: **Passenger aircraft/rail:** Forbidden **Cargo aircraft only:** 150 kg
Vessel Stowage: **Location:** B **Other:** 40

Section 15 - Regulatory Information

EPA Regulations:
RCRA 40 CFR: Listed U043 Toxic Waste
CERCLA 40 CFR 302.4: Listed per RCRA Section 3001, per CWA Section 307(a), per CAA Section 112 1 lb (0.454 kg)
SARA 40 CFR 372.65: Listed
SARA EHS 40 CFR 355: Not listed
TSCA: Listed

Section 16 - Other Information

Disclaimer: Judgments as to the suitability of information herein for the purchaser’s purposes are necessarily the purchaser’s responsibility. Although reasonable care has been taken in the preparation of such information, Genium Group, Inc. extends no warranties, makes no representations, and assumes no responsibility as to the accuracy or suitability of such information for application to the purchaser’s intended purpose or for consequences of its use.

ARCADIS

Appendix F

Emergency Action Plan and Guidance

EMERGENCY ACTION PLAN

Emergency Contact List

Emergency Contact	Phone
Local Police – Oceanside Police Department	911 (if appropriate) and 516.431.1800
Local Ambulance –	911 (if appropriate)
Local Fire Department – Oceanside Fire Department	911 (if appropriate) and 516.766.0245
Local Hospital – South Nassau Community Hospital	516.632.3000
Local Weather Data – online	www.weather.com
Poison Control	800.332.3073
National Response Center (all spills in reportable quantities)	800.424.8802
U.S. Coast Guard (spills to water)	800.424.8802
ARCADIS Project Manager – William McCune	315.671.9172
Client Contact – Peter Kasbohm	720.984.3145
H&S Manager – Charles Webster	315.671.9297
Chevron Project Manager – Peter Kasbohm	770-984-3145
Chevron Alternate Contact 1 – NA	
Chevron Alternate Contact 2 – NA	

Emergency Notification Procedure for the project:

Step 1:Dial 911 (if necessary) and/or WorkCare 800-455-6155
Step 2:Contact PIC/PM/TM
Step 3:Contact H&S Manager
Step 4:Contact Near Loss Reporting Hotline 866-242-4304

If emergency attention is not needed but professional medical attention is necessary, the employee will be taken to (see hospital route):

Medical Facility: South Nassau Community Hospital

Address: 440 Merrick Road
Oceanside, NY 11572-1404

Phone Number: 516.632.3000

Emergency Supplies and Equipment List

Emergency Supplies and Equipment (check all that apply)	Location on Project Site
☒ First Aid Kit (type):	Field vehicle/Field Kit
☒ Fire Extinguisher	Field vehicle/Field Kit
☒ Mobile Phone ☐ Satellite Phone	Field vehicle/Field Kit
☒ Traffic Cones	Field vehicle/Field Kit
☐ Walkie Talkies	
☒ Water or Other Fluid Replenishment	Field vehicle/Field Kit
☐ Eye Wash/Quick Drench Station	
☒ Eye Wash Bottle	Field vehicle/Field Kit
☒ Wash and Dry Towelettes	Field vehicle/Field Kit
☒ Sunscreen (SPF 15 or higher)	Field vehicle/Field Kit
☐ Insect Repellant	
☐ Chemical Spill Kit	
☐ *Other (specify):*	

First Aid Kit Contents
(Minimum)

Item	Quantity
Absorbent Compress, 32 sq. in. (No side smaller than 4")	1
Adhesive Bandages, 1” x 3”	16
Adhesive Tape	5 yards
Antiseptic - .5 g application	10
Burn treatment - .5 g application	6
Medical Exam Gloves	2 pair
Sterile pads, 3” x 4”	4
Triangular bandage, 40” x 40” x 56”	1

Note: In addition to the above minimum contents, a kit should have optional items added, based upon specific workplace hazards. The selection of additional supplies should be made by consulting with a health care professional or a person competent in first aid who is knowledgeable of the hazards found in that specific workplace. The optional items shall meet specifications stated in Section 5.3 of ANSI Z308.1-1998.

Emergency Response Guidelines

Emergency	Action	Notification	Comments
Fire	If incipient, attempt to extinguish using portable fire extinguisher. If free burning or exceeding portable extinguisher capability, quickly gather at off-site evacuation location identified on Evacuation Map.	1. Call 911 2. Notify ARCADIS PM (if unavailable notify Alternate) 3. Notify Chevron PM (if unavailable notify Alternate)	
Severe Weather	Shelter in location identified on Shelter Location Map.	1. Notify ARCADIS PM.	If unable to get to shelter location, take cover in vehicle.
Earthquake	If outdoors, go to an open field or parking lot with no overhead hazards. If indoors, quickly get under a table or other large object for protection from falling debris.	1. Notify ARCADIS PM (if unavailable notify Alternate) 2. Notify Chevron PM (if unavailable notify Alternate).	Be prepared for aftershocks. When possible, evacuation building and go to open field until notification can be made to ARCADIS and Client Personnel.
Material Release	If possible to do so safely, attempt to stop further release with dikes, plugs, etc. LPSA to evaluate need for air monitoring. Evacuate depending upon material released and quantity.	1. Call 911, depending upon material released and quantity. 2. Notify ARCADIS PM (if unavailable, notify Alternate). 3. Notify Chevron PM (if unavailable, notify Alternate)	
Medical Emergency	Administer first aid, as necessary. If injury occurred to self, notify terminal management immediately.	1. Call 911, depending upon extent of injury/illness. 2. Notify ARCADIS PM (if unavailable, notify Alternate). 3. Notify Chevron PM (if unavailable, notify Alternate).	
Off-Site Emergency	Potential off-site hazards should be identified on the Risk Assessment Matrix and included in this section. These may include adjoining chemical plants, railroad yard, bulk terminal facilities.	1. Call 911, depending upon emergency. 2. Assemble work crew to determine course of action. Use Shelter Location or Evacuation Route identified in Appendix H. 3. Notify ARCADIS PM (if unavailable, notify Alternate).	
Chemical Exposure	Move victim to safe location. Personnel WEARING LIKE PPE or a higher level of PPE than the victim may be required to decon and assist victim with PPE removal.	1. Call 911, depending upon emergency OR, if emergency action is not needed, call WorkCare. 2. Contact terminal management by radio. – Delete this if not a terminal site. 3. Notify ARCADIS PM (if unavailable, notify Alternate). 4. Notify Chevron PM (if unavailable, notify Alternate).	

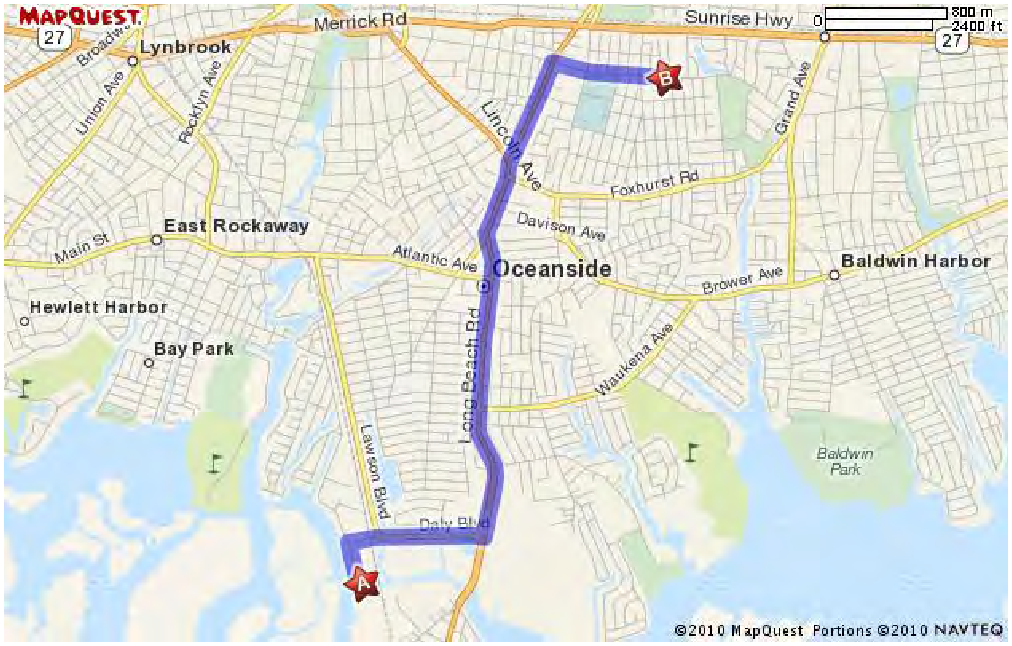
Directions to Hospital

Start: 1 Industrial Pl, Oceanside, NY, 11572-4809

1.	Start out going NORTH on HAMPTON RD toward DALY BLVD.	0.2 mi
2.	Turn RIGHT onto DALY BLVD.	0.6 mi
3.	Turn LEFT onto LONG BEACH RD.	2.4 mi
4.	Turn RIGHT onto MERRICK RD.	0.5 mi

End: 440 Merrick Road, Oceanside, NY

Total Travel Estimate 3.82 miles - about 11 minutes



©2010 MapQuest Portions ©2010 NAVTEQ

Appendix G

Chevron-Specific Requirements

G1. Introduction 1

G.1.1. Policy Statement 1

G.1.2. Loss Prevention System 1

G1.2.1. Loss Prevention Self Assessment 1

G.1.2.2 Loss Prevention System Reporting 2

G.1.2.2.1 Incident Investigation 2

G.1.2.2.2 Loss Prevention Observation 2

G.1.2.2.3. Job Loss Analysis 3

G1.3 Reporting and Investigation Requirements 3

G1.4 Motor Vehicle Safety Process 9

G.1.4.1. MVS Variance Process 10

G2. Roles and Responsibility 10

G2.1 All Personnel 10

G.2.1.1. Use of Loss Prevention System 10

G2.1.2. Stop Work Authority 10

G2.1.3. Tenets of Operational Excellence 11

G2.2. Special Emphasis Programs 12

G2.2.1 Short Service Employee Program. 12

G2.2.2. Hand Safety 12

G2.2.2.1.. Fixed Open-Blade Knives 13

G2.2.3. Excavation Safety and Borehole Clearance 13

G.2.2.4. Traffic Control 13

G.2.2.5. Drug, Alcohol, and Search Policy 13

G2.3. Verification and Validation 14

G2.4. One Team Incident Prevention System (OTIPS) 14

G.2.4.1. Chevron Compliance Reporting Process and Hotline 14

G2.4.2. Near-Loss Reporting Hotline 15

G2.4.3. Short-Service Employee Program 15

G2.5. All Onsite Personnel 16

G2.5.1. Use of Loss Prevention System 16

G2.6. Photographs 17

G2.7. Visitors 17

G2.8. Operational Control 17

G3. General Safety Practices 18

G3.1. General Safety Rules 18

G3.2. Drug, Alcohol and Search Policy 18

G3.3. Contact Lenses 19

G3.4. Use of GFCI Line Cord 19

G3.5. Spill Control 19

G3.6. Lock Out Tag Out 20

G3.7. Traffic Control 20

G3.6.1. Requirement for Two-Person Crew 21

G3.6.2 Level 1 21

G3.6.3. Level 2 22

G3.6.4. Level 3 22

G3.6.5. Level 4 22

G3.7 Waste Management 22

G3.8. Hand Safety Program 23

G3.9. Fixed Open Blade Knife (FOBK) Policy 24

G3.10. Jewelry, Hair and Loose Clothing 24

G3.11. Site-Specific Safety Rules 24

G4. Training 25

G4.1. General 25

G4.2. Permit-to-Work Approvers 25

G4.3. Management and Supervisors 26

G4.4. First Aid and CPR 26

G4.5. Site-Specific Training 26

Appendix G-Chevron-Specific Requirements

G1. Introduction

G.1.1. Policy Statement

This section of the E-HASP describes Chevron-specific policies, procedures and/or recommendations that must be followed in addition to requirements detailed in the site E-HASP. When ARCADIS and Chevron policies or procedures overlap, personnel are expected to adhere to the most stringent version. It is not the intent of this E-HASP to provide specific recommendations when policies conflict or overlap; therefore, personnel are expected to be familiar with policy variations in order to make informed decisions regarding compliance with company and client policies and procedures.

G.1.2. Loss Prevention System

Personnel will not be allowed to perform work on Chevron property or sites prior to attending an LPS training session. Contractors performing low-risk work and of short duration (totally less than 40 hours in any 12-month rolling period) may watch a LPS video instead of the full LPS training session. Proof of this training must be maintained by the HSS. This policy is at the discretion of the Chevron PM. ARCADIS personnel are required to keep a copy of Loss Prevention System™ Handbook with their field safety equipment.

G1.2.1. Loss Prevention Self Assessment

All onsite personnel are required to perform an LPSA prior to beginning any activity. This process is synonymous with ARCADIS' TRACK. This three-step process requires each individual to:

- *Assess* the risk of the task to be performed. Ask the following questions:
  - What could go wrong?
  - What is the worst thing that could happen if something does go wrong?
- *Analyze* the ways the risk can be reduced. Ask the following questions:
  - Do I have all the necessary training and knowledge to do this task safely?
  - Do I have all the proper tools and PPE?
- *Act* to control the risk and perform the task safely.
  - Take the necessary action to perform the job safely.
  - Follow written procedures, and ask for assistance if necessary.

This process must be performed prior to beginning any activity and must be performed after any near miss or other incident in order to determine if it is safe to proceed.

G.1.2.2 Loss Prevention System Reporting

Optimization of LPS depends upon timely reporting both internally and externally of all types of LPS tools (near misses, observations and incidents). Detailed descriptions of LPS tools are discussed in detail in the sections below.

G.1.2.2.1 Incident Investigation

An incident is any of the following events: first-aid cases, injuries, illnesses, near misses, spills/leaks, equipment and property damage, motor vehicle accidents, regulatory violations, fires and business interruptions. All incidents shall be reported to the PM, the PO and the HSO immediately and investigated within 24 hours. In the event that one of those individuals cannot be contacted, a voice message should be left and the next individual in the chain should be contacted. Follow-up with the absent individual should occur as soon as possible.

The purpose of an incident investigation is to prevent the recurrence of a similar hazardous event. Using the information gathered during an investigation, appropriate measures will be taken to protect personnel from the hazard in question. The CEMC Incident Reporting and Notification Guide is included in Section G.1.3 (the most updated version will be at https://emconeteam.chevron.com/oe/process_IRIP.aspx). Incident Investigation Forms are included in Appendix E.

The flowchart on page G-4 outlines steps to be taken in the event of an incident or near miss.

G.1.2.2.2 Loss Prevention Observation

The HSS or designee will perform the LPO. The purpose of the LPO is to identify and correct potential hazards and to positively reinforce behaviors and practices that are correct. The HSS must identify potential deviations from safe work practices that could possibly result in an incident and take prompt corrective action. A minimum of one observation will be performed per 400 hours for a specific task and one per 200 hours for higher risk tasks. The LPO process steps are:

- Identify tasks that have the greatest potential for hazardous incidents.
- Review the standard procedure for completing the task.
- Discuss with the observed employee the task and the HSS role in observing the task.
- Observe the employee completing the task.

- Reference the LPO form for criteria. Complete the form, documenting positive, as well as areas in need of improvement.

- Discuss the results of the LPO with the employee. Discuss corrective action necessary.

- Implement corrective action.

- Communicate the results of the LPO and corrective action to the PM and the HSO.

- Verify and validate corrective actions are implemented.

G.1.2.2.3. Job Loss Analysis

A JLA is a tool used to identify potential hazards and develop corrective or protective systems to eliminate the hazard. A JLA lists all the potential hazards associated with a task or activity and can include site-specific concerns associated with the task or activity. Hazards may be physical, such as lifting hazards or eye hazards or environmental, such as weather or biological (e.g., stinging insects, snakes). Following the identification of the hazards associated with an activity, control measures are evaluated and protective measures or procedures are then instituted. JLAs are reviewed daily to confirm that the procedures and protective equipment specified for each task or activity are current and technically correct. Any changes in site conditions and/or the scope of work will require a review and modification to the JLA in question. During this review process, comments on the JLA and its procedures should be obtained from personnel associated with the activity being analyzed. At a minimum a JLA will outline the safety requirements for a corresponding specific task as detailed in Section 4.1 of this E-HASP.

G1.3 Reporting and Investigation Requirements

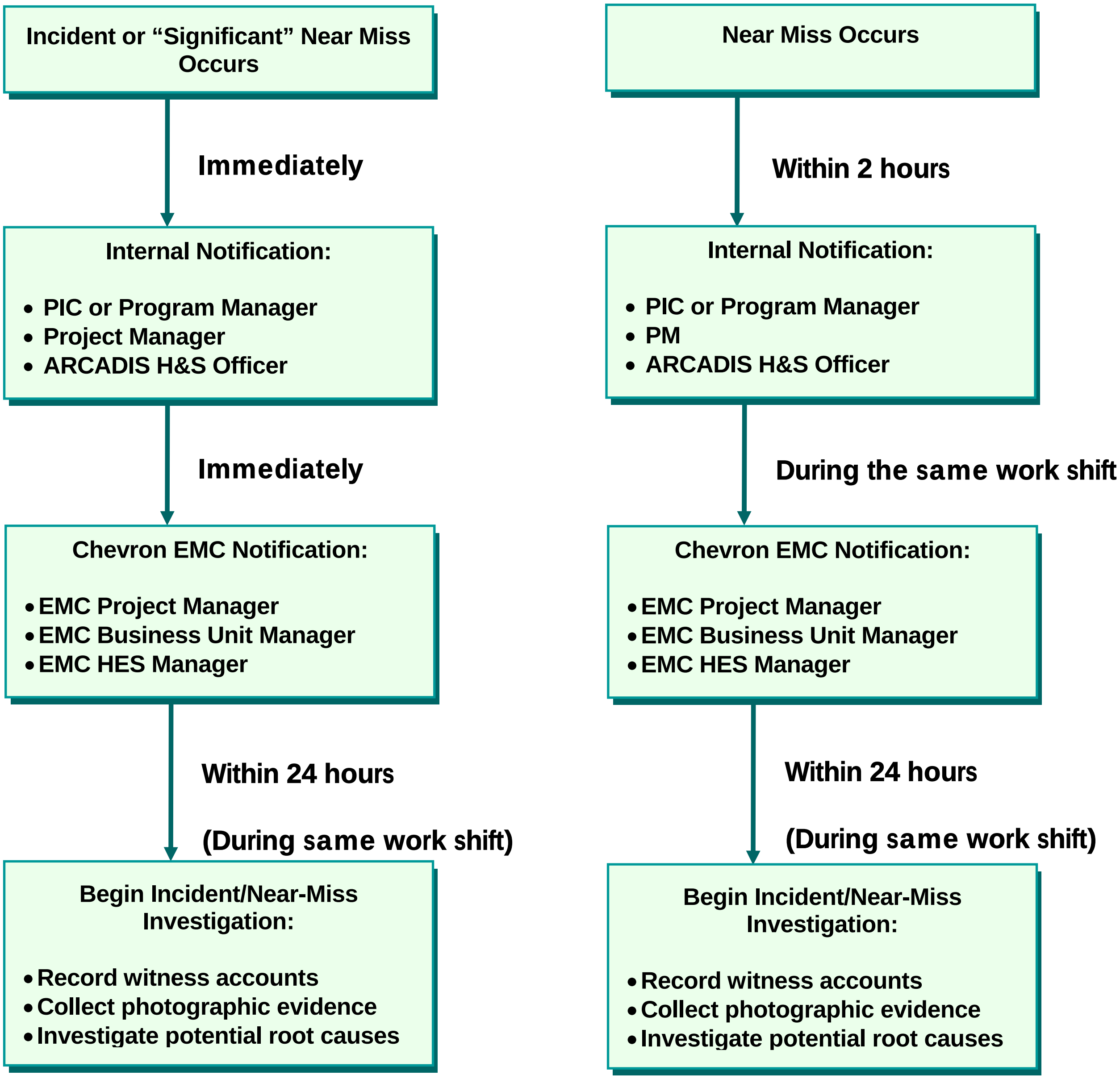
The purpose of the IRIP is to ensure that workplace incidents and significant near-misses are reported and investigated in a timely manner and appropriate corrective actions occur. The IRIP provides instructions regarding who to notify, notification timing and how to investigate and report work related injuries, illnesses and other covered incidents or near misses.

The process may be viewed its entirety at the following link:

https://emconeteam.chevron.com/oe/process_IRIP.aspx

The following Incident and Near-Miss Reporting Process Flow Chart provides information regarding notification timeliness and primary contact.

Incident and Near-Miss Reporting Process Flowchart



Incident or “Significant” Near Miss Occurs

Immediately

Internal Notification:

- PIC or Program Manager
- Project Manager
- ARCADIS H&S Officer

Immediately

Chevron EMC Notification:

- EMC Project Manager
- EMC Business Unit Manager
- EMC HES Manager

Within 24 hours

(During same work shift)

Begin Incident/Near-Miss Investigation:

- Record witness accounts
- Collect photographic evidence
- Investigate potential root causes

Near Miss Occurs

Within 2 hours

Internal Notification:

- PIC or Program Manager
- PM
- ARCADIS H&S Officer

During the same work shift

Chevron EMC Notification:

- EMC Project Manager
- EMC Business Unit Manager
- EMC HES Manager

Within 24 hours

(During same work shift)

Begin Incident/Near-Miss Investigation:

- Record witness accounts
- Collect photographic evidence
- Investigate potential root causes

Additionally, an IRIP Notification and Reporting Guide is included that illustrates response and notification requirements based upon the incident type and level of severity.

2010 CEMC Reporting Guide

	NOTIFICATIONS (to progress up "chain of command")										DOCUMENTATION				
	Chevron Chairman	CEIC	Chevron VP HES	Chevron Legal	CEMC President	General Manager	CEMC OE Mgr	BU OE/HES	CEMC Project Mgr	Additional Notifications	Flash Report	Incident Report	Safety Share	IF Report	Additional Forms
NEAR MISSES															
Low Potential Near Miss									**D**			*Short(7)*			Reported only to the BU of incident
High Potential Near Miss			Notify as Needed					**P**	**I**		X	Long	O	O	
INJURY / ILLNESS															
First Aid Injury w/ Low Potential (not likely to have been serious injury)								**P**	**D**			*Short(7)*			Reported only to the BU of incident
First Aid Injury w/ High Potential (likely could have been serious injury)			Notify as Needed					**P**	**I**		X	Long	O	O	
Minor injury requiring Medical Treatment (5)					**P**	**P**	**P**	**P**	**I**		X	Long	X	X	
Serious Injury (e.g. Days with Restricted Work (DARW) or Days Away from Work (DAFW))			**D**		**P**	**I**	**I**	**I**	**I**		X	Long	X	X	Fatality & DAFW Cases Detail Report (1) (8)
Multiple Serious Injuries requiring overnight hospitalization (except for observation only)	**P**	**I**	**P**	**P**	**I**	**I**	**I**	**I**	**I**	OSHA (3)	X	Long	X	X	(1)
Injury w/ permanent disability			**D**	**P**	**P**	**I**	**I**	**I**	**I**		X	Long	X	X	(1)
Work-related fatality of employee, contractor or 3rd party	**P**	**I**	**P**	**P**	**I**	**I**	**I**	**I**	**I**	OSHA (3)	X	Long	X	X	Fatality & DAFW Cases Detail Report (1) (8)
Non-work-related fatality of employee, contractor or third party			**D**	**P**	**P**	**I**	**I**	**I**	**I**		Not Applicable				

	Chevron Chairman	CEIC	Chevron VP HES	Chevron Legal	CEMC President	General Manager	CEMC OE Mgr	BU OE/HES	CEMC Project Mgr	Additional Notifications	Flash Report	Incident Report	Safety Share	IF Report	Additional Forms
Occupational hearing loss cases (CVX employee only)					P	P	P	I	I		X	Long	O	O	
Repetitive stress injury (RSI) cases w/ Medical Treatment (CVX Employee only)					P	P	P	I	I		X	Long	O	O	
MOTOR VEHICLE CRASHES (MVC)															
MVC-Light					P	P	P	P	I		X	Long			GO407(2)
MVC-Serious					P	P	P	P	I		X	Long	X		GO407(2)
MVC-Major					P	P	P	P	I		X	Long	X	O	GO407(2)
MVC-Catastrophic (Involves fatality)	P	I	P	P	I	I	I	I	I		X	Long	X	X	GO407(2)
SPILLS / RELEASES(6)															
Petrol. spill < 1gal or Chemical spill <16kg to land/container						P	P	P	P	Check local regulations		*Short(7)*			Reported only to the BU of incident
Petroleum spill to land/container ≥ 1gal to <1 bbl to land or container					P	P	P	P	I		X	Long			GO140
Petroleum spill to land/container ≥ 1bbl to <50 bbls					P	I	I	I	I		X	Long	X	O	GO140
Petroleum spill to land/container ≥ 50 barrels to 500 bbls			P		P	I	I	I	I		X	Long	X	X	GO140 + SVRD Report
Petroleum spill to land/container ≥ 500 bbls	P	I	P	P	I	I	I	I	I		X	Long	X	X	GO140 + SVRD Report
Petroleum spill to water (sheen to <50 bbls)			P		P	I	I	I	I	NRC (4)	X	Long	X	O	GO140
Petroleum spills to water ≥ 50 bbls	P	I	P	P	I	I	I	I	I	NRC (4)	X	Long	X	X	GO140 + SVRD Report
Chemical spill ≥16 kg to <160 kg					P	P	P	P	I	Check local regulations	X	Long			GO140
Chemical spill ≥ 160 kg to <8,000 kg					P	I	I	I	I		X	Long	X	X	GO140
Chemical release to land, water, or air greater than 8000 kg. or that	P	I	P	P	I	I	I	I	I		X	Long	X	X	GO140 + SVRD Report

	Chevron Chairman	CEIC	Chevron VP HES	Chevron Legal	CEMC President	General Manager	CEMC OE Mgr	BU OE/HES	CEMC Project Mgr	Additional Notifications	Flash Report	Incident Report	Safety Share	IF Report	Additional Forms
threatens human safety or health or adverse impact to environment															
Release of produced gas, natural gas, or LPG greater than 10 MMSCF or that resents fire/explosion hazard to populated area.	**P**	**I**	**P**	**P**	**I**	**I**	**I**	**I**	**I**		X	Long	X	X	GO140 SVRD Report
Inadvertent Release of Hazardous Vapor (IRHV) – see IRIP Section 2.3.4 (e) for reporting thresholds	**-**	**-**	**-**	**-**	**P**	**I**	**I**	**I**	**I**		X	Long	X	O	GO140 + SVRD Report
COMPLIANCE															
Releases, discharges, or emissions exceeding permitted (or other legal) limits to air, water, land						**P**	**P**	**P**	**I**		X	Long			
Citations (e.g. NOV) received from a governmental enforcement entity						**P**	**P**	**v**	**I**		X	Long			
Fines and/or settlement payments from a governmental enforcement entity						**P**	**P**	**P**	**I**		X	Long			
SECURITY															
Trespass w/o loss						P	P	P	P			*Short(7)*			
Theft or vandalism less than $10,000						P	P	P	I			*Short(7)*			
Theft or vandalism >$10,000					P	P	P	P	I		X	Long	X		
Security incident w/ injury					P	P	P	P	I		X	Long	X		
Kidnapping and ransom	See CVX Global Security Guideline								**I**		See CVX Global Security Guideline				
Murder									**I**						
FIRES AND EXPLOSIONS															
Fires or explosions resulting in <$1,000 damage and/or results in					**D**	**P**	**P**	**P**	**I**		X	Long	X		GO106

	Chevron Chairman	CEIC	Chevron VP HES	Chevron Legal	CEMC President	General Manager	CEMC OE Mgr	BU OE/HES	CEMC Project Mgr	Additional Notifications	Flash Report	Incident Report	Safety Share	IF Report	Additional Forms
any first aid injury															
Fires or explosions resulting in >$1,000 damage and/or results in an injury requiring medical treatment			**P**		**P**	**I**	**I**	**I**	**I**		X	Long	X	X	GO106
Fire, explosion, well blowout or other incident damaging Company and/or third party assets with costs likely to exceed $500,000 for physical damage, production loss of product or production and incident response	**P**	**I**	**P**	**P**	**I**	**I**	**I**	**I**	**I**		X	Long	X	X	GO106
PROPERTY DAMAGE															
Property damage <$10,000						**P**	**P**	**P**	**I**			*Short(7)*			Reported only to the BU of incident
Property damage >=$10,000 to <100,000			**P**		**P**	**I**	**I**	**I**	**I**		X	Long	X	O	
Property damage >=$100,000			**P**		**I**	**I**	**I**	**I**	**I**		X	Long	X	X	
MEDIA ATTENTION															
Incidents that attract little/no media attention									**D**						
Incidents that could have a local impact on Chevron's reputation					**P**	**P**	**P**	**P**	**I**						
Incidents that attract significant local media attention	**P**	**I**	**P**	**P**	**I**	**I**	**I**	**I**	**I**						
Incidents w/ international or broad US media attention	**P**	**I**	**P**	**P**	**I**	**I**	**I**	**I**	**I**						
OTHER															
Incident resulting in the need for employees to shelter-in-place or evacuate	**P**	**I**	**P**	**P**	**I**	**I**	**I**	**I**	**I**						
Natural disaster, political unrest, civil disturbance, or other situation that threatens safety, health, or welfare of employees or contractors	**P**	**I**	**P**	**P**	**I**	**I**	**I**	**I**	**I**						

	Chevron Chairman	CEIC	Chevron VP HES	Chevron Legal	CEMC President	General Manager	CEMC OE Mgr	BU OE/HES	CEMC Project Mgr	Additional Notifications	Flash Report	Incident Report	Safety Share	IF Report	Additional Forms
Incidents resulting in the need for employees or public to shelter-in-place or evacuate.	P	I	P	P	I	I	I	I	I						
Total Cost (before tax) > $100,000 to <$500,000		D	P	P	I	I	I	I	I						
Total Cost of Incident (before tax) >=$500,000		P	P	v	I	I	I	I	I						COI Report
Incidents that could have national or international impact on Chevron's reputation	P	I	P	P	I	I	I	I	I						

Abbreviations and Notes:

"I" = Immediate Notification Required

"P" = Prompt Notification (As soon as possible after gathering necessary info)

"D" = Notify within 24 Hours

"SVRD" = Spill and Vapor Release Detail Report (see IRIP Attachment J)

"COI Report" = Cost of Incident Report (see IRIP Attachment O)

"NRC" = US Coast Guard National Response Center (800-424-8802)

"O" = Reporting Requirement Optional -- and to be determined by GM,Area Mgr/Tm Ldr

"X" = Report Mandatory

"Low" = "Low Potential Incident" (see Sec 2.5)

"High" = "High Potential Incident" (see Sec 2.5)

(1) USCG Form 2692 may be needed for serious injuries or fatalities from offshore rig, vessel or diving accidents

(2) GO-407 required only for CVX Employees in motor vehicle crashes

(3) Notify OSHA within 8 hours of any work-related fatality or the in-patient hospitalization of three or more employees

(4) Notify US Coast Guard National Response Center (800-424-8802)

(5) Refer to HES Rep for definition of "Medical Treatment"

(6) Spills of mixtures – such as an oil/water mixture or chemical contaminated soil – should exclude water or soil volume; only the volume of oil or chemical should be reported

(7) OTIPS INLI form or other incident report form may be used as an alternative to the Short Form. Reported only to the BU of incident

(8) Spills that resulted in (i) workforce DAFW, (ii) workforce or third party Fatalities, or (iii) third party hospitalizations (requires admittance)

G1.4 Motor Vehicle Safety Process

Chevron has a Motor Vehicle Safety (MVS) Process that mainly affects motor carriers and vehicle operators under Chevron Operational Control. Traveling to and from project sites, between project sites, traveling to lunch or to purchase supplies, etc. are NOT under Chevron Operational Control. However, travel on site is under Chevron Operational Control and may affect ARCADIS employees. This is particularly important at large facilities. Chevron's Motor Vehicle Safety Process is located at https://emconeteam.chevron.com/oe/process_MVS.aspx.

G.1.4.1. MVS Variance Process

Chevron has established a MVS Variance Process in the event Suppliers cannot adhere to all of the process. Use of this process is very unlikely; however, in the event it becomes necessary, ARCADIS must define the current circumstances on a MVS Variance Request Form and submit it to the Chevron MVS Advisor and the Chevron Area Manager/Team Leader within 20 business days. ARCADIS must detail what will be done to ensure an acceptable level of risk. The most current MVS Variance Form may be found at https://emconeteam.chevron.com/oe/process_MVS.aspx.

G2. Roles and Responsibility

G2.1 All Personnel

All ARCADIS and subcontractor personnel will receive training in accordance with applicable regulations and be familiar with the requirements and procedures contained in this E-HASP prior to initiating site activities. In addition, all personnel will attend an initial hazard briefing prior to beginning work at the site and take a written test that documents their understanding of the site-specific risks. The test can be found in Appendix E. The test will be administered at the beginning of each phase of work for personnel involved in that specific phase of work. A phase of work may involve more than 1 day and more than one specific task.

G.2.1.1. Use of Loss Prevention System

All ARCADIS and subcontractor personnel that will be performing or overseeing work on this project must attend a LPS training session. This training session explains the objectives, elements, and requirements of LPS to personnel that will be expected to perform or oversee work on Chevron property or sites. Personnel will not be allowed to perform work on Chevron property or sites prior to attending an LPS training session. Contractors performing low-risk work and of short duration (totaling less than 40 hours in any 12-month rolling period) may watch a LPS video instead of the full LPS training session. Proof of this training must be maintained by the HSS. This policy is at the discretion of the Chevron PM.

G2.1.2. Stop Work Authority

Every ARCADIS employee, ARCADIS subcontractor and Chevron employee at a Chevron work site is empowered, expected and responsible for stopping the work of another coworker if the working conditions or behaviors are considered unsafe. No repercussions will result from this action.

If anyone is discouraged from exercising the "Stop Work Authority" or are penalized for doing so, they should report this action to ARCADIS' Principal-In-Charge. If corrective measures are not satisfactorily implemented in a timely manner, they should report this action to the Chevron Project Manager or Chevron's (Health and Environmental Safety) Manager at (713) 432-2645.

G2.1.3. Tenets of Operational Excellence

The Tenets of Operation Excellence (OE Tenets) are an extension of the Chevron's values and principles that apply to all ARCADIS employees, ARCADIS subcontractors, and Chevron employees at Chevron work sites. Any deviation from "always" in the OE Tenets requires a Management of Change, including a risk assessment with involvement of appropriate subject matter experts. A Management of Change Form and the Management of Change Tracking Log is included in Appendix E. The following OE Tenets are intended to protect people, the environment, and equipment:

1. Always operate within design or environmental limits.

2. Always operate in a safe and controlled condition.

3. Always confirm that safety devices are in place and functioning.

4. Always follow safe work practices and procedures.

5. Always meet or exceed customer's requirements.

6. Always maintain integrity of dedicated systems.

7. Always comply with all applicable rules and regulations.

8. Always address abnormal conditions.

9. Always follow written procedures for high-risk of unusual situations.

10. Always involve the right people in decisions that affect procedures and equipment.

Every ARCADIS employee, ARCADIS subcontractor, and Chevron employee at a Chevron work site must take action if a tenet is violated.

Well designed and effectively implemented processes are necessary to deliver Operational Excellence (OE). CEMC currently has 27 OE processes. Eight of these OE processes directly affect H&S on ARCADIS projects. These OE processes include requirements, roles and responsibilities for ARCADIS.

The following processes should be viewed in their entirety by clicking on the links provided:

https://project.bbl-inc.com/IPS/index.cfm?TabID=2

G2.2. Special Emphasis Programs

G2.2.1 Short Service Employee Program.

Recognizing that employees who are new to ARCADIS or one of their subcontractors are at a greater risk for incidents, the following guidelines are established to identify those employees and ease their transition. Short-Service Employees (SSEs) will have an assigned field mentor to assist them in adjusting to the project requirements and procedures. SSEs will be identified in the field by wearing an orange hardhat or hat. The following procedures apply to SSEs:

- Employees new to ARCADIS or one of their subcontractors will be designated as an SSE for 6 months.
- ARCADIS or subcontractor firm employees with less than 6 months experience in a similar job will be designated as an SSE.

Additionally, the following apply:

- All SSEs have to be approved by the CEMC PM by submitting the SSE Form. The SSE Form is included in Appendix E.
- A two to four person crew may have only one SSE onsite.
- A crew of five or more may have no more than 20 percent of the crew staffed with SSEs onsite.

G2.2.2. Hand Safety

The objective of this guidance is to remind all employees, suppliers and subcontractors working on retail and terminal sites of this potential workplace hazard and to understand the various levels of protection that are available for hand protection. It is our collective responsibility to select and apply the right level of protection.

- Brightly colored gloves are provided by Suppliers to their employees once per month; color changed once per month.
- Glove type must be appropriate for the task.

- Hand stickers are placed on equipment to remind everyone of pinch points.

- Work Commentary– Helps with communicating to each other what is going on and what your co-worker(s) will be doing next.

- Stop and Lock – Stop all moving parts before placing hands on them, let the energy out of moving parts, make sure hands are away from potentially moving part before adding energy.

- JSA revisions to accurately define work task risks to hands.

- Specifically define what is minimum PPE for the job (e.g., Nitrile, cotton, leather, vinyl coated, Kevlar, steel mesh gloves). Not simply have “PPE as appropriate.”

G2.2.2.1.. Fixed Open-Blade Knives

Fixed Open Blade Knives (FOBK) are prohibited without written approval from MBU. You must use an alternative cutting tool that is suitable for the job whenever possible.

G2.2.3. Excavation Safety and Borehole Clearance

The overall goal of this program is to provide a procedural outline for utility clearance that can be applied to all facility investigations, and to minimize, if not eliminate, potential for damage caused by hitting utilities during intrusive operations. When applicable, crews must hand clear subsurface utilities to a depth of 8 feet below the ground surface.

G.2.2.4. Traffic Control

The objective is to standardize and clarify minimum expectations for practices in traffic control.

Personnel working on and around retail and terminal sites must be aware of workplace hazards related to traffic control. Personnel must also understand the various levels of workspace protection and personal protective equipment requirements.

G.2.2.5. Drug, Alcohol, and Search Policy

Employees will comply with CEMC’s Drug, Alcohol, and Search Policy (Exhibit-Safety Regulations, Contract No. 99014516). According to the Policy:

- ARCADIS shall have the right to perform controlled substance and alcohol tests based on a reasonable belief by ARCADIS or CEMC that an individual is using alcohol or drugs on the basis of specific physical, behavioral, or performance indicators.

- ARCADIS shall have the right to remove any employee from performing work if the individual exhibits unusual job behavior or unacceptable job performance and it is believed by ARCADIS or CEMC that he/she may be using controlled substances or presently under the influence of alcohol.

Work should be stopped immediately if at any time an employee or subcontractor's employee is suspected of being under the influence. The employee's supervisor should be contacted and arrangements made for transportation for the employee from the site. The cut-off level for a positive alcohol test is a blood alcohol concentration of no more than 0.04 percent.

G2.3. Verification and Validation

Safety processes are verified and validated through use of OTIPS and through Level III safety audits.

G2.4. One Team Incident Prevention System (OTIPS)

CEMC utilizes a behavior-based safety system database known as One Team Incident Prevention System (OTIPS). ARCADIS' LPS database is similar to CEMC's OTIPS, in that both systems use a set of tools to systematically prevent incidents or, at a minimum, reduce their severity. ARCADIS employees are required to have ARCADIS-led LPS training or CEMC OTIPS training prior to working on a CEMC job site.

The OTIPS site may be accessed at:

https://project.bbl-inc.com/IPS/index.cfm?TabID=2

G.2.4.1. Chevron Compliance Reporting Process and Hotline

Chevron has established a **Compliance Reporting Hotline Number at 800.284.3084.** This is a risk-free and anonymous way to report to Chevron, a Chevron or Contractor violation of U.S., State or Local Laws and Company Policies, including but not limited to:

- Health, safety and environmental (not reporting incidents).

- Discrimination/harassment/retaliation.

- Conflicts of interest.

- Theft, bribes, kickbacks.
- Fraudulent transactions.

When appropriate (when appropriate action is not taken by ARCADIS and/or Chevron management), Chevron contractors may use the reporting hotline.

G2.4.2. Near-Loss Reporting Hotline

In an effort to streamline near-miss reporting, especially for employees conducting field work who do not have real-time access to the web, a Near-Miss Reporting Hotline is available. The hotline will be checked daily and data will be entered into the Chevron OTIPS with the caller listed as the primary contact for the event. All entries will be saved as initial and can be accessed by the caller when they return to their computers. Entry into the database does not relieve the caller from the responsibility of following through with the near-miss investigation or of notifying other employees in the office or project team of the occurrence.

NEAR-LOSS REPORTING HOTLINE: 1.866.242.4304

G2.4.3. Short-Service Employee Program

Recognizing that employees who are new to ARCADIS or one of their subcontractors are at a greater risk for incidents, the following guidelines are established to identify those employees and ease their transition. SSEs will have an assigned field mentor to assist them in adjusting to the project requirements and procedures. SSEs will be identified in the field by wearing an orange hardhat or hat. The following procedures apply to SSEs:

- Employees new to ARCADIS or one of their subcontractors will be designated as an SSE for 6 months.
- ARCADIS or subcontractor firm employees with less than 6 months experience in a similar job will be designated as an SSE.

Additionally, the following apply:

- All SSEs have to be approved by the Chevron PM by submitting the SSE Form (Appendix E).
- A two to four person crew may have only one SSE onsite.

- A crew of five or more may have no more than 20 percent of the crew staffed with SSEs onsite.

G2.5. All Onsite Personnel

All onsite personnel (including subcontractors) must read and acknowledge their understanding of this E-HASP before commencing work and abide by the requirements of this E-HASP. All onsite personnel shall sign the E-HASP Acknowledgement Form following their review of this E-HASP. In addition, all personnel will take and pass (passing is 90%) a written test that documents their understanding of the site-specific risks. Employees who do not pass the test will receive one-on-one retraining and will be given the opportunity to retake the exam. Employees who fail the exam the second time will not be allowed to work that day onsite but may come back at a later time to retake the exam. The test can be found in Appendix E.

G2.5.1. Use of Loss Prevention System

All onsite personnel that will be performing or overseeing work on this project must attend a Chevron LPS training session. This training session explains the objectives, elements, and requirements of LPS to personnel that will be expected to perform or oversee work on Chevron property or sites. Personnel will not be allowed to perform work on Chevron property or sites prior to attending an LPS training session.

All ARCADIS and subcontractor personnel will receive training in accordance with applicable regulations, and be familiar with the requirements and procedures contained in this E-HASP prior to initiating site activities. In addition, all onsite personnel will attend an initial hazard briefing prior to beginning work at the site and the daily safety meetings.

All onsite personnel must perform an LPSA prior to beginning each work activity. The LPSA process is synonymous with TRACK. Details of TRACK are presented in the ARCADIS Loss Prevention System™ Handbook. This process must be performed prior to beginning each activity and must be performed after any near miss or other incident in order to determine if it is safe to proceed. Chevron LPSA pocket cards are to be carried by each site worker or posted at the job site. Onsite personnel will immediately report the following to the HSO or HSS:

- Personal injuries and illnesses no matter how minor.

- Unexpected or uncontrolled release of chemical substances.

- Symptoms of chemical exposure.

- Unsafe or hazardous situations.
- Unsafe or malfunctioning equipment.
- Changes in site conditions that may affect the health and safety of project personnel.
- Damage to equipment or property.
- Situations or activities for which they are not properly trained.
- Near misses.

G2.6. Photographs

The taking of photographs is not permitted while onsite without approval from the client.

G2.7. Visitors

All visitors to ARCADIS work areas must check in with the HSS. Visitors will be cautioned to avoid skin contact with surfaces, soil, groundwater or other materials that may impacted or be suspected to be impacted by constituents of concern (COCs). Visitors must complete a Site-Specific Visitors Test prior to being escorted by the HSS. Visitors must remain in contact with the HSS, or HSS-designated individual at all times. The Site-Specific Visitors Test can be found in Appendix E.

Visitors requesting to observe work at the site must wear appropriate PPE prior to entry to the work area and must have the appropriate training and medical clearances to do so. If respiratory protective devices are necessary, visitors who wish to enter the work area must have been respirator-trained and fit tested for a respirator within the past 12 months. No photographs are permitted while onsite without approval from the client.

G2.8. Operational Control

Operational control determines whether CEMC personnel or ARCADIS personnel are responsible for managing CEMC's Permit to Work process. Section 12.1 of the E-HASP provides information relating to CEMC operational control at this facility. When CEMC is the permit issuing authority at the project site, ARCADIS personnel are expected to assist with form completion and meet all of the hazard assessment and pre-job work requirements at the site. When CEMC does NOT have operational control at the facility, ARCADIS personnel are required to complete the General Permit-to-Work and High-Risk Permit-to-Work, when applicable. These forms are tailored for CEMC projects.

G3. General Safety Practices

G3.1. General Safety Rules

- The E-HASP and this Appendix must be in a location at the site that is readily available to personnel and all project personnel shall review and sign this E-HASP prior to starting work. Copies of this E-HASP, if they are necessary, must be updated when the primary E-HASP is updated. Outdated copies or sections of the E-HASP are to be discarded.

- Employees operating heavy equipment must have a letter from his/her supervisor indicating qualifications and/or prior training on the specific piece of equipment.

- When passenger vehicles are in motion, all occupants must be seated in designated areas of the passenger vehicle and wear safety belts. Personnel may not occupy the bed of trucks or trailers while the vehicle is in motion.

- Cell phone use while driving is prohibited when working on all ARCADIS and Chevron projects. This includes driving onsite, as well as driving to and from the site. Hands-free devices are also prohibited.

G3.2. Drug, Alcohol and Search Policy

Employees will comply with Chevron’s Drug, Alcohol, and Search Policy (Exhibit-Safety Regulations, Contract No. 99014516). Subcontractors, by acceptance of ARCADIS Subcontractor Agreement Form and the Schedule “E” Flowdown provisions, must also comply with this policy. According to the Policy:

- CONTRACTOR (ARCADIS) shall have the right to perform controlled substance and alcohol tests based on a reasonable belief by CONTRACTOR or COMPANY (Chevron) that an individual is using alcohol or drugs on the basis of specific physical, behavioral, or performance indicators.

- CONTRACTOR shall have the right to remove any employee from performing work if the individual exhibits unusual job behavior or unacceptable job performance and it is believed by CONTRACTOR or COMPANY that he/she may be using controlled substances or presently under the influence of alcohol.

Work should be stopped immediately if at any time an employee or subcontractor’s employee is suspected of being under the influence. The employee’s supervisor should be contacted and arrangements made for transportation for the employee from the site. The cut-off level for a positive alcohol test is a blood alcohol concentration of no more than 0.04 percent.

G3.3. Contact Lenses

According to NIOSH, wearing contact lenses does not appear to require enhanced eye and face protection. For chemical vapor, liquid or caustic dust hazards, the minimum protection consists of well-fitting non-vented or indirectly vented goggles or full-facepiece respirators. Close-fitting safety glasses with side protection provide limited chemical protection but do not prevent chemicals from bypassing the protection. Workers should wear face shields over other eye protection when needed for additional face protection but they should not wear face shields instead of goggles or safety glasses – regardless of contact lens wear. ARCADIS personnel and subcontractors are permitted to wear contact lens at this site when performing routine tasks identified in this E-HASP.

G3.4. Use of GFCI Line Cord

GFCI line protection must be utilized when operating “portable corded equipment and power tools”. Even if built in line protection exists, such as on a generator or other power source, ARCADIS requires the use of a GFCI line cord or “whip cord” at the power providing end of the setup. It is important that the GFCI be placed as close to the power source as possible.

G3.5. Spill Control

All personnel must take every precaution to minimize the potential for spills during site operations. All onsite personnel shall immediately report any discharge, no matter how small, to the HSS.

Spill control equipment and materials will be located onsite at locations that present the potential for discharge. All sorbent materials used for the cleanup of spills will be containerized and labeled appropriately. In the event of a spill, the HSS will follow the provisions in Appendix E of the E-HASP to contain and control released materials and to prevent their spread to offsite areas.

G3.6. Lock Out Tag Out

Personnel must adhere to ARCADIS Lock Out/Tag Out (LOTO) (re: ARC HSFS004-Control of Hazardous Energy (Lockout/Tagout) procedures when de-energizing equipment. This typically occurs when maintenance is required on energized equipment, hoses or lines. ARCADIS personnel must notify Chevron onsite employees prior to applying a LOTO and when removing a LOTO. Always test live circuitry with a voltmeter, turn off power, apply a lock and tag and retest the circuitry with the voltmeter before proceeding.

G3.7. Traffic Control

A Journey Management Plan (JMP) must be generated for each facility and updated in accordance with the JMP guidelines. The JMP incorporates a TCP to be implemented at the subject facility; this includes the designation of pedestrian and motor vehicle areas at the site. The HSS may make modifications to the TCP, as needed, to address potential changes is site conditions. A detailed TCP, including a site map depicting traffic control measures is included with the JMP in Appendix H of the E-HASP.

All personnel working at the project site must wear Class 2 or higher high-visibility vests. The Class identification is located on the garment tag. Class 3 vests are required for any night-time work or work where traffic flows at over 50 mph. If drums, cones or tubular markers are used to channelize pedestrians, the devices shall be located so that there are no gaps between the bases in order to create a continuous bottom. The cap height of each individual drum, cone or tubular marker shall be no less than 36 inches to be detectable to people who use walking canes. Type II or Type III Barricades are acceptable for use on Chevron project sites.

Type 1 Barricade	

Type 2 Barricade	
Type 3 Barricade	

When establishing work zones in parking lots or other areas with the potential for vehicle traffic, high-visibility traffic cones (with one or more flags) or delineators with a height of 48-inches must be utilized.

G3.6.1. Requirement for Two-Person Crew

If there are any questions regarding the number of personnel required to work safely at the site, a two-person crew must be dispatched for the first site visit. Subsequent review with these employees will determine if the site remains a two-person job. A two-person crew (minimum) is required if the location requires traffic to be redirected into another lane, a public traffic lane is temporarily closed, work is completed along the shoulder of a heavily trafficked roadway or it is deemed otherwise necessary for the safety of the field crew. Consider the buddy system also for work in areas where hybrid or electric vehicles are popular since they can’t be heard at slow speeds.

G3.6.2 Level 1

Use of Level 1 protection may be acceptable for routine tasks of very short duration (less than 10 minutes in one area) where a worker can stay aware of traffic in the vicinity, such as during initial well gauging or surveying.

Maintain a maximum distance of 4-feet between cones and always leave an exit point from the work zone. If possible, use vehicle with flashers activated to provide visual warning and protection from traffic. Use the buddy system or watchperson when conditions warrant.

G3.6.3. Level 2

If tasks of any duration are such that the worker cannot remain alert regarding traffic, traffic control should be elevated to Level 2. Level 2 traffic control is also necessary when working more than 10 minutes in one location, where Level 3 control is not warranted.

Use barrier tape on delineators with flags. Use field vehicles with flashing yellow light or vehicle hazard lights and/or other lane control devices. Barricades may also be used in conjunction with tape to improve warning because of the noise they make if overturned. The buddy system should be used in areas where the worker cannot give undivided attention to traffic when exposed to traffic hazards. Level 2 should be used when temporarily securing an area for such activities as groundwater sampling, hand digging or temporarily isolating an area for storage or moving equipment/supplies.

G3.6.4. Level 3

High task loads that distract attention from vehicle traffic (including noise), work that is performed in low light conditions or when there is a closure of access from public right-of-way to a work site (e.g. station entrance is closed) warrants Level 3 traffic control.

Use plastic security fencing and/or Type 2 or Type 3 barricades. A flashing yellow light on the vehicle may be used for additional visibility. Be cognizant of blocking of pump island or any other facility concerns. Use Level 3 traffic control when where heavy equipment is or power tools in areas near electrically energized or rotating equipment. Use Level 3 as protection for workers or the public near slow-moving traffic or with high traffic volume, in driveways, around tripping hazards and to control third-party foot traffic. If there is no third-party traffic potential, Level 3 is NOT required.

G3.6.5. Level 4

Level 4 traffic control is warranted when work is being performed in a public right-of-way. Traffic control in Level 4 often requires oversight by a licensed traffic controller. Traffic control requirements may vary depending upon the authority having jurisdiction over the roadway, city, county, state or country.

G3.7 Waste Management

The handling and disposal of investigation-derived waste (IDW) material generated at the site during investigative activities will be managed in accordance with ARCADIS Standard Operating Procedure (SOP): Handling and Storing Investigation-Derived Waste. The SOP will be used in accordance with state, local, and Chevron guidelines. Additionally, the site-specific requirement for the COCs will be taken into account for the handling of the IDW at this facility. Waste drums must be labeled according to contents. Generally, drums containing liquids are to be placed on secondary containment and drums containing solids are to be placed on wooden pallets. The Investigative-Derived Waste SOP is available in Appendix I.

G3.8. Hand Safety Program

Following Chevron MBU's initiative, ARCADIS's MBU Team has adopted a Hand Safety Program to reduce the frequency of hand injuries. This policy is applicable to all ARCADIS staff and subcontractors on MBU projects.

The Hand Safety Program focuses on two areas, which are outlined below. If you have any questions regarding these programs or have recommendations, please contact the PIC or your Portfolio Manager.

"Where are Your Hands?" Program

- Brightly colored gloves are to be worn in order to increase visibility and awareness of hand positions. Glove colors will be changed once per month.

- Glove type must be appropriate for the task (e.g., disposable nitrile gloves do not provide adequate protection from broken glassware, cut resistant gloves are to be worn for final tightening of glass vials).

- "Hand stickers" are to be placed on equipment that have hand hazards to remind everyone of pinch points (to be provided by Chevron).

- ***Work Commentary***– Helps with communicating to each other what is going on and what your co-worker(s) will be doing next (daily when working in teams) talk through the highest hand hazard for the day in tailgate or at the start of the task.

- ***Stop and Lock*** – All moving parts must have stopped before placing hands on them, let the energy out of moving parts, make sure hands are away from potentially moving part before adding energy (e.g., Drillers may not grab moving cables).

JLA revisions to accurately define work task risks to hands. JLAs should specifically define the minimum level of PPE for the job (e.g., Nitrile, cotton, leather, vinyl-coated Kevlar, steel mesh gloves) and not simply indicate "PPE as appropriate."

G3.9. Fixed Open Blade Knife (FOBK) Policy

The use of fixed open-blade knives (FOBKs) are prohibited without written approval from the Program Manager. If you are working on an MBU site, you must also receive written approval of MBU. You must use an alternative cutting tool that is suitable for the job whenever possible.

Always perform a thorough JLA to define the proper cutting tool for the task. If an FOBK is the defined alternative then an FOBK Exception Permit must be submitted to your Program Manager for approval. If you are working on an MBU site, the FOBK Exception permit must be signed by the MBU PM and approved by the Area Manager before the task can occur.

If you observe the use of a FOBK exercise Stop Work Authority and verify that a FOBK Exception Permit has been issued or amend the JLA and use an acceptable tool.

G3.10. Jewelry, Hair and Loose Clothing

Jewelry, hair and loose clothing can easily get caught in moving machinery, which may result in serious bodily injury or death. Always remove all jewelry before beginning work and make certain clothing and hair cannot become entangled in moving equipment.

Jewelry should not be worn if it could compromise the integrity of hand protection by tearing or puncturing protective gloves.

G3.11. Site-Specific Safety Rules

Cell phone use is not permitted within the chain-link security fence. Cell phones may be used in front of the main office building. Smoking and other sources of heat/spark/fire are not permitted within the chain-link security fence. The designated smoking area is located north of the office building, along the sidewalk adjacent to the office parking area.

Work may not proceed without the issuance of a daily work permit.

Cameras are not allowed to be used at the facility without permission from the Chevron Project Manager.

G4. Training

G4.1. General

All onsite project personnel who work in areas where they may be exposed to site contaminants must be receive the following training:

- OSHA HAZWOPER (29 CFR 1910.120). This certification requires an annual 8-hour refresher, a portion of which must be job-specific. Personnel who completed their initial training more than 12 months prior to the start of the project must have completed an 8-hour refresher course within the past 12 months.

- Loss Prevention System. This does not require an annual refresher; however, personnel are expected to remain proficient with the system by participating in JLAs, LPOs and IINLIs.

- Smith System® Defensive Driving – Hands-On. Personnel are expected to complete this course every 3 years. A commentary drive must accompany this training program.

G4.2. Permit-to-Work Approvers

In addition the training specified in Section 4.1, Permit-to-Work Approvers must receive OSHA Supervisor training. Additionally, Permit-to-Work Approvers must be familiar with the ARCADIS/CEMC Permit-to-Work Procedure.

Authorized PTW Approvers are designated by the PIC or Program Manager based on appropriate experience and training including:

- OSHA HAZWOPER (40-hour initial and current 8-hour refresher).

- OSHA HAZWOPER Supervisor Training.

- Loss Prevention System (including OE Tenets and Stop Work Authority).

- At least six months in current job (not a Short Service Employee).

- Demonstrated understanding of ARCADIS PTW procedure.

The above are minimum criteria. In addition, the permit approver or other on-site person must have documentation of appropriate training to approve the High Risk Work Permit. The following areas require specialized training:

- Confined Space Entry – Confined space training relevant to OSHA 1910.146.
- Excavation and Trenching – Competency training relevant to OSHA 1926.650.
- Hot Work – Fire prevention and general safety requirements associated with welding, cutting and brazing relevant to OSHA 1910.252.
- Lock out/Tag out – Lock out/Tag out program training relating to OSHA 1910.147.
- Elevated Work – Fall Protection training relevant to OSHA 1926.500.
- Diving – Commercial diving operations relevant to OSHA 1910.401.

G4.3. Management and Supervisors

In addition the training specified in Section 5.1 of the E-HASP, Management and Supervisors must receive OSHA Supervisor training.

G4.4. First Aid and CPR

At least one employee current in first aid/CPR will be assigned to the work crew and will be onsite during operations. Refresher training in first aid and CPR are required to keep the certificate current. The individual must also receive training regarding the precautions and protective equipment necessary to protect against exposure to blood-borne pathogens.

G4.5. Site-Specific Training

Site-specific training will be accomplished by onsite personnel reading this HASP or through a thorough site briefing by the PM or HSS on the contents of this E-HASP before work begins. The review must include a discussion of the chemical, physical, and biological hazards; the protective equipment and safety procedures and emergency procedures. In addition, all personnel entering the work areas will take a written test that documents their understanding of the site-specific risks.

ARCADIS

Appendix H

Journey Management Plan

ARCADIS
Infrastructure, environment, buildings

Imagine the result

Chevron Environmental Management Company

Journey Management Plan

Chevron Facility #6518040
Oceanside, New York

June 2010

ARCADIS

William T. McCune
Principal Geologist

Journey Management Plan

Chevron Facility #6518040
Oceanside, New York

Prepared for:
Chevron Environmental Management Company

Prepared by:
ARCADIS U.S., Inc.
1 International Boulevard
Suite 406
Mahwah
New Jersey 07495
Tel 201.684.1410
Fax 201.684.1420

Our Ref.:
B0047517.0003.00001

Date:
June 2010

1. Introduction **1**

1.1 Plan Objectives 1

1.2 Plan Organization 1

2. Background Information **2**

2.1 Site Description 2

2.2 Operational History 2

3. Remedial Investigation Activities **4**

4. Waste Characterization **6**

5. Traffic Control Transportation and Mobilization Procedures **7**

5.1 Description of Remedial Investigation Activities 7

5.1.1 Site Preparation Activities 7

5.1.2 Drilling and Test Pitting Activities 7

5.1.3 Waste Transportation 7

5.2 State and Federal Regulations 7

5.3 Waste Disposal Facilities 8

5.4 Transportation Modes 8

5.5 Driving/Hauling Routes 8

5.6 Security Traffic Control and Loading Procedures 8

5.7 Record Keeping 9

5.8 Health and Safety Procedures 10

6. Emergency Contacts **11**

Table

Table 6-1. Emergency Contacts 11

Figures

1 Site Location Map

2 Driving Routes

2-1 Driving Route – ARCADIS Syracuse, NY to the Project Site

2-2 Driving Route – ARCADIS Melville, NY to the Project Site

2-3 Driving Route – ARCADIS Cranbury, NJ to the Project Site

1. Introduction

This Journey Management Plan has been prepared by ARCADIS, on behalf of Chevron Products Company (Chevron), to provide a description of transportation procedures for mobilization of personnel to, from, and around the former Gulf Terminal (Terminal) located in Oceanside, NY. Remedial Investigation activities planned at this site include the additional site characterization and pre-design support activities including soil boring, test pit/trench excavations, well installations, and sampling. Figure 1 presents a map of the location of the site.

The purpose of this Journey Management Plan is to minimize potential health, safety, and environmental risks when ARCADIS and contractor personnel mobilize and demobilize to and from the Site, and while navigating onsite during the implementation of remedial activities at the Site. The Journey Management Plan provides procedures for transportation mobilization to and from the Site, how to safely enter and exit the site, as well as procedures for navigating onsite. This plan will be communicated to all ARCADIS and contractor personnel.

1.1 Plan Objectives

The objectives of the Journey Management Plan include the following:

- provide an introduction to the site
- provide a summary of remedial action activities
- provide description of material to be generated
- identify the facilities for waste disposal
- provide transportation modes and routes for ARCADIS and contractor personnel
- provide a sequence of site entry/exit procedures when arriving and departing from the Site

1.2 Plan Organization

The sections of this Journey Management Plan are as follows.

- Section 2 presents background information
- Section 3 provides a summary of remedial action activities
- Section 4 presents a description of wastes generated
- Section 5 presents traffic control, transportation and mobilization procedures
- Section 6 details the contingency plan
- Section 7 presents references

2. Background Information

This section presents a site description, history of operations, and regulatory status of the Site.

2.1 Site Description

The site consists of 7.2 acres located at 1 Industrial Place in Oceanside, Township of Hempstead, Nassau County, New York. A Site Location Map is presented on Figure 1. The site is currently unoccupied and all former buildings have been demolished. The site is bound to the south by Barnum's Channel, to the east by the Long Island Rail Road, to the north by the former Exxon Petroleum Terminal, and to the west by Hampton Road. A junk yard and office buildings are located west of Hampton Road fronting Hog Island Channel. Daly Boulevard is located north of the former Exxon Petroleum Terminal and another petroleum terminal (Sprague) is located north of Daly Boulevard.

2.2 Operational History

The site consists of three lots. A portion of the site (Lot 504) was first developed as a petroleum terminal by Gulf Oil Corporation in 1931. Lots 503 and 502 were purchased by Gulf Oil Corporation in 1950 and 1956, respectively. Chevron acquired Gulf Oil Corporation in the mid 1980s. As part of the purchase, Chevron was required to divest the northeast division. In 1986, Cumberland Farms, Inc. (CFI) purchased the northeast marketing assets of the Gulf Oil Corporation from Chevron. The purchase included the Oceanside Terminal, which was transferred to CFI in May 1986. In December 1993, CFI, in a joint venture with Catamount Petroleum LP (Catamount), formed Gulf Oil Limited Partnership (Gulf). CFI owned two-thirds of Gulf as a limited partner and Catamount owned one-third of Gulf as the general partner. In September 2005, CFI exercised its option to buy out Catamount and reorganized Gulf under a new general partner. However, ownership percentages of Gulf did not change during the reorganization. Gulf remains as the current owner/operator of the site.

Operation of the petroleum terminal was ceased in the early 1990s. The demolition of the aboveground storage tanks (ASTs) at the former petroleum terminal was completed in 2003. The remaining on-site buildings (maintenance building, office building and several small buildings associated with historical petroleum terminal operations) were demolished in 2005. The site is currently vacant.

The site has undergone extensive investigation and remediation since the 1990s under NYSDEC oversight (Spill No. 92-03883). In January 2007, the NYSDEC accepted an application for this site under the Brownfield Cleanup Program (BCP) – Site No. C130165. However, following the decision by prospective developer to withdraw from the BCP in 2009, the NYSDEC transferred the site into the Hazardous Waste Program (State Superfund Site). An Order on Consent and Administrative Settlement was executed between the NYSDEC and CEMC on December 23, 2009 (Index # W3-1142-09-08, Site # 130165).

3. Remedial Investigation Activities

The following site activities will occur during RI activities.

Site activities will commence with a health and safety kickoff meeting. Each work day will also start with a site safety meeting. Site features and investigation areas are identified on Figure 1.

Remedial Investigation activities will generally be performed at the specified locations described below, also identified on Figure 2.

The RI scope of work consists of the following:

1. Inspect existing monitoring wells on site to assess their conditions (e.g., number of existing wells, suitability for sampling).

2. Install additional monitoring wells in the shallow sand fill layer and the deep sand layer for vertical delineation of petroleum impacts as well as CVOCs. ARCADIS proposes to install seven monitoring wells in the shallow sand fill layer: four monitoring wells along the bulkhead and three monitoring wells in VRU area. ARCADIS also proposes to install three deep monitoring wells: two monitoring wells replacing known destroyed wells (MW-26D and MW-31D) and one new monitoring well for vertical delineation (monitoring well MW-25D3) at the former VRU.

3. Perform test pit/trench excavations in the vicinity of the former barge dock and former garage building. A total of nine test pits/trenches will be excavated: six test pits near the former locations of the barge dock buildings and three test pits near the former garage building. The test pits will be excavated to a depth between 5 and 9 feet, depending upon the depth to water. The test pit excavations will be used to assess the extent of residual LNAPL and petroleum impacts in the shallow sand fill layer.

4. Conduct a soil boring investigation at the former VRU, including advancing three soil borings to complete further vertical delineation within the meadow mat. The soil borings will be drilled to an approximate maximum depth of 16 feet.

5. Analyze select soil samples from soil borings and test pits for VOCs, SVOCs, Target Analyte List (TAL) metals, total organic carbon and ferrous iron.

6. Conduct aquifer testing of the deep sand layer. ARCADIS proposes to conduct a step pumping test and/or a 24-hour pumping test at monitoring well MW-30D. MW-30D was selected because this area and depth horizon may be the subject of future focus, to reduce the amount of and exposure to groundwater affected by higher concentrations of CVOCs.

7. Perform tracer testing at the deep sand layer. A nonreactive tracer (e.g., fluorescein) will be used to further assess hydraulic characteristics of the water-bearing units beneath the site.

8. Groundwater sampling of existing and newly installed monitoring wells. The groundwater samples will be analyzed for VOCs, SVOCs, methane, ethane, ethene, total dissolved solids, alkalinity, alkalinity-bicarbonate, sulfate, sulfide, nitrate, nitrite, nitrogen as ammonia, phosphorous as orthophosphate, calcium, total dissolved organic carbon, magnesium (total and dissolved), iron (total and dissolved), and TAL metals (total and dissolved).

9. Conduct specific-capacity testing during the proposed groundwater sampling event (Item 7 above).

10. Conduct bench-scale testing of soil and groundwater to estimate the soil oxidant demand and efficacy of persulfate under differing activation scenarios to evaluate the feasibility of an in-situ chemical oxidation (ISCO) remedial option for saturated soil and groundwater in both the shallow and deep sand layers. Soil samples for bench-scale testing will be collected during installation of the additional monitoring wells and during the soil boring investigation. Groundwater samples for bench-scale testing will be collected during the proposed groundwater sampling event (Item 7 above). Bench-scale tests will be conducted on samples collected from the meadow mat for thermal resistivity. Other bench-scale tests may be conducted, as necessary, as RI work progresses at the site.

11. Perform remediation hydraulics evaluations as needed to refine the understanding of contaminant fate and transport, aquifer architecture, and refinement of the conceptual site model.

4. Waste Characterization

Waste generated during RI activities will be containerized and characterized for appropriate disposal at a Chevron approved facility.

5. Traffic Control Transportation and Mobilization Procedures

This section includes descriptions of some of the remedial action activities, applicable state and federal regulations, waste disposal facilities, transportation modes, driving/hauling routes, onsite traffic control and loading procedures, record keeping, and health and safety procedures.

5.1 Description of Remedial Investigation Activities

5.1.1 Site Preparation Activities

Site preparation activities include identifying investigation locations and areas subject to subsurface activities during the remedial actions. Site visits will be conducted by ARCADIS personnel via personal or rented vehicles.

Site preparation activities also involve appropriate notification to the NYSDEC and the property owner.

5.1.2 Drilling and Test Pitting Activities

Subsurface investigation activities will be performed using appropriate drilling and excavation equipment. Following completion of investigation activities, the required equipment will be decontaminated prior to demobilization from the site. Transportation for this activity generally consists of a mobilization/demobilization. Traffic control on site during each phase of the investigation will be established depending on the area to be investigated and the potential vehicular risks which need to be mitigated.

Stop work will be issued if any unsafe condition is identified during the investigation activities.

5.1.3 Waste Transportation

Waste will be transported via truck from the Site to an appropriate approved facility. Traffic control of these trucks vehicles will be coordinated with property owner. Cones will be used as traffic control. The transporter will be shown a route through the Terminal to avoid conflict with ongoing activities.

5.2 State and Federal Regulations

Material generated during the remedial activities will be considered non-hazardous or hazardous based on material of origin. All appropriate state and federal laws and regulations will be followed during the transport and disposal of these materials. All wastes will be transported with documentation as required by applicable state and federal laws and regulations.

5.3 Waste Disposal Facilities

Investigation derived waste material will be disposed of at a Chevron approved waste facility to be determined following waste characterization.

5.4 Transportation Modes

The following lists transportation mode for site and contractor mobilization and demobilization during the activities described above in Section 3:

- semi-tractor trailer
- box truck
- pick-up truck
- truck-mounted vacuum rig
- automobile

5.5 Driving/Hauling Routes

Driving/hauling routes for all personnel anticipated to perform activities associated with remedial actions onsite are summarized in Attachment. Driving/hauling routes will be appended as needed.

5.6 Security Traffic Control and Loading Procedures

A generalized description of onsite traffic patterns is presented in Figure 1. The following procedures for onsite traffic control and loading will be executed upon arrival at the Site:

- Notify the property owner in advance of all ARCADIS and subcontractor personnel that will be on site.

- Only enter the site via the entrance gate. Contractor personnel will check in with the ARCADIS site supervisor upon arrival.

- Barricades, caution tape, and trucks will be used to set up work zones, prevent unauthorized entrances, and direct vehicular traffic around work zones.

- A uniform waste manifest will be prepared for all vehicles carrying hazardous wastes prior to exiting the Site. Waste manifests will be carried by waste haulers for all hazardous wastes hauled from the Site.

All loading will be done using the appropriate equipment (e.g. hydraulic lift). All equipment and materials will be properly secured before moving the vehicles.

Traffic and mobilization/demobilization procedures will be repeated as necessary.

5.7 Record Keeping

All material will be appropriately manifested with a non-hazardous or hazardous waste manifest prior to transporting material offsite. The following information will be recorded on all manifests:

- Generator’s Contact Information
- Generator’s Name and Signature
- Transporter's Contact Information
- Transporter’s Name and Signature
- Designated Facility Name and Site Address
- Facility Signature and Ticket Number
- Number of Containers
- Container Type
- Total Quantity
- Weight or Volume Units
- PPE Required
- Special Handling Instructions
- Shipment Date

ARCADIS personnel will complete the waste manifests for the hazardous wastes on behalf of Chevron, and a Chevron representative will sign the manifests. Appropriate copies of manifests will be compiled and stored in the project files in Syracuse, NY.

This Journey Management Plan will be transmitted to all ARCADIS and contractor personnel prior to their mobilization to the Site. A record of transmittal will be stored in project files in Syracuse, New York.

In conjunction with the onsite health and safety kick-off meeting, all ARCADIS and contractor personnel will sign the acknowledgement sheet located at the front of the onsite copy of the Journey Management Plan. As new personnel are added to the acknowledgement sheet, copies of this form will be stored in the project files in Syracuse, New York.

5.8 Health and Safety Procedures

All ARCADIS and contractor personnel will be aware of the ARCADIS Driver Safety Program. All drivers must have a valid driving license, and be familiar with the site-specific driving Job Safety Analysis (JSA).

In accordance with the HASP updated September 2007 (ARCADIS, 2007), no person will be allowed in the work area during site operations without first receiving a site orientation and hazard briefing. This orientation will be presented by the Site Supervisor, and will consist of a review of the HASP. Following this initial meeting, daily safety meetings will be held each day before work begins. Daily safety meetings will be held to cover the work to be accomplished, the hazards anticipated, protective equipment and procedures required to minimize site hazards, and emergency procedures. The Site Supervisor will present these meetings prior to beginning the day’s fieldwork. No personnel will mobilize to any work area or perform any work task before the daily safety meeting has been held. The safety meeting will also be held prior to new tasks, and repeated if new hazards are encountered. All people entering site work areas, including visitors, must document their attendance at the site orientation and hazard briefing, as well as the daily safety meeting.

All business-related visitors to the work areas must check in with the ARCADIS Site Supervisor when arriving onsite. Visitors will not be allowed to enter the “Restricted Work Zone" unless they have had all required training and possess the proper personnel protective equipment.

All persons authorized to enter the "Restricted Work Zone" as workers and/or visitors must have the appropriate safety training and medical clearance including OSHA 40 hour training. The ARCADIS Site Supervisor will maintain a log of those individuals entering the “Restricted Work Zone." In the event of an onsite release, emergency procedures will be executed in accordance with the HASP. Prior to the start of operations, the work area will be evaluated for the potential for fire, contaminant release, or other catastrophic event. Unusual conditions or events, activities, chemicals, and conditions will be reported to the Site Supervisor immediately.

The ARCADIS and/or Site Supervisor will establish evacuation routes and assembly areas for the Site. All ARCADIS and contractor personnel entering the Site will be informed of this route and the assembly area.

6. Emergency Contacts

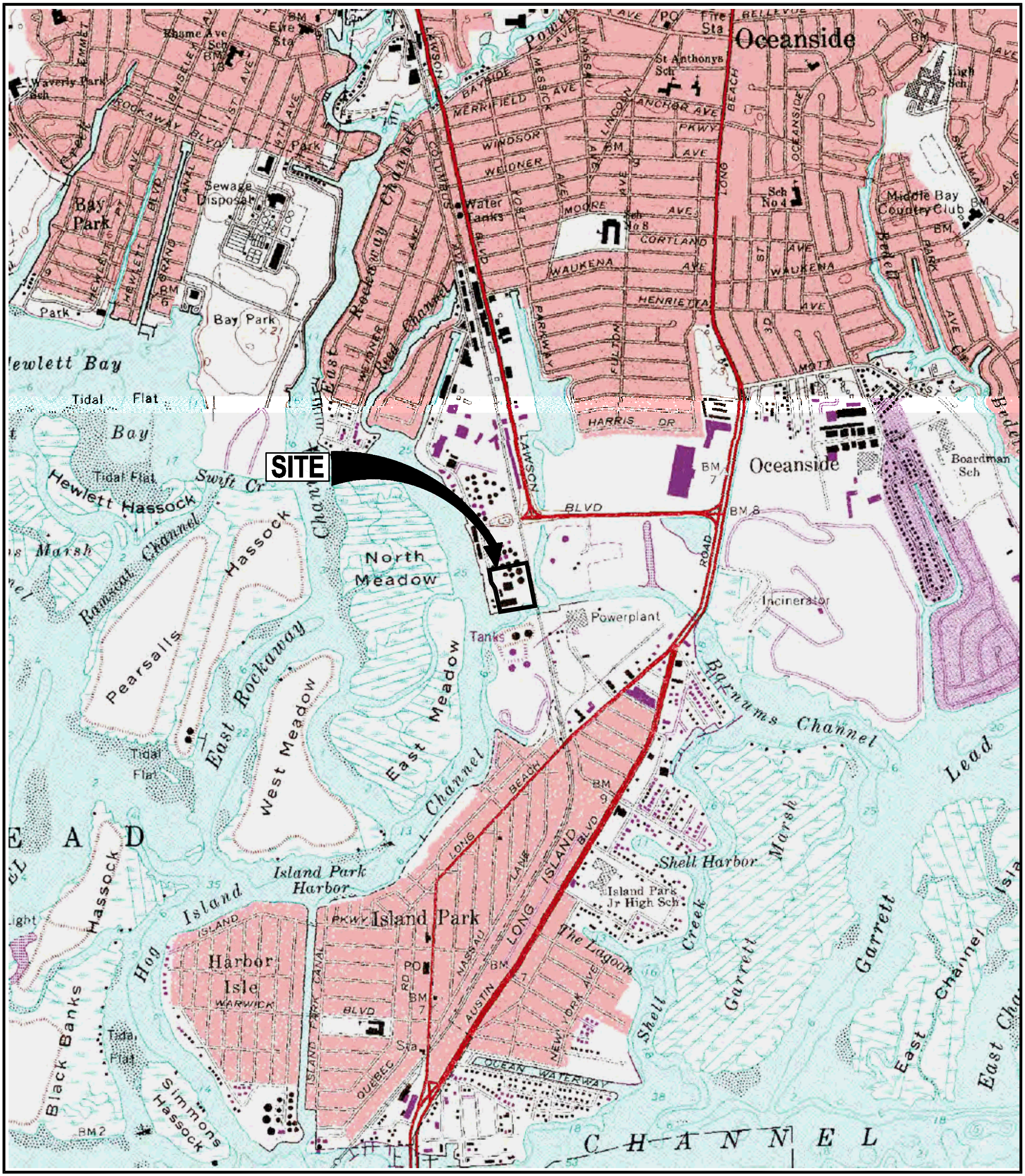
In the event of an emergency, the means to summon local public response agencies such as police, fire, and ambulance are identified in Table 6-1.

Table 6-1. Emergency Contacts

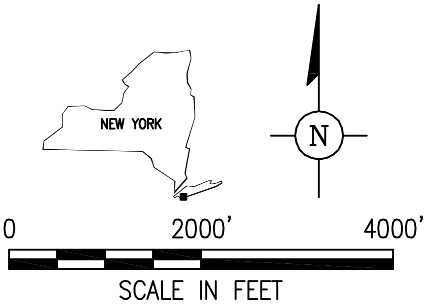
LOCAL EMERGENCY CONTACTS	PHONE NUMBER
Fire Department (Non-Emergency)	911
Local Police (Non-Emergency)	911
Ambulance	911
PROJECT EMERGENCY CONTACTS	**PHONE NUMBER**
Emergency Client Contact: Peter Kasbohm	770.984.3145, if not available, contact switchboard at 770.984.3000 and ask for a Site Assessment and Remediation PM
Gulf L.P.: Don Smith	617.884.5980
ARCADIS Project Manager: Bill McCune	315.671.9172, or 315.420.4348 cell
ARCADIS Site Supervisor: TBD	

For emergencies requiring response from the fire department, police or an ambulance, dial 911. Calmly explain the situation to the security operator and they will coordinate with the appropriate emergency services.

Figures



MAP SOURCE: USGS 7.5 MINUTE QUADRANGLE 1979 LYNBROOK AND LAWRANCE, NEW YORK



GULF OIL TERMINAL
(CHEVRON FACILITY #6518040)
OCEANSIDE, NEW YORK

SITE LOCATION MAP

ARCADIS

FIGURE
1

CITY:Melville - NY DIV/GROUP:ENR1 DB:A.Sanchez LD: PIC: PM: TM: LYR:ON=*;OFF=*REF*
G:\ENVCAD\Melville-NY\RETURN-TO\Mahwah-NJ\B0047517\0003\00001\Location Map.dwg LAYOUT: 1SAVED: 5/6/2010 4:15 PM ACADVER: 17.1S (LMS TECH) PAGESETUP: ---PLOTSTYLETABLE: ARCADIS_MELVILLE.CTB PLOTTED: 5/6/2010 4:15 PM BY: SANCHEZ, ADRIAN

XREFS: IMAGES: PROJECTNAME:
LAWRENCE.TIF
LYNBROOK.TIF
SEA CLIFF.TIF

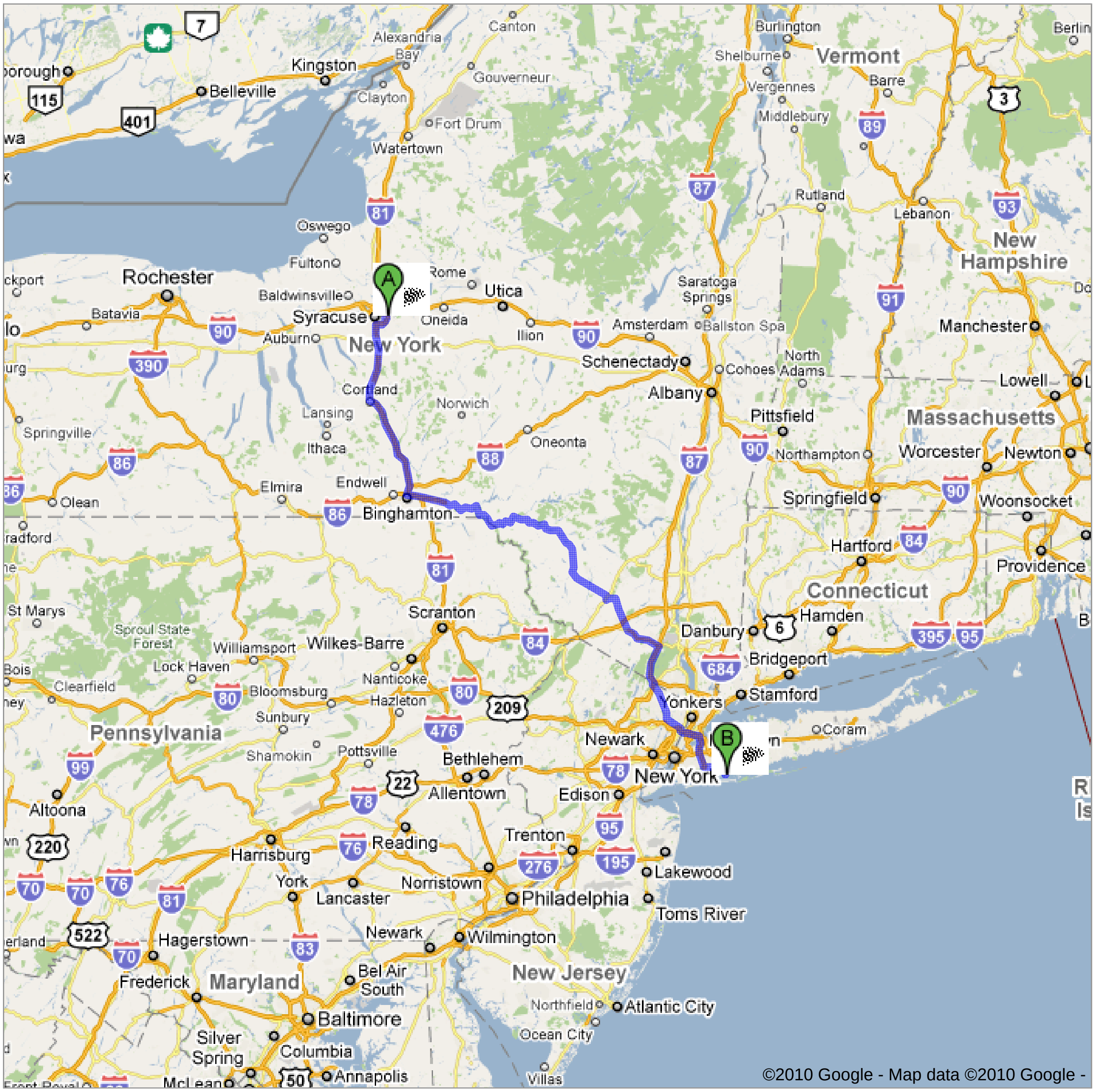
Figure 2

Driving Routes

Figure 2-1

Driving Route – ARCADIS Syracuse, NY to the Project Site

Directions to Hampton Rd, Oceanside, NY 11572
280 mi – about **4 hours 56 mins** – up to 5 hours 50 mins in traffic
Directions From Syracuse Office



©2010 Google - Map data ©2010 Google -

A 6723 Towpath Rd, East Syracuse, NY 13057

	1. Head **northwest** on **Towpath Rd** toward **Widewaters Pkwy** About 1 min	go 0.7 mi total 0.7 mi
↱	2. **Towpath Rd** turns slightly **right** and becomes **Celi Dr**	go 0.2 mi total 0.9 mi
↰	3. Turn **left** at **Bridge St** About 1 min	go 0.1 mi total 1.1 mi
5	4. Turn **left** at **NY-5 E/Erie Blvd E** About 1 min	go 1.1 mi total 2.2 mi
5	5. Turn **left** at **NY-5 E/NY-92 E/E Genesee St**	go 0.1 mi total 2.3 mi
481	6. Merge onto **I-481 S** via the ramp to **Jamesville** About 4 mins	go 4.6 mi total 6.8 mi
	7. Take the exit toward **I-81 S**	go 0.3 mi total 7.1 mi
81	8. Keep **left** at the fork to continue toward **I-81 S** and merge onto **I-81 S** About 1 hour 16 mins	go 73.2 mi total 80.3 mi
86	9. Slight **left** at **I-86 E** About 9 mins	go 9.9 mi total 90.2 mi
17	10. Continue onto **NY-17 E** About 1 hour 42 mins	go 107 mi total 198 mi
6	11. Continue onto **US-6 E** About 12 mins	go 12.5 mi total 210 mi
17	12. Continue onto **NY-17 E** Partial toll road About 1 min	go 1.1 mi total 211 mi
	13. Take the **I-87 S/New York** exit Toll road	go 0.5 mi total 212 mi
87	14. Merge onto **I-87 S** Partial toll road About 13 mins	go 14.1 mi total 226 mi
↱	15. Take exit **15** for **NY-17 S/I-287 S** toward **New Jersey**	go 0.8 mi total 227 mi
287	16. Merge onto **I-287 S** Entering New Jersey	go 0.6 mi total 227 mi
17	17. Take exit **66** on the **left** to merge onto **NJ-17 S** toward **Mahwah** About 18 mins	go 14.1 mi total 241 mi
↱	18. Take the **NJ-4 E** ramp to **Fort Lee/New York**	go 0.1 mi total 242 mi
4	19. Keep **left** at the fork to continue toward **NJ-4 E** and merge onto **NJ-4 E** About 10 mins	go 7.4 mi total 249 mi
	20. Take the **I-95 N** ramp to **G W Bridge/New York City**	go 0.4 mi total 249 mi
9	21. Merge onto **US-9 N** Partial toll road	go 1.6 mi total 251 mi

	Entering New York About 2 mins	
22.	Continue onto **George Washington Bridge/Interstate 95 Upper Level N/U.S. 1 Upper Level N** About 1 min	go 0.8 mi total 252 mi
23.	Continue onto **I-95 N** About 6 mins	go 4.5 mi total 256 mi
24.	Take exit **6A** toward **I-678 S/Whitestone Bridge**	go 0.5 mi total 257 mi
25.	Keep **right** at the fork to continue toward **I-678 S** and merge onto **I-678 S** Partial toll road About 13 mins	go 12.3 mi total 269 mi
26.	Take exit **1 E** on the **left** for **Belt Pkwy E/NY-27 E** toward **Eastern Long Lsland**	go 0.6 mi total 270 mi
27.	Follow signs for **Belt Pkwy E/Eastern Li** and merge onto **Belt Pkwy/Shore Pkwy E** About 2 mins	go 2.5 mi total 272 mi
28.	Exit onto **NY-27 E/S Conduit Ave** Continue to follow NY-27 E About 9 mins	go 5.0 mi total 277 mi
29.	Turn **right** at **Ocean Ave** About 3 mins	go 1.2 mi total 278 mi
30.	Turn **left** at **Atlantic Ave** About 1 min	go 0.2 mi total 278 mi
31.	Take the 2nd **right** onto **Bayview Ave/Lawson Blvd** Continue to follow Lawson Blvd About 1 min	go 0.5 mi total 279 mi
32.	Take the 3rd **right** onto **Weidner Ave**	go 338 ft total 279 mi
33.	Turn **left** at **Royal Ave** About 1 min	go 0.4 mi total 279 mi
34.	Slight **right** at **Hampton Rd** About 1 min	go 0.4 mi total 280 mi

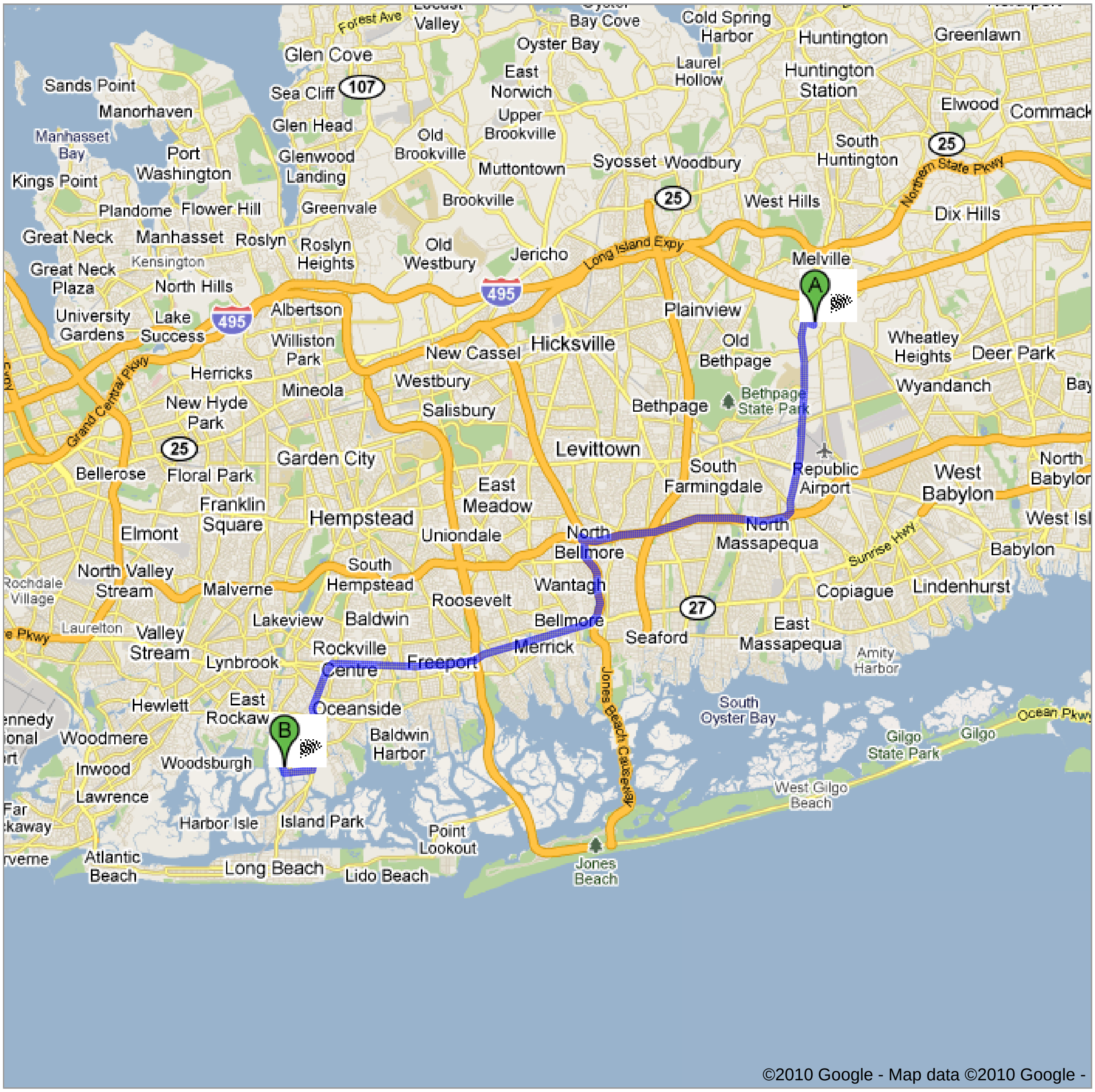
B Hampton Rd, Oceanside, NY 11572

Figure 2-2

Driving Route – ARCADIS Melville, NY to the Project Site

Google maps

Directions to Hampton Rd, Oceanside, NY 11572
21.7 mi – about **33 mins**
Directions from Melville Office



©2010 Google - Map data ©2010 Google -

A 2 Huntington Quadrangle, Melville, NY 11747

	1.	Head **south** on **Huntington Quadrangle** toward **Baylis Rd**	go 387 ft total 387 ft
↱	2.	Turn **right** at **Baylis Rd**	go 0.2 mi total 0.3 mi
110	3.	Take the 1st **left** onto **NY-110 S/Broad Hollow Rd** About 9 mins	go 4.5 mi total 4.7 mi
↗	4.	Take the ramp to **W New York**	go 0.2 mi total 5.0 mi
	5.	Merge onto **Southern State Pkwy** About 4 mins	go 4.8 mi total 9.8 mi
↗	6.	Take exit **27S** for **Wantagh State Pkwy S** toward **Jones Beach**	go 0.2 mi total 9.9 mi
	7.	Merge onto **Wantagh State Pkwy** About 2 mins	go 1.9 mi total 11.8 mi
↗	8.	Take exit **W5 W** for **NY-27 W** toward **New York**	go 0.2 mi total 12.1 mi
27	9.	Slight **right** at **NY-27 W/Sunrise Hwy** About 7 mins	go 6.2 mi total 18.2 mi
↰	10.	Turn **left** at **N Long Beach Rd** About 7 mins	go 2.6 mi total 20.9 mi
↗	11.	Slight **right** at **Daly Blvd** About 2 mins	go 0.6 mi total 21.5 mi
↱	12.	Turn **right** at **Hampton Rd**	go 0.2 mi total 21.7 mi

B Hampton Rd, Oceanside, NY 11572

Figure 2-3

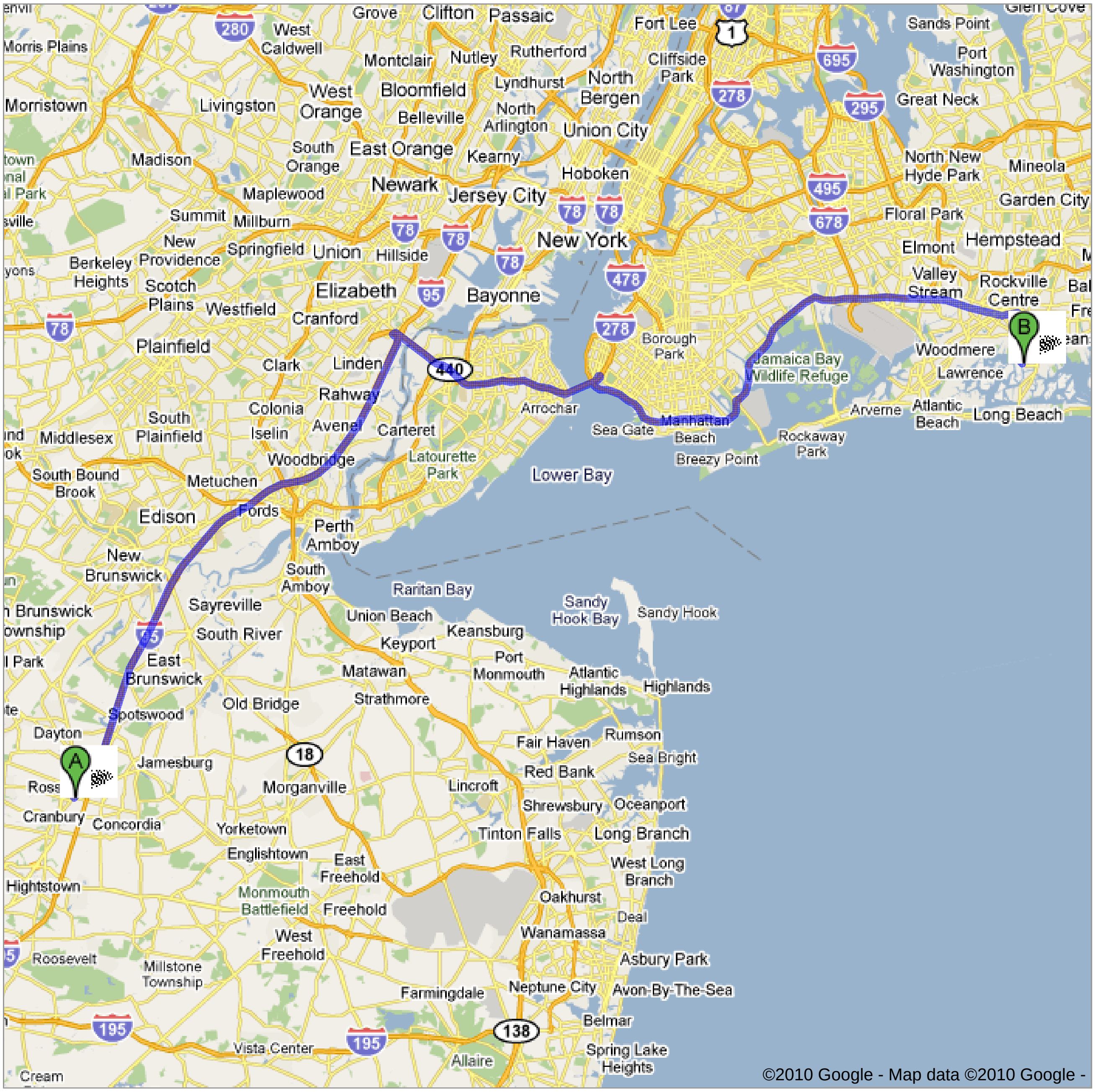
Driving Route – ARCADIS Cranbury, NJ to the Project Site

Google maps

Directions to Hampton Rd, Oceanside, NY 11572

66.9 mi – about **1 hour 23 mins** – up to 1 hour 50 mins in traffic

Directions from Cranbury Office



©2010 Google - Map data ©2010 Google -

A Cranbury South River Rd, Cranbury, NJ 08512

	1. Head **northeast** on **Co Rd 614/County Rd 614/Cranbury South River Rd** toward **County Rd 614/Prospect Plains Rd** Continue to follow Cranbury South River Rd About 3 mins	go 1.5 mi total 1.5 mi
32	2. Turn **right** at **NJ-32 E/Forsgate Dr**	go 135 ft total 1.5 mi
↗	3. Take the ramp to **I-95 N** Toll road About 1 min	go 0.6 mi total 2.1 mi
↰	4. Keep **left** at the fork to continue toward **I-95 N** Toll road About 1 min	go 0.5 mi total 2.6 mi
95	5. Keep **left** at the fork to continue toward **I-95 N** and merge onto **I-95 N** Toll road About 26 mins	go 25.0 mi total 27.6 mi
278	6. Take exit **13** to merge onto **I-278 E** toward **Goethals Bridge/Verrazano Bridge** Partial toll road Entering New York About 15 mins	go 11.6 mi total 39.3 mi
↖	7. Take the **Belt Pkwy E** exit on the **left** toward **Kennedy Airport** Toll road About 1 min	go 0.9 mi total 40.2 mi
	8. Merge onto **Belt Pkwy/Shore Pkwy E** About 19 mins	go 19.0 mi total 59.2 mi
27	9. Exit onto **NY-27 E/S Conduit Ave** Continue to follow NY-27 E About 9 mins	go 5.0 mi total 64.1 mi
↱	10. Turn **right** at **Ocean Ave** About 3 mins	go 1.2 mi total 65.3 mi
↰	11. Turn **left** at **Atlantic Ave** About 1 min	go 0.2 mi total 65.5 mi
↱	12. Take the 2nd **right** onto **Bayview Ave/Lawson Blvd** Continue to follow Lawson Blvd About 1 min	go 0.5 mi total 66.0 mi
↱	13. Take the 3rd **right** onto **Weidner Ave**	go 338 ft total 66.1 mi
↰	14. Turn **left** at **Royal Ave** About 1 min	go 0.4 mi total 66.5 mi
↗	15. Slight **right** at **Hampton Rd** About 1 min	go 0.4 mi total 66.9 mi

B Hampton Rd, Oceanside, NY 11572

ARCADIS

Appendix I

Investigative-Derived Waste SOP

ARCADIS
Infrastructure, environment, buildings

Imagine the result

Investigation-Derived Waste Handling and Storage

Rev. #: 2

Rev Date: March 6, 2009

Approval Signatures

Prepared by: Andrew Kamik Date: 3/6/09

Reviewed by: Liz Marsh Date: 3/6/09
(Technical Expert)

I. Scope and Application

The objective of this Standard Operating Procedure (SOP) is to describe the procedures to manage investigation-derived wastes (IDW), both hazardous and non-hazardous, generated during site activities, which may include, but are not limited to - drilling, trenching/excavation, construction, demolition, monitoring well sampling, soil sampling, decontamination and remediation. Please note that this SOP is intended for materials that have been deemed a solid waste as defined by 40 CFR § 261.2 (which may includes liquids, solids, and sludges). In some cases, field determinations will be made based on field screening or previous data that materials are not considered a solid waste. IDW may include soil, groundwater, drilling fluids, decontamination liquids, personal protective equipment (PPE), sorbent materials, construction and demolition debris, and disposable sampling materials that may have come in contact with potentially impacted materials. IDW will be collected and staged at the point of generation. Quantities small enough to be containerized in 55-gallon drums will be taken to a designated temporary storage area (discussed in further detail under Drum Storage) onsite pending characterization and disposal. Waste materials will be analyzed for constituents of concern to evaluate proper disposal methods. PPE and disposable sampling equipment will be placed in DOT-approved drums prior to disposal and typically does not require laboratory analysis. This SOP describes the necessary equipment, field procedures, materials, regulatory references, and documentation procedures necessary for proper handling and storage of IDW up to the time it is properly disposed. The procedures for handling IDW are based on the United States Environmental Protection Agency's Guide to Management of Investigation Derived Wastes (USEPA, 1992). IDW is assumed to be contaminated with the site constituents of concern (COCs) until analytical evidence indicates otherwise. IDW will be managed to ensure the protection of human health and the environment and will comply with all applicable or relevant and appropriate requirements (ARAR). The following Laws and Regulations on Hazardous Waste Management are potential ARAR for this site.

State Laws and Regulations

- To Be Determined Based on Location of Site and Location of Treatment, Storage, and/or Disposal Facility (TSDF) to be utilized

Federal Laws and Regulations

- Resource Conservation and Recovery Act (RCRA) 42 USC § 6901-6987
- Comprehensive Environmental Response, Compensation and Liability Act (CERCLA) 42 USC § 9601-9675

- Superfund Amendments and Reauthorization Act (SARA)
- Department of Transportation (DOT) Hazardous Materials Transportation

Pending characterization, IDW will be stored appropriately within each area of contamination (AOC). Under RCRA, "storage" is defined as the holding of hazardous waste for a temporary period, at the end of which the hazardous waste is treated, disposed of, or stored elsewhere" (40 CFR § 260.10). The onsite waste staging area will be in a secure and controlled area. Waste characterization can either be based on generator knowledge, such as using materials safety data sheets (MSDS'), or can be based upon analytical results. The laboratory used for waste characterization analysis must have the appropriate state and federal certifications and be approved by ARCADIS and Client. IDW will be classified as RCRA hazardous or non-regulated under RCRA based on the waste characterization.

If IDW is characterized as RCRA hazardous waste, RCRA and DOT requirements must be followed for packaging, labeling, transporting, storing, and record keeping as described in 40 CFR § 262 and 49 CFR § 171-178. Wastes judged to potentially meet the criteria for hazardous wastes shall be stored in DOT approved packaging. Waste material classified as RCRA non-hazardous may be handled and disposed of as an industrial waste.

Liquid wastes judged to potentially meet the criteria for hazardous wastes shall be stored in DOT approved 55 gallon drums or other approved containers that are compatible with the type of material stored therein. Solid materials deemed to potentially meet hazardous criteria will be drummed where practicable. Large quantities of potentially hazardous solid materials must be containerized (such as in a roll-off box) for up to a maximum of 90 or 180 days as described in the Excavated Solids Section. Waste material classified as non-hazardous may be handled and disposed of as an industrial waste and is not subject to the 90-day or 180-day on-site storage limitation.

This is a standard (i.e., typically applicable) operating procedure which may be varied or changed as required, dependent upon site conditions, equipment limitations, or limitations imposed by the procedure. The ultimate procedure employed will be documented in the project work plans or reports. If changes to the sampling procedures are required due to unanticipated field conditions, the changes will be discussed with the Project Manager and Client as soon as practicable and documented in the report.

II. Personnel Qualifications

ARCADIS field sampling personnel will have current health and safety training including 40-hour HAZWOPER training, site supervisor training, site-specific training, first aid, and CPR, as needed. ARCADIS personnel may sign manifests on a case-to-case basis for clients, provided the appropriate agreement is in place between ARCADIS and the client documenting that ARCADIS is not the generator, but is acting as authorized representative for the generator. ARCADIS personnel who sign hazardous waste manifests will have the current DOT hazardous materials transportation training according to 49 CFR § 172.704. ARCADIS field personnel will also comply with client-specific training such as LPS. In addition, ARCADIS field sampling personnel will be versed in the relevant SOPs and posses the required skills and experience necessary to successfully complete the desired field work.

III. Equipment List

The following materials, as required, shall be available for IDW handling and storage:

Appropriate personal protective equipment as specified in the Site Health and Safety Plan

- 55-gallon steel drums, DOT 1A2 or equivalent
- ¾ -inch socket wrench
- Hammer
- Leather gloves
- Drum dolly
- Appropriate drum labels (outdoor waterproof self adhesive)
- Polyethylene storage tank
- Appropriate labeling, packing, chain-of-custody forms, and shipping materials as specified in the *Chain-of-Custody* SOP and *Field Sampling Handling, Packing, and Shipping* SOP.
- Indelible ink and/or permanent marking pens
- Plastic sheeting

- Appropriate sample containers, labels, and forms
- Stainless-steel bucket auger
- Stainless steel spatula or knife
- Stainless steel hand spade
- Stainless steel scoop
- Digital camera
- Field logbook.

IV. Cautions

- Filled drums can be very heavy, always use appropriate moving techniques and equipment.
- Similar media will be stored in the same drums to aid in sample analysis and disposal.
- Drum lids must be secured to prevent rainwater from entering the drums.
- Drums containing solid material may not contain any free liquids.
- Waste containers stored for extended periods of time may be subject to deterioration. Drum over packs may be used as secondary containment.
- All drums must be in good condition to prevent potential leakage and facilitate subsequent disposal. Inspect the drums for dents and rust, and verify the drum has a secure lid prior to use.

V. Health and Safety Considerations

- Appropriate personal protective equipment must be worn by all field personnel within the designated work area.
- Air monitoring may be required during certain field activities as required in the Site Health and Safety Plan.

- If excavating in potentially hazardous areas is possible, contingency plans should be developed to address the potential for encountering gross contamination or non-aqueous phase liquids.

- ARCADIS field personnel will be familiar and compliant with Client-specific health and safety requirements such as Chevron's hand safety policy including the prohibition of fixed and/or folding blade knives.

VI. Procedure

Waste storage and handling procedures to be used depend upon the type of generated waste. For this reason, IDW should be stored in a secure location onsite in separate 55-gallon storage drums, solids can be stockpiled onsite (if non-hazardous), and purge water may be stored in polyethylene tanks. Waste materials such as broken sample bottles or equipment containers and wrappings will be stored in 55-gallon drums unless they were not in contact with sample media.

Management of IDW

Minimization of IDW should be considered by the Project Manager during all phases of the project. Site managers may want to consider techniques such as replacing solvent-based cleaners with aqueous-based cleaners for decontamination of equipment, reuse of equipment (where it can be decontaminated), limitation of traffic between exclusion and support zones, and drilling methods and sampling techniques that generate little waste. Alternative drilling and subsurface sampling methods may include the use of small diameter boreholes, as well as borehole testing methods such as a core penetrometer or direct push technique instead of coring (EPA, 1993).

Drum Storage

Drums containing hazardous waste shall be stored in accordance with the requirements of 40 CFR 265 Subpart I (for containers) and 265 Subpart DD (for containment buildings). All 55-gallon drums will be stored at a secure, centralized on-site location that is readily accessible for vehicular pick-up. Drums confirmed as, or believed to contain hazardous waste will be stored over an impervious surface provided with secondary containment. The storage location will, for drums containing liquid, have a containment system that can contain at least the larger of 10% of the aggregate volume of staged materials or 100% of the volume of the largest container. Drums will be closed during storage and be in good condition in accordance with the Guide to Management of Investigation-Derived Wastes (USEPA, 1992).

Hazardous Waste Determination

Waste material must be characterized to determine if it meets any of the federal definitions of hazardous waste as required by 40 CFR § 262.11. If the waste does not meet any of the federal definitions, it must then be established if any state-specific hazardous waste criteria exist/apply.

Generator Status

Once hazardous waste determination has been made, the generator status will be determined. Large quantity generators (LQG) are generators who generate more than 1,000 kilograms of hazardous waste in a calendar month. Small quantity generators (SQG) of hazardous waste are generators who generate greater than 100 kilograms but less than 1,000 kilograms of hazardous waste in a calendar month. Conditionally exempt small quantity generators (CESQG) are generators who generate less than 100 kilograms of hazardous waste per month. Please note that a generator status may change from month to month and that a notice of this change is usually required by the generator's state agency.

Accumulation Time for Hazardous Waste

A LQG may accumulate hazardous waste on site for 90 days or less without a permit and without having interim status provided that such accumulation is in compliance with specifications in 40 CFR § 262.34. A SQG may accumulate hazardous waste on site for 180 days or less without a permit or without having interim status subject to the requirements of 40 CFR § 262.34(d). CESQG requirements are found in 40 CFR § 261.5. **NOTE**: The CESQG and SQG provisions of 40 CFR § 261.5, 262.20(e), 262.42(b) and 262.44 may not be recognized by some states (e.g. Rhode Island). **State-specific regulations must be reviewed and understood prior to the generation of hazardous waste.**

<u>Satellite Accumulation of Hazardous Waste</u>

Satellite accumulation (SAA) shall mean the accumulation of as much as fifty-five (55) gallons of hazardous waste, or the accumulation of as much as one quart of acutely hazardous waste, in containers at or near any point of generation where the waste initially accumulates, which is under the control of the operator of the process generating the waste, without a permit or interim status and without complying with the requirements of 40 CFR § 262.34(a) and without any storage time limit, provided that the generator complies with 40 CFR § 262.34(c)(1)(i).

Once more than 55 gallons of hazardous waste accumulates in SAA, the generator has three days to move this waste into storage.

Storage recommendations for hazardous waste include:

- Ignitable Hazardous wastes must be >50 feet from the property line per 40 CFR § 265.176 (LQG generators only).
- Hazardous waste must be stored on a concrete slab (asphalt is acceptable if there are no free liquids in the waste) per 40 CFR § 265.176.
- Drainage must be directed away from the accumulation area.
- Area must be properly vented.
- Area must be secure.

Drum/Container Labeling

Drums will be labeled on both the side and lid of the drum using a permanent marking pen. Old drum labels must be removed to the extent possible, descriptions crossed out should any information remain, and new labels affixed on top of the old labels. Other containers used to store various types of waste (polyethylene tanks, roll-off boxes, end-dump trailers, etc.) will be labeled with an appropriate "Waste Container" or “Testing in Progress” label pending characterization. Drums and containers will be labeled as follows:

- Appropriate waste characterization label (Testing In Progress, Hazardous, or Non-Hazardous)
- Waste generator's name (e.g., client name)
- Project name
- Name and telephone number of ARCADIS project manager
- Composition of contents (e.g., used oil, acetone 40%, toluene 60%)
- Media (e.g., solid, liquid)
- Accumulation start date

- Drum number of total drums as reconciled with the Drum Inventory maintained in the field log book.

IDW containers will remain closed except when adding or removing waste. Immediately upon beginning to place waste into the drum/container, a “Waste Container” or “Testing in Progress” label will be filled out to include the information specified above, and affixed to the container. Once the contents of the container are identified as either non-hazardous or hazardous, the following additional labels will be applied. Containers with waste determined to be non-hazardous will be labeled with a green and white "Non-Hazardous Waste" label over the "Waste Container" label. Containers with waste determined to be hazardous will be stored in an onsite storage area and will be labeled with the "Hazardous Waste" label and affixed over the "Waste Container" label. The ACCUMULATION DATE for the hazardous waste is the date the waste is first placed in the container and is the same date as the date on the "Waste Container" label. DOT hazardous class labels must be applied to all hazardous waste containers for shipment offsite to an approved disposal or recycling facility. In addition a DOT proper shipping name shall be included on the hazardous waste label. The transporter should be equipped with the appropriate DOT placards. However, placarding or offering placards to the initial transporter is the responsibility of the generator per 40 CFR § 262.33.

Inspections and Documentation

All IDW will be documented as generated on a Drum Inventory Log maintained in the field log book. The Drum Inventory will record the generation date, type, quantity, matrix and origin (e.g. Boring-1, Test Pit 3, etc) of materials in every drum, as well as a unique identification number for each drum. The drum inventory will be used during drum pickup to assist with labeling of drums. The drum storage area and any other areas of temporarily staged waste, such as soil/debris piles, will be inspected weekly. The weekly inspections will be recorded in the field notebook or on a Weekly Inspection Log. Digital photographs will be taken upon the initial generation and drumming/staging of waste, and final labeling after characterization to document compliance with labeling and storage protocols, and condition of the container. Evidence of damage, tampering or other discrepancy should be documented photographically.

Emergency Response and Notifications

Specific procedures for responding to site emergencies will be detailed in the HASP. If the generator is designated as a LQG, a Contingency Plan will need to be prepared to include emergency response and notification procedures per 40 CFR § 265 Subpart D. In the event of a fire, explosion, or other release which could threaten human health outside of the site or when Client or ARCADIS has knowledge of a spill that has reached surface water, Client or ARCADIS must immediately notify the National Response Center (800-424-8802) in accordance with 40 CFR § 262.34. Other notifications to state agencies may also be necessary.

Drilling Soil Cuttings and Muds

Soil cuttings are solid to semi-solid soils generated during trenching activities, subsurface soil sampling, or installation of monitoring wells. Depending on the drilling method, drilling fluids known as "muds" may be used to remove soil cuttings. Drilling fluids flushed from the borehole must be directed into a settling section of a mud pit. This allows reuse of the decanted fluids after removal of the settled sediments. Soil cuttings will be labeled and stored in 55-gallon drums with bolt-sealed lids.

Excavated Solids

Excavated solids may include, but are not limited to soil, fill and construction and demolition debris. Excavated solids may be temporarily stockpiled onsite as long as the material is a RCRA non-hazardous waste and the solids will be treated onsite pursuant to a certified, authorized, or permitted treatment method, or properly disposed off-site. Stockpiled materials characterized as hazardous must be immediately containerized and removed from the site within 90 days of generation (except for soils using satellite accumulation). Excavated solids should be stockpiled and maintained in a secure area onsite. At a minimum, the floor of the stockpile area will be covered with a 20-mil high density polyethylene liner that is supported by a foundation or at least a 60-mil high density polyethylene liner that is not supported by a foundation. The excavated material will not contain free liquids. The owner/operator will provide controls for windblown dispersion, run-on control, and precipitation runoff. The run-on control system will prevent flow onto the active portion of the pile during peak discharge from at least a 25-year storm and the run-off management system will collect and control at least the water volume resulting from a 24-hour, 25-year storm (EPA, 1992). Additionally, the stockpile area will be inspected on a weekly basis and after storm events. Individual states may require that the stockpile be inspected/certified by a licensed professional engineer. Stockpiled material will be covered with a 6-mil polyvinyl chloride (PVC) liner. The stockpile cover will be secured in place with appropriate material (concrete blocks, weights, etc.) to prevent the movement of the cover. Excavated solids may also be placed in roll off containers and covered with a 6-mil PVC liner pending results for waste characterization.

Decontamination Solutions

Decontamination solutions are generated during the decontamination of personal protective equipment and sampling equipment. Decontamination solutions may range from detergents, organic solvents and acids used to decontaminate small field sampling equipment to steam cleaning rinsate used to wash heavy field equipment. These solutions are to be labeled and stored in 55-gallon drums with bolt-sealed lids.

Disposable Equipment

Disposable equipment includes personal protective equipment (tyvek coveralls, gloves, booties and APR cartridges) and disposable sampling equipment such as trowels or disposable bailers. If the media sampled exhibits hazardous characteristics per results of waste characterization sampling, disposable equipment will also be disposed of as a hazardous waste. These materials will be stored onsite in labeled 55-gallon drums pending analytical results for waste characterization.

Purge Water

Purge water includes groundwater generated during well development, groundwater sampling, or aquifer testing. The volume of groundwater generated will dictate the appropriate storage procedure. Monitoring well development and groundwater sampling may generate three well volumes of groundwater or more. This volume will be stored in labeled 55-gallon drums. Aquifer tests may generate significantly greater volumes of groundwater depending on the well yield and the duration of the test. Therefore, large-volume portable polyethylene tanks will be considered for temporary storage pending groundwater-waste characterization.

Purged Water Storage Tank Decontamination and Removal

The following procedures will be used for inspection, cleaning, and offsite removal of storage tanks used for temporary storage of purge water. These procedures are intended to be used for rented portable tanks such as Baker Tanks or Rain for Rent containers. Storage tanks will be made of inert polyethylene materials.

The major steps for preparing a rented tank for return to a vendor include characterizing the purge water, disposing of the purge water, decontaminating the tank, final tank inspection, and mobilization. Decontamination and inspection procedures are describe in further detail below.

- Tank Cleaning: Most vendors require that tanks be free of any sediment and water before returning, a professional cleaning service may be required. Each specific vendor should be consulted concerning specific requirements for returning tanks.

- Tank Inspection: After emptying the tank, purged water storage tanks should be inspected for debris, chemical staining, and physical damage. The vendors require that tanks be returned in the original condition (i.e., free of sediment, staining and no physical damage).

VII. Waste Characterization Sampling and Shipping

Soil/Solids Characterization

Waste characterization will be conducted in accordance with waste hauler, waste handling facility, and state/federal requirements. In general, RCRA hazardous wastes are those solid wastes determined by a Toxicity Characteristic Leaching Procedure (TCLP) test or to contain levels of certain toxic metals, pesticides, or other organic chemicals above specific federally regulated thresholds. If the one or more of 40 toxic compounds listed in Table I of 40 CFR § 261.24 are detected in the sample at levels above the maximum unregulated concentrations, the waste must be characterized as a toxic hazardous waste. Wastes can also be considered “listed” hazardous waste depending on site-specific processes.

Composite soil samples will be collected at a frequency of one sample per 10 cubic yard basis for stockpiled soil or one per 55-gallon drum for containerized. A four point composite sample will be collected per 10 cubic yards of stockpiled material and for each drum. Sample and composite frequencies may be adjusted in accordance with the waste handling facility’s requirements. Waste characterization samples may be analyzed for the TCLP volatile organic compounds (VOCs), TCLP semi-volatile organic compounds (SVOCs), TCLP RCRA metals, and polychlorinated biphenyls, as well as corrosivity (pH), reactivity and flammability (flashpoint). Additional samples may be collected and analyzed by the laboratory on a contingency basis.

Wastewater Characterization

Waste characterization will be conducted in accordance with the requirements of the waste hauler, waste handling facility, and state/federal governments. In general, purge water should be analyzed by methods appropriate for the known contaminants, if any, that have been historically detected in the monitoring wells. Samples will be collected and analyzed in accordance with the requirements of the waste disposal facility.

Wastewater characterization samples may be analyzed for TCLP volatile organic compounds (VOCs), TCLP semi-volatile organic compounds (SVOCs), TCLP RCRA metals, and polychlorinated biphenyls, as well as corrosivity (pH), reactivity and flammability (flashpoint). Additional samples may be collected and analyzed by the laboratory on a contingency basis.

Sample Handling and Shipping

All samples will be appropriately labeled, packed, and shipped, and the chain-of-custody will be filled out in accordance with the Chain-of-Custody SOP and Field Sampling Handling, Packing, and Shipping SOP and Hazardous Materials Packaging and Shipping SOP.

It should be noted that additional training is required for packaging and shipping of hazardous and/or dangerous materials. Please reference the following ARCADIS intranet team page for more information: http://team/sites/hazmat/default.aspx.

Preparing Waste Shipment Documentation (Hazardous and Non-Hazardous)

Waste profiles will be prepared by the ARCADIS PM and forwarded, along with laboratory analytical data to the Client PM for approval/signature. The Client PM will then return the profile to ARCADIS who will then forward to the waste removal contractor for preparation of a manifest. The manifest will be reviewed by ARCADIS prior to forwarding to the Client PM for approval. Upon approval of the manifest, the Client PM will return the original signed manifest directly to the waste contractor or to the ARCADIS PM for forwarding to the waste contractor.

Final drum labeling and pickup will be supervised by an ARCADIS representative who is experienced with waste labeling procedures. The ARCADIS representative will have a copy of the drum inventory maintained in the field book and will reconcile the drum inventory with the profile numbers on the labels and on the manifest. Different profile numbers will be generated for different matrices or materials in the drums. For example, the profile number for drill cuttings will be different than the profile number for purge water. **When there are multiple profiles it is critical that the proper label, with the profile number appropriate to a specific material be affixed to the proper drums.** A copy of the ARCADIS drum inventory will be provided to the waste transporter during drum pickup and to the facility receiving the waste.

VIII. Data Recording and Management

Waste characterization sample handling, packing, and shipping procedures will be documented in accordance with the *Quality Assurance Project Plan*, if one exists. Copies of the chains-of-custody forms will be maintained in the project file.

Following waste characterization, IDW containers will be re-labeled with the appropriate waste hazardous or non-hazardous waste labels and the client will initiate disposal at the appropriate waste disposal facility.

IX. Quality Assurance

The chain-of-custody and sample labels for waste characterization samples will be filled out in accordance with the *Quality Assurance Project Plan*.

X. References

United States Environmental Protection Agency (USEPA). 1992. Guide to Management of Investigation-Derived Wastes. Office of Remedial and Emergency Response. Hazardous Site Control Division. January 1992.

USEPA. 1991. *Guide to Discharging CERCLA Aqueous Wastes to Publicly Owned Treatment Works (POTWs)*. Office of Remedial and Emergency Response. Hazardous Site Control Division 0S-220W. March 1991.

ARCADIS

Appendix C

Preliminary Implementation Schedule

Preliminary Implementation Schedule

The overall schedule to implement the Remedial Investigation is anticipated to extend for a period of 18 weeks as summarized in the table below.

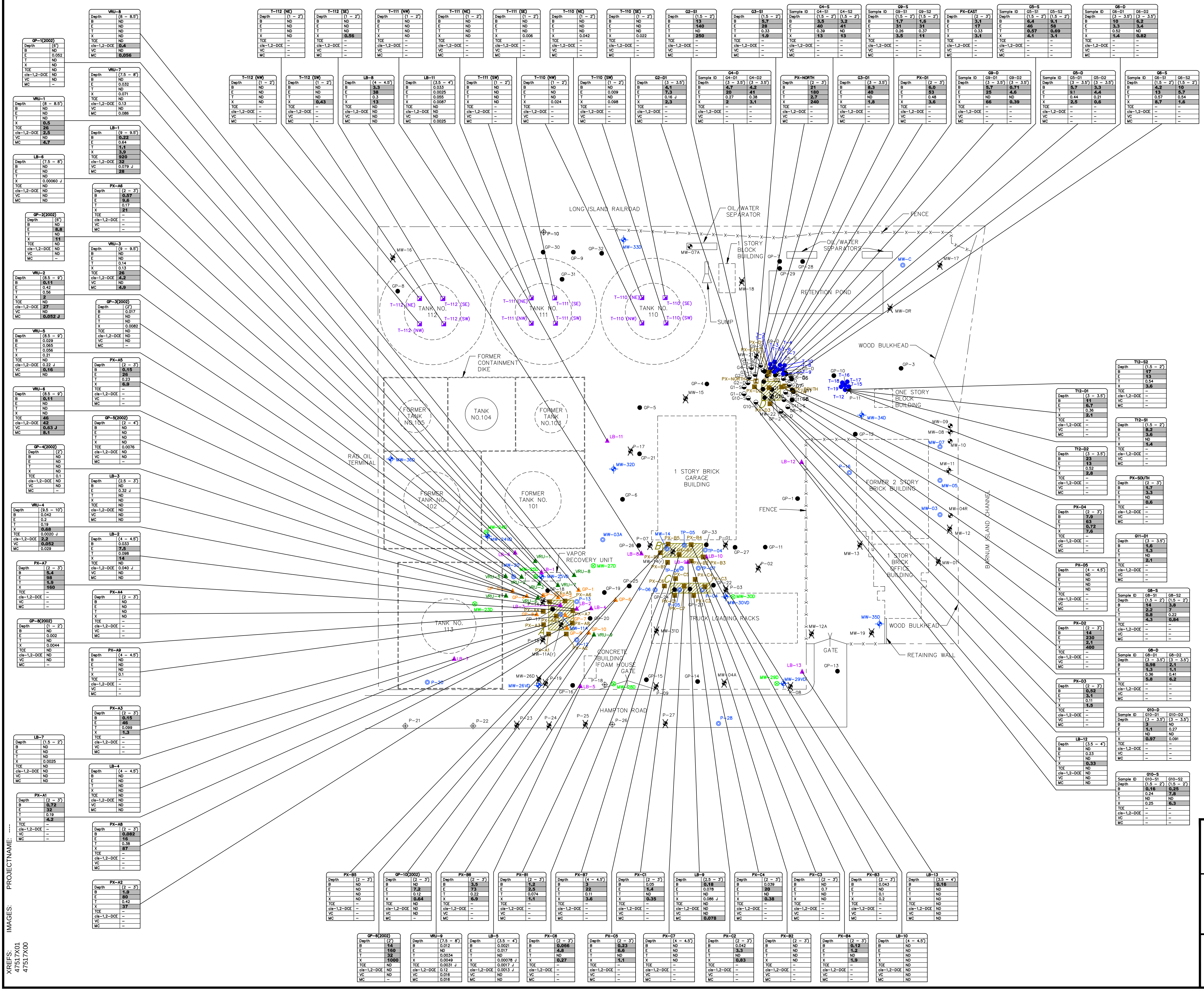
RI Task	**Duration (weeks)**
Mobilization of Field Investigation	2 Weeks
Implement Field Investigation	6 Weeks
Laboratory Testing	2 Weeks
Data Reduction & Analysis	4 Weeks
Remedial Investigation Reporting	4 Weeks

ARCADIS

Appendix D

Historical Soil and Groundwater Data

CITY: SYRACUSE DIV/GROUP: ENVCAD DB: G. STOWELL LD: G. STOWELL PIC: J. VOGELEY PM: W. McCUNE TM: W. McCUNE LYR: ON=*; OFF=*REF*
G:\ENVCAD\SYRACUSE\ACT\B0047517\0003\00001\DWG\DATABOXES\47517C01.dwg LAYOUT: 1 SAVED: 9/2/2010 4:03 PM ACADVER: 18.0S (LMS TECH) PAGESETUP: C-LD28-PDF-CONV PLOTSTYLETABLE: PLTFULL.CTB PLOTTED: 9/2/2010 4:04 PM BY: STOWELL, GARY
XREFS: 47517X01 47517X00 IMAGES: PROJECTNAME: ----



LEGEND:

- ⌖ TEMPORARY PIEZOMETER (INSTALLED BY LBG)
- ⌖ DEEP UNDERLYING SAND WELL
- ⌖ EXISTING MONITORING WELL
- ◎ ABANDONED MONITORING POINT
- ⊗ PAIRED WELL LOCATION
- ✕ DESTROYED MONITORING POINT (MARCH – AUGUST 2005 GULF SITE DEMOLITION)
- ● 2000 LBG GEOPROBE SOIL BORING LOCATION
- ▲ 2000 BBL SOIL BORING LOCATION
- ▲ 2002 BBL SOIL BORING LOCATION
- ■ 2002 BBL POST EXCAVATION BORING LOCATION
- ◪ 2003 LBG SOIL SAMPLING TANK LOCATION
- ▲ 2004 LANGAN SOIL BORING LOCATION
- ● 2004 CEDAR CREEK TEMPORARY WELL
- ◒ 2005 BBL BORING LOCATION
- ▣ 2005 BBL POST EXCAVATION BORING LOCATION
- 2005 BBL PAIRED WELL LOCATION
- ▨ EXCAVATION LOCATIONS (OCTOBER 2002)

ANALYTE	SHORT NAME	PART 375 UNRESTRICTED USE SCOs
Benzene	B	0.06
Ethylbenzene	E	1
Toluene	T	0.7
Xylenes (total)	X	0.26
Trichloroethene	TCE	0.47
cis-1,2-Dichloroethene	cis-1,2-DCE	0.25
Vinyl Chloride	VC	0.02
Methylene Chloride	MC	0.05

NOTES:

1. SITE PLAN DERIVED FROM STORB, INC. DRAWING ENTITLED "PAVING PLAN–GULF OIL COMPANY–U.S., OCEANSIDE TERMINAL" AUGUST 7, 1976 AND LANGAN DRAWING ENTITLED "HISTORICAL SOIL AND GROUNDWATER SAMPLING PLAN MAY 11, 2007.
2. MONITORING WELL LOCATIONS SURVEYED BY A. TAY.
3. LOCATIONS OF DEEP UNDERLYING SAND WELLS AND SOIL BORINGS ARE APPROXIMATE.
4. CONSTITUENT LIST IS BASED ON THE CONSTITUENTS OF CONCERNS IDENTIFIED IN THE REMEDIAL INVESTIGATION/FEASIBILITY STUDY WORK PLAN.
5. ALL CONCENTRATIONS ARE MILLIGRAMS PER KILOGRAM (mg/kg) AND DEPTH INTERVALS ARE IN FEET BELOW GROUND SURFACE.
6. SHADED VALUES EXCEED PART 375 UNRESTRICTED USE SOIL CLEANUP OBJECTIVES (SCOs).
7. ND = NOT DETECTED.
8. – = NOT ANALYZED.
9. J = INDICATES AN ESTIMATED VALUE.

GULF OIL TERMINAL
(CHEVRON FACILITY #6518040)
OCEANSIDE, NEW YORK

VOC CONCENTRATIONS SHALLOW SAND FILL - SOIL

ARCADIS

FIGURE
1

CITY: SYRACUSE DIV/GROUP: ENVCAD DB: G. STOWELL L. FORAKER LD: G. STOWELL PIC: J. VOGELEY PM: W. McCUNE TM: W. McCUNE LYR: ON=*; OFF=*REF*
G:\ENVCAD\SYRACUSE\ACT\B0047517\0003\00001\DWG\DATABOXES\47517C02.dwg LAYOUT: 2 SAVED: 9/2/2010 3:41 PM ACADVER: 18.0S (LMS TECH) PAGESETUP: C-LD2B-PDF-CONV PLOTSTYLETABLE: PLTFULL.CTB PLOTTED: 9/2/2010 4:05 PM BY: STOWELL, GARY
XREFS: 47517X01 47517X00 IMAGES: PROJECTNAME: ----

VRU-1

Depth	(9.5 – 10')	(14 – 14.5')
B	**1.6 J**	ND
E	ND	ND
T	ND	ND
X	**4.9 J**	ND
TCE	**1200**	**4.2**
cis–1,2–DCE	**87**	**1.1 J**
VC	ND	ND
MC	**380**	**120**

VRU-7

Depth	(12 – 12.5')	(14 – 14.5')
B	0.0093	ND
E	0.38	ND
T	0.005	ND
X	**0.7**	ND
TCE	ND	ND
cis–1,2–DCE	**0.44**	ND
VC	**1.4 E**	ND
MC	**0.14**	0.03

VRU-8

Depth	(10.5')	(14 – 14.5')
B	**0.083**	ND
E	0.012	ND
T	0.029	ND
X	0.044	ND
TCE	ND	ND
cis–1,2–DCE	ND	0.051
VC	**0.026 J**	ND
MC	0.039	**0.19**

LB-12

Depth	(11.5 – 12')
B	0.0013 J
E	ND
T	ND
X	ND
TCE	ND
cis–1,2–DCE	ND
VC	ND
MC	ND

VRU-2

Depth	(10 – 10.5')	(15 – 15.5')
B	**2.7 J**	ND
E	**6.1**	ND
T	**16**	ND
X	**37**	ND
TCE	**570**	ND
cis–1,2–DCE	**560**	ND
VC	**8.5 J**	ND
MC	**660**	**33**

VRU-5

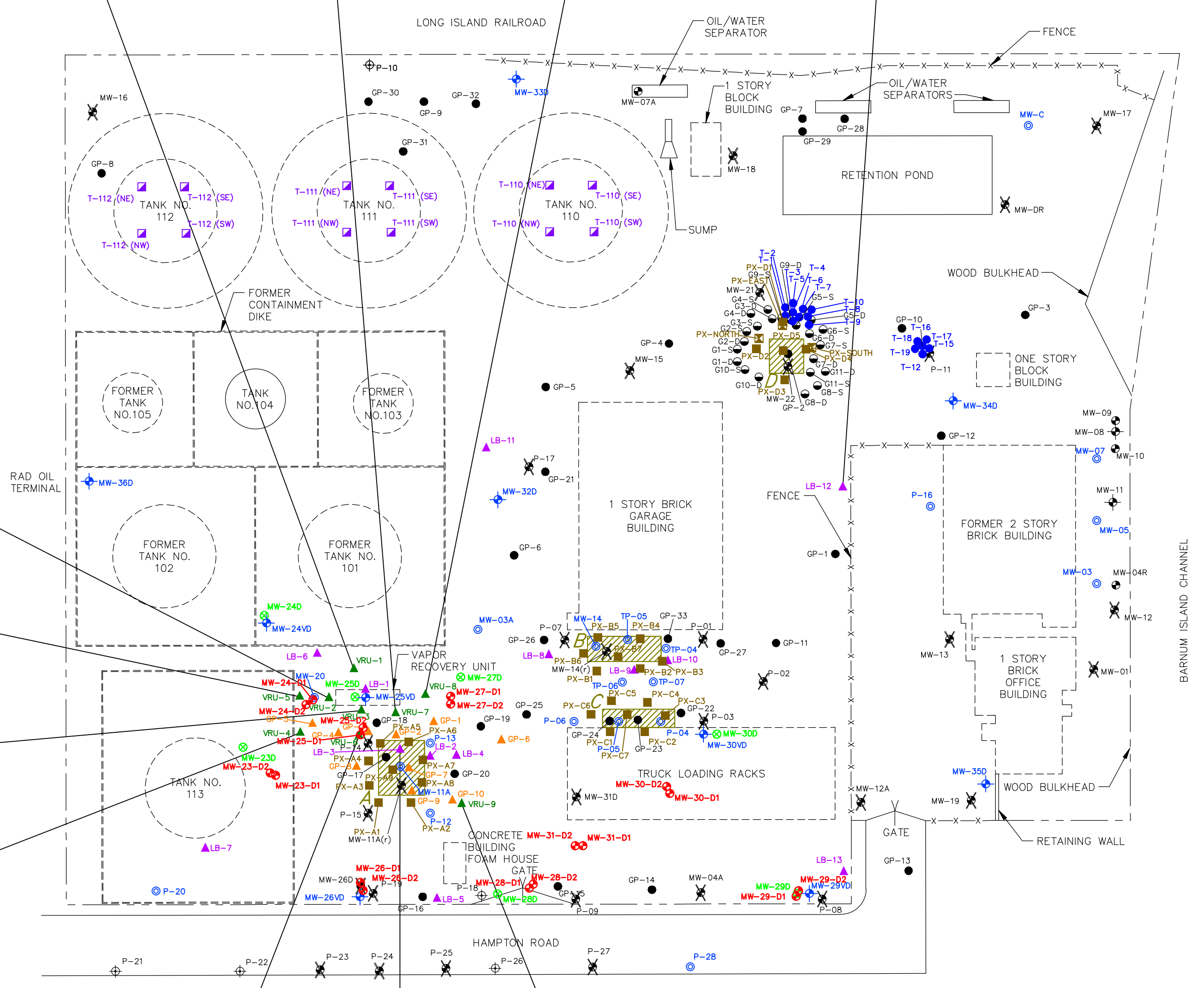
Depth	(10.5 – 11')	(15.5 – 16')
B	**0.065**	ND
E	0.013	ND
T	0.015	ND
X	0.042	ND
TCE	0.0031 J	ND
cis–1,2–DCE	0.019	ND
VC	**0.2**	ND
MC	**0.18**	ND

VRU-3

Depth	(10.5 – 11')	(14 – 14.5')
B	**0.63**	ND
E	0.75	ND
T	**2.4**	ND
X	**4.4**	ND
TCE	**160**	0.071
cis–1,2–DCE	**90**	0.034
VC	**0.89 J**	ND
MC	**190**	**18**

VRU-4

Depth	(10.5 – 11')	(17.5 – 18')
B	**0.76**	ND
E	0.21	ND
T	0.32	ND
X	**0.61**	ND
TCE	ND	ND
cis–1,2–DCE	0.052	0.0054 J
VC	**0.2**	ND
MC	0.0099	**0.24**



VRU-6

Depth	(9.5 – 10')	(14 – 14.5')
B	**1.7**	0.0086
E	ND	ND
T	ND	0.0046
X	**6.0 J**	0.0068
TCE	**150**	0.33 J
cis–1,2–DCE	**530**	**1.4**
VC	**22**	**0.27**
MC	**31**	**29**

LB-3

Depth	(13.5 – 14')
B	ND
E	ND
T	ND
X	ND
TCE	ND
cis–1,2–DCE	ND
VC	ND
MC	ND

VRU-9

Depth	(9.5 – 10')	(12.5 – 13')
B	0.04	**0.083**
E	ND	ND
T	ND	ND
X	ND	ND
TCE	0.0059 J	0.036
cis–1,2–DCE	0.13	**0.27**
VC	**0.42**	**0.21**
MC	**0.051**	**0.12**

LEGEND:

⌖	TEMPORARY PIEZOMETER (INSTALLED BY LBG)
⌖	DEEP UNDERLYING SAND WELL
⌖	EXISTING MONITORING WELL
◎	ABANDONED MONITORING POINT
⊗	PAIRED WELL LOCATION
⊗	DESTROYED MONITORING POINT (MARCH – AUGUST 2005 GULF SITE DEMOLITION)
●	2000 LBG GEOPROBE SOIL BORING LOCATION
▲	2000 BBL SOIL BORING LOCATION
▲	2002 BBL SOIL BORING LOCATION
■	2002 BBL POST EXCAVATION BORING LOCATION
◪	2003 LBG SOIL SAMPLING TANK LOCATION
▲	2004 LANGAN SOIL BORING LOCATION
●	2004 CEDAR CREEK TEMPORARY WELL
◒	2005 BBL BORING LOCATION
⊠	2005 BBL POST EXCAVATION BORING LOCATION
⊕	2005 BBL PAIRED WELL LOCATION
▨	EXCAVATION LOCATIONS (OCTOBER 2002)

ANALYTE	SHORT NAME	PART 375 UNRESTRICTED USE SCOs
Benzene	B	0.06
Ethylbenzene	E	1
Toluene	T	0.7
Xylenes (total)	X	0.26
Trichloroethene	TCE	0.47
cis–1,2–Dichloroethene	cis–1,2–DCE	0.25
Vinyl Chloride	VC	0.02
Methylene Chloride	MC	0.05

NOTES:

1. SITE PLAN DERIVED FROM STORB, INC. DRAWING ENTITLED "PAVING PLAN–GULF OIL COMPANY–U.S., OCEANSIDE TERMINAL" AUGUST 7, 1976 AND LANGAN DRAWING ENTITLED "HISTORICAL SOIL AND GROUNDWATER SAMPLING PLAN MAY 11, 2007.
2. MONITORING WELL LOCATIONS SURVEYED BY A. TAY.
3. LOCATIONS OF DEEP UNDERLYING SAND WELLS AND SOIL BORINGS ARE APPROXIMATE.
4. CONSTITUENT LIST IS BASED ON THE CONSTITUENTS OF CONCERNS IDENTIFIED IN THE REMEDIAL INVESTIGATION/FEASIBILITY STUDY WORK PLAN.
5. ALL CONCENTRATIONS ARE MILLIGRAMS PER KILOGRAM (mg/kg) AND DEPTH INTERVALS ARE IN FEET BELOW GROUND SURFACE.
6. SHADED VALUES EXCEED PART 375 UNRESTRICTED USE SOIL CLEANUP OBJECTIVES (SCOs).
7. ND = NOT DETECTED.
8. – = NOT ANALYZED.
9. J = INDICATES AN ESTIMATED VALUE.
10. E = ANALYTE EXCEEDED THE CALIBRATION RANGE.

0 60' 120'
GRAPHIC SCALE

GULF OIL TERMINAL
(CHEVRON FACILITY #6518040)
OCEANSIDE, NEW YORK

VOC CONCENTRATIONS
MEADOW MAT - SOIL

ARCADIS

FIGURE
2

LB-6	
Depth	(18 – 18.5')
B	0.012
E	0.011
T	0.028
X	0.035
TCE	**2.9**
cis–1,2–DCE	**0.57**
VC	0.0068
MC	0.024

LB-11	
Depth	(24 – 24.5')
B	ND
E	ND
T	ND
X	ND
TCE	ND
cis–1,2–DCE	ND
VC	ND
MC	ND

MW-33D		
Depth	(19 – 19.5')	(23 – 23.5')
B	ND	ND
E	ND	ND
T	ND	ND
X	ND	ND
TCE	ND	ND
cis–1,2–DCE	ND	ND
VC	ND	ND
MC	ND	ND

MW-32D		
Depth	(27 – 27.5')	(31 – 31.5')
B	ND	ND
E	ND	ND
T	ND	ND
X	ND	ND
TCE	ND	ND
cis–1,2–DCE	ND	ND
VC	ND	ND
MC	ND	ND

LB-8	
Depth	(22 – 22.5')
B	ND [ND]
E	ND [ND]
T	ND [ND]
X	ND [ND]
TCE	ND [ND]
cis–1,2–DCE	ND [ND]
VC	ND [ND]
MC	ND [ND]

LB-12	
Depth	(21 – 21.5')
B	ND
E	ND
T	ND
X	ND
TCE	ND
cis–1,2–DCE	ND
VC	ND
MC	ND

MW-36D		
Depth	(23 – 23.5')	(31 – 31.5')
B	ND	ND
E	ND	ND
T	ND	ND
X	ND	ND
TCE	ND	ND
cis–1,2–DCE	ND	ND
VC	ND	ND
MC	0.012	ND

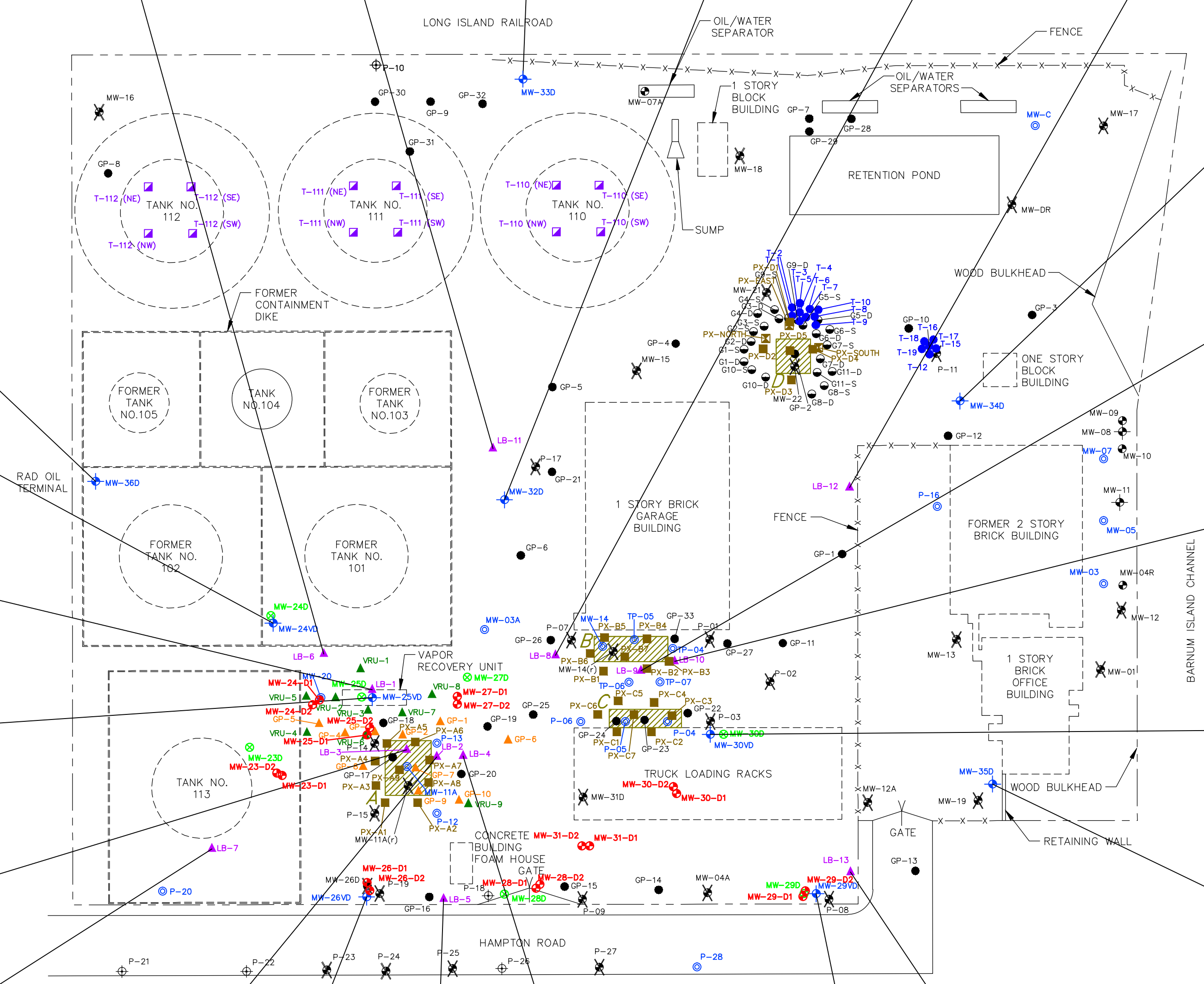
MW-24VD			
Depth	(19 – 19.5')	(68 – 68.5')	(73 – 73.5')
B	ND	ND [ND]	ND
E	ND	ND [ND]	ND
T	ND	ND [ND]	ND
X	ND	ND [ND]	ND
TCE	0.0030 J	ND [ND]	ND
cis–1,2–DCE	ND	ND [ND]	ND
VC	ND	ND [ND]	ND
MC	0.019	ND [ND]	ND

LB-1	
Depth	(17.5 – 18')
B	0.0013
E	0.0011
T	0.002
X	0.00072 J
TCE	**1.3**
cis–1,2–DCE	**0.26 J**
VC	0.01
MC	0.045

MW-25VD		
Depth	(18 – 18.5')	(68 – 68.5')
B	ND	ND
E	ND	ND
T	ND	ND
X	ND	ND
TCE	0.011	ND
cis–1,2–DCE	0.015	ND
VC	ND	ND
MC	0.031	ND

LB-3	
Depth	(18.5 – 19')
B	ND
E	ND
T	ND
X	ND
TCE	0.0075 J
cis–1,2–DCE	0.043
VC	ND
MC	ND

LB-7	
Depth	(14 – 14.5')
B	0.0041
E	ND
T	ND
X	ND
TCE	ND
cis–1,2–DCE	ND
VC	ND
MC	ND



MW-34D		
Depth	(25 – 25.5')	(29 – 29.5')
B	ND	ND
E	ND	ND
T	ND	ND
X	ND	ND
TCE	ND	ND
cis–1,2–DCE	ND	ND
VC	ND	ND
MC	0.0029	0.023

LB-9	
Depth	(19.5 – 20')
B	ND
E	ND
T	ND
X	ND
TCE	ND
cis–1,2–DCE	ND
VC	ND
MC	ND

LB-10	
Depth	(24 – 24.5')
B	ND
E	ND
T	ND
X	ND
TCE	ND
cis–1,2–DCE	ND
VC	ND
MC	ND

MW-30VD		
Depth	(28 – 30')	(68 – 70')
B	ND	0.0046
E	ND	ND
T	ND	ND
X	ND	ND
TCE	ND	ND
cis–1,2–DCE	ND	ND
VC	ND	ND
MC	0.0083	**0.12**

MW-35D		
Depth	(15 – 15.5')	(20 – 20.5')
B	ND	ND
E	ND	ND
T	ND	ND
X	ND	ND
TCE	ND	ND
cis–1,2–DCE	ND	ND
VC	ND	ND
MC	0.0049	0.0051

LB-2	
Depth	(16.5 – 17')
B	0.00071
E	0.00087 J
T	ND
X	0.0016
TCE	0.091
cis–1,2–DCE	0.098
VC	0.0085
MC	ND

MW-26VD		
Depth	(19 – 21')	(58 – 60')
B	ND	ND
E	ND	ND
T	ND	ND
X	ND	ND
TCE	ND	ND
cis–1,2–DCE	ND	ND
VC	ND	ND
MC	0.044	ND

LB-5	
Depth	(16.5 – 17')
B	ND
E	ND
T	ND
X	ND
TCE	0.0026 J
cis–1,2–DCE	0.0011 J
VC	ND
MC	ND

LB-4	
Depth	(17.5 – 18')
B	ND
E	ND
T	ND
X	ND
TCE	0.0027 J
cis–1,2–DCE	0.24 J
VC	0.0021 J
MC	ND

MW-29VD			
Depth	(15 – 17')	(19 – 21')	(58 – 60')
B	ND	ND	ND
E	ND	ND	ND
T	ND	ND	ND
X	ND	ND	ND
TCE	ND	ND	ND
cis–1,2–DCE	ND	ND	ND
VC	ND	ND	ND
MC	**0.064 J**	**0.11**	ND

LB-13	
Depth	(17.5 – 18')
B	0.014
E	ND
T	ND
X	0.0016 J
TCE	ND
cis–1,2–DCE	ND
VC	ND
MC	ND

LEGEND:

⌖	TEMPORARY PIEZOMETER (INSTALLED BY LBG)
⌖	DEEP UNDERLYING SAND WELL
⌖	EXISTING MONITORING WELL
◎	ABANDONED MONITORING POINT
⊗	PAIRED WELL LOCATION
✗	DESTROYED MONITORING POINT (MARCH – AUGUST 2005 GULF SITE DEMOLITION)
●	2000 LBG GEOPROBE SOIL BORING LOCATION
▲	2000 BBL SOIL BORING LOCATION
▲	2002 BBL SOIL BORING LOCATION
■	2002 BBL POST EXCAVATION BORING LOCATION
◪	2003 LBG SOIL SAMPLING TANK LOCATION
▲	2004 LANGAN SOIL BORING LOCATION
●	2004 CEDAR CREEK TEMPORARY WELL
◒	2005 BBL BORING LOCATION
■	2005 BBL POST EXCAVATION BORING LOCATION
⊗	2005 BBL PAIRED WELL LOCATION
▨	EXCAVATION LOCATIONS (OCTOBER 2002)

ANALYTE	SHORT NAME	PART 375 UNRESTRICTED USE SCOs
Benzene	B	0.06
Ethylbenzene	E	1
Toluene	T	0.7
Xylenes (total)	X	0.26
Trichloroethene	TCE	0.47
cis–1,2–Dichloroethene	cis–1,2–DCE	0.25
Vinyl Chloride	VC	0.02
Methylene Chloride	MC	0.05

NOTES:

1. SITE PLAN DERIVED FROM STORB, INC. DRAWING ENTITLED "PAVING PLAN–GULF OIL COMPANY–U.S., OCEANSIDE TERMINAL" AUGUST 7, 1976 AND LANGAN DRAWING ENTITLED "HISTORICAL SOIL AND GROUNDWATER SAMPLING PLAN MAY 11, 2007.
2. MONITORING WELL LOCATIONS SURVEYED BY A. TAY.
3. LOCATIONS OF DEEP UNDERLYING SAND WELLS AND SOIL BORINGS ARE APPROXIMATE.
4. CONSTITUENT LIST IS BASED ON THE CONSTITUENTS OF CONCERNS IDENTIFIED IN THE REMEDIAL INVESTIGATION/FEASIBILITY STUDY WORK PLAN.
5. ALL CONCENTRATIONS ARE MILLIGRAMS PER KILOGRAM (mg/kg) AND DEPTH INTERVALS ARE IN FEET BELOW GROUND SURFACE.
6. SHADED VALUES EXCEED PART 375 UNRESTRICTED USE SOIL CLEANUP OBJECTIVES (SCOs).
7. ND = NOT DETECTED.
8. – – = NOT ANALYZED.
9. J = INDICATES AN ESTIMATED VALUE.
10. DUPLICATE RESULTS ARE PROVIDED IN BRACKETS.

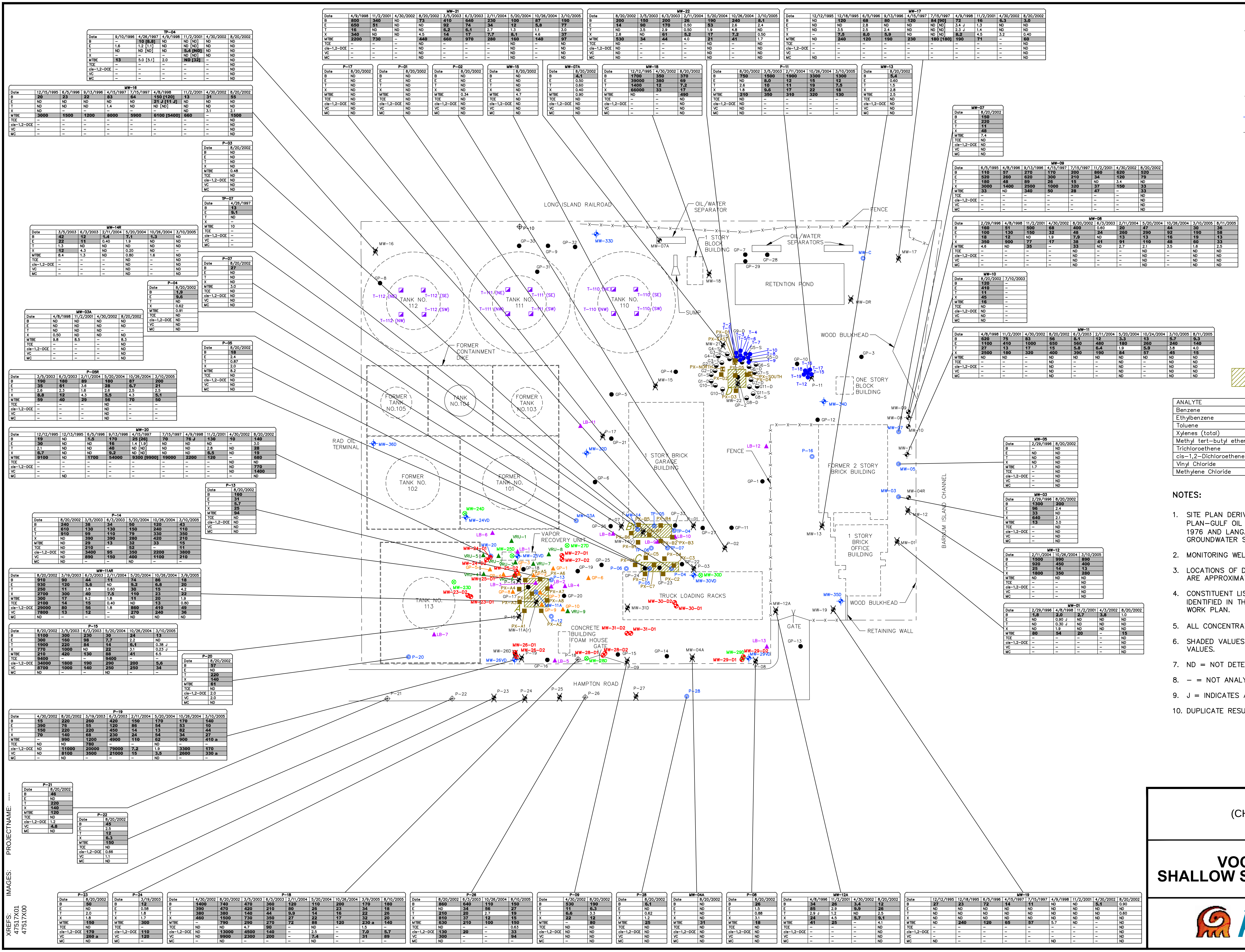
0 60' 120'
GRAPHIC SCALE

GULF OIL TERMINAL
(CHEVRON FACILITY #6518040)
OCEANSIDE, NEW YORK

VOC CONCENTRATIONS
DEEP SAND UNIT - SOIL

ARCADIS

FIGURE
3



LEGEND:

⌖	TEMPORARY PIEZOMETER (INSTALLED BY LBG)
⌖	DEEP UNDERLYING SAND WELL
⌖	EXISTING MONITORING WELL
○	ABANDONED MONITORING POINT
⊗	PAIRED WELL LOCATION
✕	DESTROYED MONITORING POINT (MARCH – AUGUST 2005 GULF SITE DEMOLITION)
●	2000 LBG GEOPROBE SOIL BORING LOCATION
▲	2000 BBL SOIL BORING LOCATION
▲	2002 BBL SOIL BORING LOCATION
■	2002 BBL POST EXCAVATION BORING LOCATION
◪	2003 LBG SOIL SAMPLING TANK LOCATION
▲	2004 LANGAN SOIL BORING LOCATION
●	2004 CEDAR CREEK TEMPORARY WELL
◒	2005 BBL BORING LOCATION
▣	2005 BBL POST EXCAVATION BORING LOCATION
⊘	2005 BBL PAIRED WELL LOCATION
▨	EXCAVATION LOCATIONS (OCTOBER 2002)

ANALYTE	SHORT NAME	NYSDEC TOGS 1.1.1 WATER GUIDANCE VALUES
Benzene	B	1
Ethylbenzene	E	5
Toluene	T	5
Xylenes (total)	X	5
Methyl tert-butyl ether	MTBE	10
Trichloroethene	TCE	5
cis-1,2-Dichloroethene	cis-1,2-DCE	5
Vinyl Chloride	VC	2
Methylene Chloride	MC	5

NOTES:

1. SITE PLAN DERIVED FROM STORB, INC. DRAWING ENTITLED "PAVING PLAN–GULF OIL COMPANY–U.S., OCEANSIDE TERMINAL" AUGUST 7, 1976 AND LANGAN DRAWING ENTITLED "HISTORICAL SOIL AND GROUNDWATER SAMPLING PLAN MAY 11, 2007.
2. MONITORING WELL LOCATIONS SURVEYED BY A. TAY.
3. LOCATIONS OF DEEP UNDERLYING SAND WELLS AND SOIL BORINGS ARE APPROXIMATE.
4. CONSTITUENT LIST IS BASED ON THE CONSTITUENTS OF CONCERNS IDENTIFIED IN THE REMEDIAL INVESTIGATION/FEASIBILITY STUDY WORK PLAN.
5. ALL CONCENTRATIONS ARE MILLIGRAMS PER LITER (mg/L).
6. SHADED VALUES EXCEED THE NYSDEC TOG 1.1.1 WATER GUIDANCE VALUES.
7. ND = NOT DETECTED.
8. – = NOT ANALYZED.
9. J = INDICATES AN ESTIMATED VALUE.
10. DUPLICATE RESULTS ARE PROVIDED IN BRACKETS.

0 60' 120'
GRAPHIC SCALE

GULF OIL TERMINAL
(CHEVRON FACILITY #6518040)
OCEANSIDE, NEW YORK

VOC CONCENTRATIONS
SHALLOW SAND FILL - GROUNDWATER

ARCADIS

FIGURE
4

CITY: SYRACUSE DIV/GROUP: ENVCAD DB: G. STOWELL L. FORAKER LD: G. STOWELL PIC: J. VOGELEY PM: W. McCUNE TM: W. McCUNE LYR: ON=*; OFF=REF* PLOTSTYLETABLE: PLTFULL.CTB PLOTTED: 9/2/2010 4:08 PM BY: STOWELL, GARY
G:\ENVCAD\SYRACUSE\ACT\B0047517\0003\00001\DWG\DATABOXES\47517C04.dwg LAYOUT: 4 SAVED: 9/2/2010 3:43 PM ACADVER: 18.0S (LMS TECH) PAGESETUP: C-LD2B-PDF-CONV
IMAGES: PROJECTNAME: ----
XREFS: 47517X01 47517X00

MW-27-D2

Date	10/27/2004	3/9/2005	8/10/2005	3/15/2006
B	1.0	**1.9**	**1.7**	ND
E	ND	**13**	**9.3**	**6.6 J**
T	2.8	1.7	2.0	ND
X	**270**	**41**	**43**	**6.6 J**
MTBE	**64**	**85**	**100**	**110**
TCE	4.0	2.5	**18**	**8.6**
cis-1,2-DCE	**12**	**30**	**45**	**43**
VC	ND	**2.3**	ND	**18**
MC	ND	ND	2.5	**16**

MW-27-D1

Date	10/27/2004	3/9/2005	8/10/2005	3/15/2006
B	**5.2**	**6.2 J**	**5.2**	**5.4**
E	ND	ND	ND	ND
T	ND	ND	ND	ND
X	3.7	ND	ND	ND
MTBE	**150**	**280**	**240**	**340**
TCE	**9.4**	**77**	**60**	**8.6 J**
cis-1,2-DCE	**520**	**1700**	**1100**	**1200**
VC	ND	ND	**15**	**18**
MC	ND	ND	**9.0**	ND

MW-33D

Date	1/9/2007
B	ND
E	0.41 J
T	0.44 J
X	0.39
MTBE	3.6
TCE	ND
cis-1,2-DCE	ND
VC	ND
MC	ND

MW-32D

Date	1/8/2007
B	**11**
E	0.28 J
T	0.43 J
X	0.90 J
MTBE	**80**
TCE	ND
cis-1,2-DCE	ND
VC	ND
MC	ND

MW-36D

Date	1/9/2007
B	ND
E	ND
T	0.36 J
X	ND
MTBE	**180**
TCE	1.6
cis-1,2-DCE	ND
VC	ND
MC	ND

MW-24VD

Date	1/9/2007
B	ND
E	ND
T	1.3 J
X	ND
MTBE	5.6
TCE	**18**
cis-1,2-DCE	**7.8**
VC	ND
MC	ND

MW-25VD

Date	1/9/2007
B	ND [ND]
E	ND [ND]
T	0.29 J [0.22 J]
X	0.35 [ND]
MTBE	0.43 J [ND]
TCE	**24 [20]**
cis-1,2-DCE	**5.1 [4.4]**
VC	ND [ND]
MC	**12 [11]**

MW-24-D1

Date	10/27/2004	3/9/2005	8/10/2005	3/15/2006
B	**4.7**	**3.7 J**	**1.7**	**4.8**
E	ND	ND	ND	1.8 J
T	2.3	ND	0.90	2.5 J
X	ND	ND	ND	3.8 J
MTBE	**1100 J**	**820**	**590**	**1200**
TCE	**860**	**1400**	**530**	**1400**
cis-1,2-DCE	**280**	**240**	**240**	**670**
VC	**18**	**21**	**6.5**	**26**
MC	**6.6**	ND	ND	ND

MW-24-D2

Date	10/27/2004	3/9/2005	8/10/2005	3/15/2006
B	ND	0.50 J	ND	ND
E	ND	ND	ND	ND
T	0.30 J	0.30 J	0.20	ND
X	0.90 J	0.30 J	ND	ND
MTBE	**120**	**270**	**220**	**290**
TCE	**20**	**160**	**130**	**20**
cis-1,2-DCE	**5.9**	**33**	ND	**62**
VC	ND	**5.2**	ND	ND
MC	1.2 J	3.0	ND	ND

MW-25-D2

Date	10/27/2004	3/9/2005	8/10/2005	3/15/2006
B	**7.5**	ND	**220**	ND
E	**10**	ND	ND	ND
T	**19**	ND	**290**	ND
X	**30**	ND	ND	ND
MTBE	**280**	**2600**	**3500**	**3200 J**
TCE	**11000**	**500000**	**500000**	**880000**
cis-1,2-DCE	**820**	**21000**	**26000**	**26000**
VC	ND	ND	ND	ND
MC	**6500**	**140000**	**230000**	**320000**

MW-25-D1

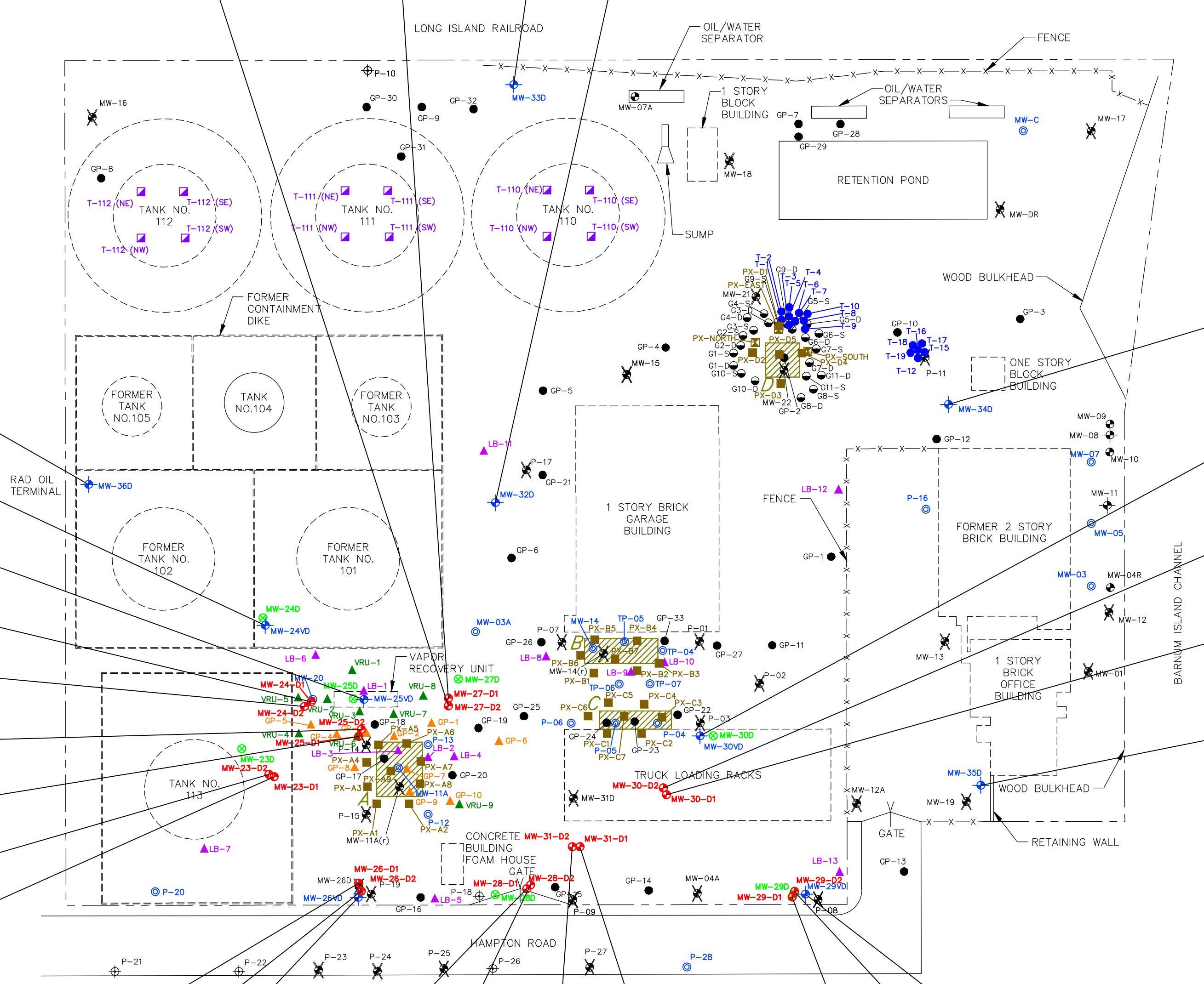
Date	10/27/2004	3/9/2005	3/15/2005	8/10/2005
B	ND	ND	**15**	**8.6**
E	ND	ND	**9.5 J**	ND
T	**120**	**16 J**	**23 J**	**12**
X	ND	**21**	**28 J**	**21**
MTBE	**1100**	**1100**	**1300**	**1100**
TCE	**72000**	**1500**	**900**	**410**
cis-1,2-DCE	**6500**	**5500**	**10000**	**5000**
VC	ND	**2300**	**3100**	**620**
MC	**25000**	**1100**	**1000**	**1200**

MW-23-D2

Date	10/27/2004	3/9/2005	8/10/2005	3/15/2006
B	**1.2**	0.73 J	0.69	ND
E	ND	ND	ND	ND
T	0.30 J	ND	0.20	ND
X	ND	ND	ND	ND
MTBE	**65**	**200**	**220**	**470**
TCE	ND	ND	ND	ND
cis-1,2-DCE	ND	ND	ND	ND
VC	ND	ND	ND	ND
MC	ND	ND	ND	ND

MW-23-D1

Date	10/27/2004	3/9/2005	8/10/2005	3/15/2006
B	**1.4**	**2.3**	**12**	**13**
E	0.32 J	1.2	4.0	4.3 J
T	0.40	ND	**9.6**	**9.0 J**
X	0.90 J	0.20	**12**	**11 J**
MTBE	**180**	**620**	**470**	**680**
TCE	ND	ND	0.31	ND
cis-1,2-DCE	1.5	2.8	**160**	**55**
VC	ND	1.2	**140**	**250**
MC	4.1	ND	ND	ND



MW-34D

Date	1/8/2007
B	**70**
E	0.87 J
T	2.2
X	3.6
MTBE	9.8
TCE	ND
cis-1,2-DCE	ND
VC	ND
MC	ND

MW-30VD

Date	1/8/2007
B	0.52 J
E	0.58 J
T	1.0
X	1.3
MTBE	9.1
TCE	ND
cis-1,2-DCE	ND
VC	ND
MC	ND

MW-30-D2

Date	10/27/2004	3/10/2005	8/11/2005	3/15/2006
B	**18**	**3.0**	**1.1**	**1.1**
E	0.70 J	ND	ND	ND
T	0.80	0.20 J	0.20	ND
X	3.3	0.30 J	ND	ND
MTBE	**150**	**69**	**37**	**42**
TCE	**7.0**	0.80 J	ND	**54**
cis-1,2-DCE	0.30 J	ND	ND	ND
VC	ND	ND	ND	ND
MC	ND	ND	ND	2.5 J

MW-30-D1

Date	10/27/2004	3/10/2005	8/11/2005	3/15/2006
B	**1600**	**2100**	**1500**	**1800**
E	**12**	**16 J**	**13**	**13 J**
T	**7.9**	**10 J**	**9.6**	ND
X	**16**	**30**	**21**	ND
MTBE	**9100**	**11000**	**11000**	**13000**
TCE	ND	ND	ND	ND
cis-1,2-DCE	ND	ND	ND	ND
VC	ND	ND	ND	ND
MC	ND	ND	ND	ND

MW-35D

Date	1/8/2007
B	**2.7**
E	0.31 J
T	0.60 J
X	0.48 J
MTBE	3.6
TCE	ND
cis-1,2-DCE	ND
VC	ND
MC	ND

MW-26-D1

Date	10/26/2004	3/9/2005	8/10/2005
B	**2.9**	**7.8**	**7.5**
E	0.90	4.5	2.8
T	0.50	0.50 J	0.60
X	0.80	1.1	1.1
MTBE	**110**	**520**	**640**
TCE	**88**	0.50 J	2.8
cis-1,2-DCE	**6.2**	ND	**12**
VC	ND	**23**	**75**
MC	**8.4**	ND	ND

MW-26-D2

Date	10/26/2004	3/9/2005	8/10/2005
B	ND	ND	ND
E	ND	ND	ND
T	ND	ND	ND
X	0.40	ND	ND
MTBE	**130**	**140**	**120**
TCE	**80**	**15**	**6.1**
cis-1,2-DCE	2.7	0.60 J	ND
VC	ND	ND	ND
MC	3.5	0.70 J	ND

MW-26VD

Date	1/9/2007
B	0.60 J
E	0.52 J
T	2.1
X	1.5
MTBE	ND
TCE	0.59 J
cis-1,2-DCE	ND
VC	ND
MC	ND

MW-28-D1

Date	10/27/2004	3/10/2005	8/10/2005
B	**11**	**8.5**	**15**
E	2.6	4.0	3.2
T	4.8	**8.2**	1.4
X	**5.5**	**8.9**	**6.9**
MTBE	**150**	**380**	**360**
TCE	**14**	2.0	0.60
cis-1,2-DCE	**12**	0.90 J	2.6
VC	ND	**6.7**	**7.5**
MC	0.60 J	ND	ND

MW-28-D2

Date	10/27/2004	3/10/2005	8/10/2005	3/15/2006
B	0.60 J	0.60 J	0.40	ND
E	ND	ND	ND	ND
T	0.50 J	0.30 J	0.20	ND
X	0.70 J	0.30 J	ND	ND
MTBE	**15**	9.7	6.2	6.5
TCE	2.0	0.80 J	ND	ND
cis-1,2-DCE	1.3	0.90 J	0.30	ND
VC	ND	ND	ND	ND
MC	ND	ND	ND	ND

MW-31-D2

Date	10/27/2004	3/10/2005	8/10/2005
B	**8.4**	ND	ND
E	0.60	ND	ND
T	0.40	ND	ND
X	1.0	ND	ND
MTBE	**18**	0.30 J	0.32 J
TCE	0.90	0.80 J	ND
cis-1,2-DCE	0.40	ND	ND
VC	ND	ND	ND
MC	ND	ND	ND

MW-31-D1

Date	10/27/2004	3/10/2005	8/10/2005
B	**75**	**130**	**130**
E	**15**	**33**	**33**
T	**11**	**24**	**24**
X	**20**	**49**	**49**
MTBE	**160**	**180**	**180**
TCE	0.50	ND	ND
cis-1,2-DCE	**6.4**	**14 J**	**14**
VC	ND	**6.3**	**6.3**
MC	ND	ND	ND

MW-29-D1

Date	10/27/2004	3/10/2005	8/10/2005	3/15/2006
B	**85**	**42**	**74**	**39**
E	ND	ND	ND	ND
T	1.1 J	0.50	1.0	ND
X	2.2	1.6	2.0	ND
MTBE	**650**	**550**	**560**	**390**
TCE	1.8	ND	1.1	ND
cis-1,2-DCE	ND	ND	ND	ND
VC	ND	ND	ND	ND
MC	ND	ND	ND	ND

MW-29-D2

Date	10/27/2004	3/10/2005	8/11/2005	3/15/2006
B	**5.6**	0.50 J	**1.2**	ND
E	ND	ND	ND	ND
T	ND	ND	0.40	ND
X	ND	ND	ND	ND
MTBE	**770**	**530**	**480**	**1500**
TCE	1.5 J	1.1	0.70	**49**
cis-1,2-DCE	ND	ND	ND	2.2 J
VC	ND	ND	ND	ND
MC	**6.0**	ND	ND	2.5 J

MW-29VD

Date	1/9/2007
B	ND
E	ND
T	0.43 J
X	ND
MTBE	1.3
TCE	ND
cis-1,2-DCE	ND
VC	ND
MC	ND

LEGEND:

- ⌖ TEMPORARY PIEZOMETER (INSTALLED BY LBG)
- ⌖ DEEP UNDERLYING SAND WELL
- ⌖ EXISTING MONITORING WELL
- ◎ ABANDONED MONITORING POINT
- ⊗ PAIRED WELL LOCATION
- ✕ DESTROYED MONITORING POINT (MARCH – AUGUST 2005 GULF SITE DEMOLITION)
- ● 2000 LBG GEOPROBE SOIL BORING LOCATION
- ▲ 2000 BBL SOIL BORING LOCATION
- ▲ 2002 BBL SOIL BORING LOCATION
- ■ 2002 BBL POST EXCAVATION BORING LOCATION
- ◪ 2003 LBG SOIL SAMPLING TANK LOCATION
- ▲ 2004 LANGAN SOIL BORING LOCATION
- ● 2004 CEDAR CREEK TEMPORARY WELL
- ◒ 2005 BBL BORING LOCATION
- ⊠ 2005 BBL POST EXCAVATION BORING LOCATION
- ⊕ 2005 BBL PAIRED WELL LOCATION
- ▨ EXCAVATION LOCATIONS (OCTOBER 2002)

Analyte	Short Name	NYSDEC TOGS 1.1.1 Water Guidance Values
Benzene	B	1
Ethylbenzene	E	5
Toluene	T	5
Xylenes (total)	X	5
Methyl tert-butyl ether	MTBE	10
Trichloroethene	TCE	5
cis-1,2-Dichloroethene	cis-1,2-DCE	5
Vinyl Chloride	VC	2
Methylene Chloride	MC	5

NOTES:

1. SITE PLAN DERIVED FROM STORB, INC. DRAWING ENTITLED "PAVING PLAN-GULF OIL COMPANY-U.S., OCEANSIDE TERMINAL" AUGUST 7, 1976 AND LANGAN DRAWING ENTITLED "HISTORICAL SOIL AND GROUNDWATER SAMPLING PLAN MAY 11, 2007.
2. MONITORING WELL LOCATIONS SURVEYED BY A. TAY.
3. LOCATIONS OF DEEP UNDERLYING SAND WELLS AND SOIL BORINGS ARE APPROXIMATE.
4. CONSTITUENT LIST IS BASED ON THE CONSTITUENTS OF CONCERNS IDENTIFIED IN THE REMEDIAL INVESTIGATION/FEASIBILITY STUDY WORK PLAN.
5. ALL CONCENTRATIONS ARE MILLIGRAMS PER LITER (mg/L).
6. SHADED VALUES EXCEED THE NYSDEC TOG 1.1.1 WATER GUIDANCE VALUES.
7. ND = NOT DETECTED.
8. J = INDICATES AN ESTIMATED VALUE.
9. DUPLICATE RESULTS ARE PROVIDED IN BRACKETS.

0 60' 120'
GRAPHIC SCALE

GULF OIL TERMINAL
(CHEVRON FACILITY #6518040)
OCEANSIDE, NEW YORK

VOC CONCENTRATIONS
DEEP SAND UNIT - GROUNDWATER

ARCADIS

FIGURE
5

CITY: SYRACUSE DIV/GROUP: ENVCAD DB: G. STOWELL L. FORAKER LD: G. STOWELL PIC: J. VOGELEY PM: W. McCUNE TM: W. McCUNE LYR: ON=*; OFF=*REF*
G:\ENVCAD\SYRACUSE\ACT\B0047517\0003\00001\DWG\DATABOXES\47517C05.dwg LAYOUT: 5 SAVED: 9/2/2010 3:42 PM ACADVER: 18.0S (LMS TECH) PAGESETUP: C-LD2B-PDF-CONV PLOTSTYLETABLE: PLTFULL.CTB PLOTTED: 9/2/2010 4:06 PM BY: STOWELL, GARY
IMAGES: PROJECTNAME: ----
XREFS: 47517X01 47517X00

ARCADIS

Appendix E

Community Air Monitoring Plan

ARCADIS
Infrastructure, environment, buildings

Imagine the result

Chevron Environmental Management Company

Community Air Monitoring Plan

Former Gulf Oil Terminal Oceanside
Town of Hempstead, New York
NYSDEC Site No. 130165

September 2010

ARCADIS

William McCune, P.G.
Project Manager

Community Air Monitoring Plan

Former Gulf Oil Terminal
Oceanside
Town of Hempstead, New York
NYSDEC Site No. 130165

Prepared for:
Chevron Environmental Management Company

Prepared by:
ARCADIS U.S., Inc.
6723 Towpath Road
P.O. Box 66
Syracuse
New York 13214-0066
Tel 315.446.9120
Fax 315.446.8053

Our Ref.:
B0047517.0003.0001

Date:
September 2010

1. Introduction 1

1.1 General 1

1.2 Site Description 1

1.3 Summary of Selected Site Remedial Investigation Activities 1

1.4 Potential Air Emissions Related to Remedial Action Activities 1

1.5 Air/Odor Emissions and Control Measures 2

2. Air Monitoring Procedures 3

2.1 General 3

2.2 Sampling Location Selection 3

2.3 VOCs Monitoring 3

2.4 Particulate Matter Monitoring 3

2.5 Action Levels 4

2.5.1 Action Levels for VOCs 4

2.5.2 Action Level for PM_{10} 5

2.6 Meteorological Monitoring 5

2.7 Instrument Calibration 5

3. Monitoring Schedule and Data Collection and Reporting 6

3.1 General 6

3.2 Monitoring Schedule 6

3.3 Data Collection and Reporting 6

Attachments

A Generic Community Air Monitoring Plan

B Fugitive Dust Suppression and Particulate Monitoring Programs at Inactive Hazardous Waste Sites

C Monitoring Equipment Specifications

1. Introduction

1.1 General

On behalf of Chevron Environmental Management Company (CEMC), ARCADIS U.S., Inc. (ARCADIS) is submitting this *Community Air Monitoring Plan* (CAMP) to the New York State Department of Environmental Conservation (NYSDEC) in accordance with the Order on Consent and Administrative Settlement for the former Gulf Oil Terminal in Oceanside, Township of Hempstead, New York (site). This CAMP fulfills the requirements set forth by the New York State Department of Health (NYSDOH) Generic Community Air Monitoring Plan, dated June 2000 (Attachment A), and the NYSDEC's Technical and Administrative Guidance Memorandum (TAGM) 4031, "Fugitive Dust Suppression and Particulate Monitoring Program at Inactive Hazardous Waste Sites" (Attachment B). The intent of this CAMP is to provide for a measure of protection of the downwind communities from potential airborne releases of constituents of concern during Remedial Investigation/Feasibility Study (RI/FS) investigation activities. As such, this CAMP specifies the potential air emissions, air monitoring procedures, monitoring schedule and data collection and reporting for the investigation activities to be conducted as described below.

1.2 Site Description

The site consists of 7.2 acres located at 1 Industrial Place in Oceanside, Township of Hempstead, Nassau County, New York. A Site Location Map is presented on Figure 1. The site is currently unoccupied and all former buildings have been demolished. The site is bound to the south by Barnum's Channel, to the east by the Long Island Rail Road, to the north by the former Exxon Petroleum Terminal, and to the west by Hampton Road. A junk yard and office buildings are located west of Hampton Road fronting Hog Island Channel. Daly Boulevard is located north of the former Exxon Petroleum Terminal and another petroleum terminal (Sprague) is located north of Daly Boulevard.

1.3 Summary of Selected Site Remedial Investigation Activities

The proposed RI/FS includes soil boring and sampling, test pit excavation, monitoring well/piezometer installation, and groundwater sampling. A more detailed description of the investigation activities can be found in the RI/FS Work Plan.

1.4 Potential Air Emissions Related to Remedial Action Activities

Certain intrusive RI activities to be conducted at the site have the potential to generate localized impacts to air quality including drilling, test pit excavation, and soil sampling. Some non-intrusive RI activities to be conducted may also have the potential to generate impacts to air quality, and include the collection of groundwater samples.

1.5 Air/Odor Emissions and Control Measures

Air emissions control and fugitive dust suppression techniques will be used during the RI/FS activities identified above, as necessary, to limit the air/odor emissions from the site. Air monitoring for the specific purpose of protecting the community from site activity impacts (and verification thereof) will take place during both intrusive and non-intrusive site activities.

During intrusive and non-intrusive site RI/FS activities (excluding groundwater sampling), odor and dust control measures will be available at the site and used when necessary. The following dust and odor suppression measures may be used during these activities, depending upon specific circumstances and air monitoring results:

- water spray; and
- polyethylene sheeting (for covering drill cuttings, soil stockpiles, etc.).

Polyethylene sheeting will be used to control nuisance odors and volatile organic compound (VOC) emissions, as needed. Also, dust emissions at the site will be controlled by spraying water on exposed dry surface soil areas (e.g., on test pit excavation faces, soils removed from test pits, stockpiled drill cuttings, etc., as appropriate), through the use of silt fences, and by covering soil stockpiles. Odor and dust control measures will be implemented based on visual or olfactory observations, and the results of airborne particulate and VOC monitoring.

2. Air Monitoring Procedures

2.1 General

Real-time air monitoring will be implemented at the site for VOCs, and particulate matter less than 10 microns in diameter (PM_{10}). A site boundary will be established for the purpose of air monitoring. Upwind and downwind monitoring locations will be determined through visual observation (wind vane, windsock, or similar technique). Monitoring will occur at each sample location and will include the use of hand-held direct-reading survey instruments.

2.2 Sampling Location Selection

Sampling activities will be determined daily based on visual observation of a wind direction. A single upwind location will be selected daily where both VOC and PM_{10} will be recorded. This upwind location will be established at the start of the workday, each day before the start of RI activities. Sampling activities will continue in a downwind direction throughout the day. If wind direction during the workday shifts greater than approximately +/-60 degrees from original upwind, then new upwind and downwind sampling locations will be established. Any location changes will be documented in the field logbook.

2.3 VOCs Monitoring

As required by the NYSDOH guidance for community air monitoring during intrusive activities, VOCs will be monitored continuously during ground intrusive site activities (test pitting and installation of soil borings or monitoring wells) with instrumentation that is equipped with electronic data-logging capabilities. A MiniRAE 2000 (or equivalent) will be used to conduct the real-time VOC monitoring. Attachment C provides detailed information on the MiniRAE 2000. All 15-minute readings will be recorded in the field logbook, as well as any instantaneous readings taken to facilitate activity decisions.

During non-intrusive site activities (collection of soil samples, collection of groundwater samples from existing monitoring wells), VOCs will be monitored periodically. Periodic monitoring may include monitoring upon arrival at the sample location, monitoring while opening a well cap, monitoring during well bailing and/or purging, and/or monitoring prior to leaving a sample location. However, if a sampling location is near potentially exposed individuals, VOCs will be monitored continuously during sampling activities at that location.

2.4 Particulate Matter Monitoring

As required by the NYSDOH guidance, real-time particulate matter will be monitored continuously during intrusive site activities using instrumentation equipped with electronic data-logging capabilities. A MIE

DataRAM (or equivalent) will be used to conduct the real-time PM_{10} monitoring. Attachment C provides detailed information on the MIE DataRAM. All 15-minute readings will be recorded in the field logbook, as well as any instantaneous readings taken to facilitate activity decisions.

Fugitive dust migration will be visually assessed during all work activities, and reasonable dust suppression techniques will be used during any site activities that may generate fugitive dust. These activities and their design controls were discussed previously in Section 1.4 of this plan.

2.5 Action Levels

The action levels provided below are to be used to initiate response actions, if necessary, based on real-time monitoring.

2.5.1 Action Levels for VOCs

As outlined in the NYSDOH guidance document for CAMPs, if the ambient air concentration of total VOCs exceeds 5 parts per million (ppm) above the background (upwind location) for the 15-minute average, intrusive site activities will be temporarily halted while monitoring continues. If the total VOC concentration readily decreases (through observation of instantaneous readings) below 5 ppm above background, then intrusive site activities can resume with continuous monitoring.

If the ambient air concentrations of total VOCs persist at levels in excess of 5 ppm above background but less than 25 ppm above background, intrusive site work activities will be halted, the source of the elevated VOC concentrations identified, corrective actions to reduce or abate the emissions undertaken, and air monitoring will be continued. Once these actions have been implemented, intrusive site work activities can resume provided the following two conditions are met:

- The 15-minute average VOC concentrations remain below 5 ppm above background.
- The VOC level 200 feet downwind of the sample location or half the distance to the nearest potential receptor or residential/commercial structure (whichever is less but in no case less than 20 feet) is below 5 ppm over background for the 15-minute average.

If the ambient air concentrations of total VOCs are above 25 ppm above background, the intrusive site activities must cease, and emissions control measures must be implemented.

Periodic monitoring for VOCs is required during non-intrusive activities such as collection of soil samples, or the collection of groundwater samples from existing monitoring wells. If these activities are undertaken at the site, ambient direct-reading (instantaneous) VOC data will be periodically collected at the location of the non-intrusive activity and recorded in the field activity logbooks.

2.5.2 Action Level for PM_{10}

As required by the NYSDOH guidance, if the ambient air concentration of PM_{10} at any one (or more) of the sampling locations is noted at levels in excess of 100 micrograms per cubic meter ($\mu g/m^3$) above the background (upwind location), or if airborne dust is observed leaving the work area, intrusive site activities will be temporarily halted. The source of the elevated PM_{10} concentration is to be identified, corrective actions to reduce or abate the emissions will be undertaken, and air monitoring will continue. Work may continue following the implementation of dust suppression techniques provided the PM_{10} levels do not exceed 150 $\mu g/m^3$ above background.

If, after implementation of dust suppression techniques, PM_{10} levels are greater than 150 $\mu g/m^3$ above background, work must be stopped and site activities must be re-evaluated. Work may only resume provided that the dust suppression measures and other controls are successful in reducing PM_{10} levels less than 150 $\mu g/m^3$ above background and in preventing visible dust from leaving the site.

If the ambient air concentration of PM_{10} is above 150 $\mu g/m^3$ above background, the intrusive site activities must cease and emissions control measures must be implemented.

2.6 Meteorological Monitoring

Wind direction is the only meteorological information considered relevant for the RI/FS activities and CAMP. Meteorological monitoring will be conducted periodically at the site using a windsock, wind vane, or other appropriate equipment. Wind direction will be established at the start of each work day and may be re-established at any time during the work day if a significant shift in wind direction is noted.

2.7 Instrument Calibration

Calibration of the VOC and PM_{10} instrumentation will occur in accordance with each of the equipment manufacturer's calibration and quality assurance requirements. The VOC and PM_{10} monitors will be calibrated at least daily, and calibrations will be recorded in the field activity logbook.

3. Monitoring Schedule and Data Collection and Reporting

3.1 General

The proposed monitoring schedule and data collection and reporting requirements are discussed below.

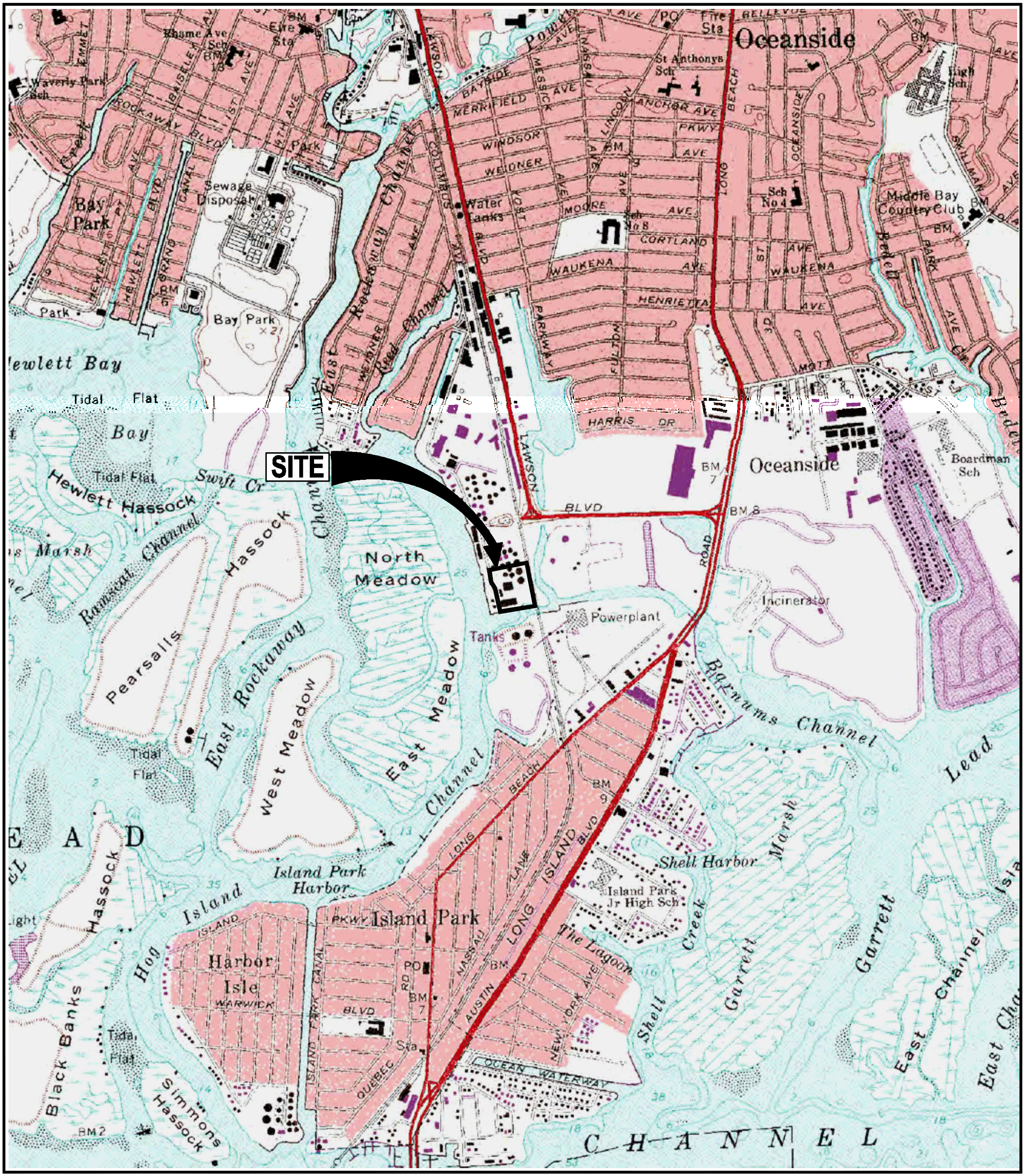
3.2 Monitoring Schedule

Real-time VOC and PM_{10} monitoring will be performed continuously throughout the remedial action during intrusive site/materials handling activities. VOC monitoring will also be performed during non-intrusive sampling-type activities. Wind direction will be determined at the start of each day and at any other appropriate time during RI/FS activities.

3.3 Data Collection and Reporting

Air monitoring data will be collected continuously from VOC and PM_{10} monitors during intrusive site activities by an electronic data-logging system. The data management software will be set up so that instantaneous observed readings would be recorded by the electronic data acquisition system and averaged over 15-minute time periods. The 15-minute readings and instantaneous readings taken to facilitate activity decisions will be recorded and archived for review by NYSDOH and NYSDEC personnel.

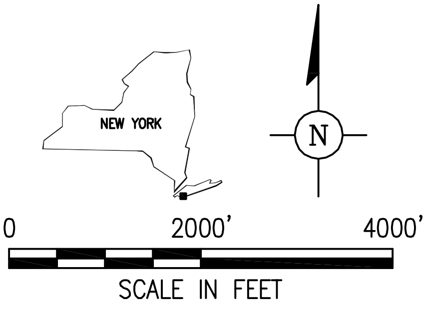
Figure



MAP SOURCE: USGS 7.5 MINUTE QUADRANGLE 1979 LYNBROOK AND LAWRANCE, NEW YORK

GULF OIL TERMINAL
(CHEVRON FACILITY #6518040)
OCEANSIDE, NEW YORK

SITE LOCATION MAP



ARCADIS

FIGURE
1

CITY:Melville - NY DIV/GROUP:ENR1 DB:A.Sanchez LD: PIC: PM: TM: LYR:ON=*;OFF=*REF*
G:\ENVCAD\Melville-NY\RETURN-TO\Mahwah-NJ\B0047517\0003\00001\Location Map.dwg LAYOUT: 1 SAVED: 5/6/2010 4:15 PM ACADVER: 17.1S (LMS TECH) PAGESETUP: ----PLOTSTYLETABLE: ARCADIS_MELVILLE.CTB PLOTTED: 5/6/2010 4:15 PM BY: SANCHEZ, ADRIAN

XREFS: IMAGES: LAWRENCE.TIF LYNBROOK.TIF SEA CLIFF.TIF PROJECTNAME:

ARCADIS

Attachment A

Generic Community Air Monitoring Plan

APPENDIX 1A

New York State Department of Health
Generic Community Air Monitoring Plan

A Community Air Monitoring Plan (CAMP) requires real-time monitoring for volatile organic compounds (VOCs) and particulates (i.e., dust) at the downwind perimeter of each designated work area when certain activities are in progress at contaminated sites. The CAMP is not intended for use in establishing action levels for worker respiratory protection. Rather, its intent is to provide a measure of protection for the downwind community (i.e., off-site receptors including residences and businesses and on-site workers not directly involved with the subject work activities) from potential airborne contaminant releases as a direct result of investigative and remedial work activities. The action levels specified herein require increased monitoring, corrective actions to abate emissions, and/or work shutdown. Additionally, the CAMP helps to confirm that work activities did not spread contamination off-site through the air.

The generic CAMP presented below will be sufficient to cover many, if not most, sites. Specific requirements should be reviewed for each situation in consultation with NYSDOH to ensure proper applicability. In some cases, a separate site-specific CAMP or supplement may be required. Depending upon the nature of contamination, chemical- specific monitoring with appropriately-sensitive methods may be required. Depending upon the proximity of potentially exposed individuals, more stringent monitoring or response levels than those presented below may be required. Special requirements will be necessary for work within 20 feet of potentially exposed individuals or structures and for indoor work with co-located residences or facilities. These requirements should be determined in consultation with NYSDOH.

Reliance on the CAMP should not preclude simple, common-sense measures to keep VOCs, dust, and odors at a minimum around the work areas.

Community Air Monitoring Plan

Depending upon the nature of known or potential contaminants at each site, real-time air monitoring for volatile organic compounds (VOCs) and/or particulate levels at the perimeter of the exclusion zone or work area will be necessary. Most sites will involve VOC and particulate monitoring; sites known to be contaminated with heavy metals alone may only require particulate monitoring. If radiological contamination is a concern, additional monitoring requirements may be necessary per consultation with appropriate NYSDEC/NYSDOH staff.

Continuous monitoring will be required for all ground intrusive activities and during the demolition of contaminated or potentially contaminated structures. Ground intrusive activities include, but are not limited to, soil/waste excavation and handling, test pitting or trenching, and the installation of soil borings or monitoring wells.

Periodic monitoring for VOCs will be required during non-intrusive activities such as the collection of soil and sediment samples or the collection of groundwater samples from existing monitoring wells. "Periodic" monitoring during sample collection might reasonably consist of taking a reading upon arrival at a sample location, monitoring while opening a well cap or overturning soil, monitoring during well baling/purging, and taking a reading prior to leaving a sample location. In some instances, depending upon the proximity of potentially exposed individuals, continuous monitoring may be required during sampling activities. Examples of such situations include groundwater sampling at wells on the curb of a busy urban street, in the midst of a public park, or adjacent to a school or residence.

VOC Monitoring, Response Levels, and Actions

Volatile organic compounds (VOCs) must be monitored at the downwind perimeter of the immediate work area (i.e., the exclusion zone) on a continuous basis or as otherwise specified. Upwind concentrations should be measured at the start of each workday and periodically thereafter to establish background conditions. The monitoring work should be performed using equipment appropriate to measure the types of contaminants known or suspected to be present. The equipment should be calibrated at least daily for the contaminant(s) of concern or for an appropriate surrogate. The equipment should be capable of calculating 15-minute running average concentrations, which will be compared to the levels specified below.

- If the ambient air concentration of total organic vapors at the downwind perimeter of the work area or exclusion zone exceeds 5 parts per million (ppm) above background for the 15-minute average, work activities must be temporarily halted and monitoring continued. If the total organic vapor level readily decreases (per instantaneous readings) below 5 ppm over background, work activities can resume with continued monitoring.

- If total organic vapor levels at the downwind perimeter of the work area or exclusion zone persist at levels in excess of 5 ppm over background but less than 25 ppm, work activities must be halted, the source of vapors identified, corrective actions taken to abate emissions, and monitoring continued. After these steps, work activities can resume provided that the total organic vapor level 200 feet downwind of the exclusion zone or half the distance to the nearest potential receptor or residential/commercial structure, whichever is less - but in no case less than 20 feet, is below 5 ppm over background for the 15-minute average.

- If the organic vapor level is above 25 ppm at the perimeter of the work area, activities must be shutdown.

All 15-minute readings must be recorded and be available for State (DEC and DOH) personnel to review. Instantaneous readings, if any, used for decision purposes should also be recorded.

Particulate Monitoring, Response Levels, and Actions

Particulate concentrations should be monitored continuously at the upwind and downwind perimeters of the exclusion zone at temporary particulate monitoring stations. The particulate monitoring should be performed using real-time monitoring equipment capable of measuring particulate matter less than 10 micrometers in size (PM-10) and capable of integrating over a period of 15 minutes (or less) for comparison to the airborne particulate action level. The equipment must be equipped with an audible alarm to indicate exceedance of the action level. In addition, fugitive dust migration should be visually assessed during all work activities.

- If the downwind PM-10 particulate level is 100 micrograms per cubic meter (mcg/m^3) greater than background (upwind perimeter) for the 15-minute period or if airborne dust is observed leaving the work area, then dust suppression techniques must be employed. Work may continue with dust suppression techniques provided that downwind PM-10 particulate levels do not exceed 150 mcg/m^3 above the upwind level and provided that no visible dust is migrating from the work area.

- If, after implementation of dust suppression techniques, downwind PM-10 particulate levels are greater than 150 mcg/m^3 above the upwind level, work must be stopped and a re-evaluation of activities initiated. Work can resume provided that dust suppression measures and other controls are successful in reducing the downwind PM-10 particulate concentration to within 150 mcg/m^3 of the upwind level and in preventing visible dust migration.

All readings must be recorded and be available for State (DEC and DOH) personnel to review.

Attachment B

Fugitive Dust Suppression and Particulate Monitoring Programs at Inactive Hazardous Waste Sites

New York State Department of Environmental Conservation

TECHNICAL AND ADMINISTRATIVE GUIDANCE MEMORANDUM #4031

FUGITIVE DUST SUPPRESSION AND PARTICULATE MONITORING PROGRAM AT INACTIVE HAZARDOUS WASTE SITES

TO: Regional Hazardous Waste Remediation Engrs., Bur. Directors & Section Chiefs

FROM: Michael J. O'Toole, Jr., Director, Division of Hazardous Waste Remediation

SUBJECT: DIVISION TECHNICAL AND ADMINISTRATIVE GUIDANCE MEMORANDUM -- FUGITIVE DUST SUPRESSION AND PARTICULATE MONITORING PROGRAM AT INACTIVE HAZARDOUS WASTE SITES

DATE: Oct 27, 1989

Michael J. O'Toole, Jr. (signed)

1 Introduction

Fugitive dust suppression, particulate monitoring, and subsequent action levels for such must be used and applied consistently during remedial activities at hazardous waste sites. This guidance provides a basis for developing and implementing a fugitive dust suppression and particulate monitoring program as an element of a hazardous waste site's health and safety program.

2. Background

Fugitive dust is particulate matter--a generic term for a broad class of chemically and physically diverse substances that exist as discrete particles, liquid droplets or solids, over a wide range of sizes--which becomes airborne and contributes to air quality as a nuisance and threat to human health and the environment.

On July 1, 1987, the United States Environmental Protection Agency (USEPA) revised the ambient air quality standard for particulates so as to reflect direct impact on human health by setting the standard for particulate matter less than ten microns in diameter (PM_{10}); this involves fugitive dust whether contaminated or not. Based upon an examination of air quality composition, respiratory tract deposition, and health effects, PM_{10} is considered conservative for the primary standard--that requisite to protect public health with an adequate margin of safety. The primary standards are 150 ug/m^3 over a 24-hour averaging time and 50 ug/m^3 over an annual averaging time. Both of these standards are to be averaged arithmetically.

There exists real-time monitoring equipment available to measure PM_{10} and capable of integrating over a period of six seconds to ten hours. Combined with an adequate fugitive dust suppression program, such equipment will aid in preventing the off-site migration of contaminated soil. It will also protect both on-site personnel from exposure to high levels of dust and the public around the site from any exposure to any dust. While specifically intended for the protection of on-site personnel as well as the public, this program is not meant to replace long-term monitoring which may be required given the contaminants inherent to the site and its air quality.

3. Guidance

A program for suppressing fugitive dust and monitoring particulate matter at hazardous waste sites can be developed without placing an undue burden on remedial activities while still being protective of health and environment. Since the responsibility for implementing this program ultimately will fall on the party performing the work, these procedures must be incorporated into appropriate work plans. The following fugitive dust suppression and particulate monitoring program will be employed at hazardous waste sites during construction and other activities which warrant its use:

1 Reasonable fugitive dust suppression techniques must be employed during all site activities which may generate fugitive dust.

Particulate monitoring must be employed during the handling of waste or contaminated soil or when activities on site may generate fugitive dust from exposed waste or contaminated soil. Such activities shall also include the excavation, grading, or placement of clean fill, and control measures therefore should be considered.

3. Particulate monitoring must be performed using real-time particulate monitors and shall monitor particulate matter less than ten microns (PM_{10}) with the following minimum performance standards:

Object to be measured: Dust, Mists, Aerosols
Size range: <0.1 to 10 microns
Sensitivity: 0.001 mg/m^3
Range: 0.001 to 10 mg/m^3
Overall Accuracy: ±10% as compared to gravimetric analysis of stearic acid or reference dust

Operating Conditions:
  Temperature: 0 to 40°C
  Humidity: 10 to 99% Relative Humidity

Power: Battery operated with a minimum capacity of eight hours continuous operation

Automatic alarms are suggested.

Particulate levels will be monitored immediately downwind at the working site and integrated over a period not to exceed 15 minutes. Consequently, instrumentation

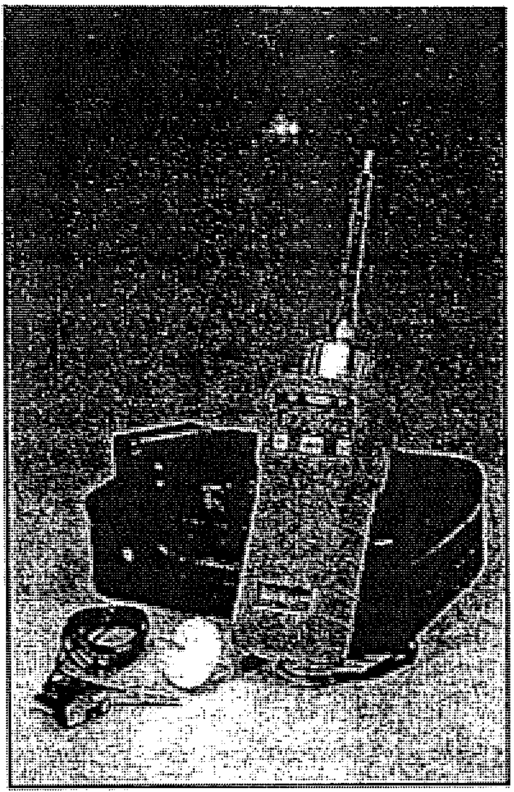
ARCADIS

Attachment C

Monitoring Equipment
Specifications

MiniRAE 2000
Handheld VOC Monitor

- Intrinsically safe
- Smallest handheld VOC monitor
- Datalogging workhorse



This VOC monitor with PID (photoionization detector) sensor weighs just over one pound, yet it's a heavyweight for leak detection, fugitive emissions monitoring to EPA Method 21 and inspecting leaking underground storage tanks. The MiniRAE 2000 is also a highly useful tool in industrial hygiene applications, including confined space entry, personnel and work place monitoring and for emergency response to hazardous spills. This rugged instrument comes with a belt clip.

With built-in correction factors for more than 100 chemicals, the MiniRAE 2000 provides excellent all-around sensitivity to most VOCs, down to 0.1 ppm. Selectable survey and hygiene modes permit the user to set appropriate alarm thresholds for STEL, TWA and low/high level peak values. Datalogging and custom software.

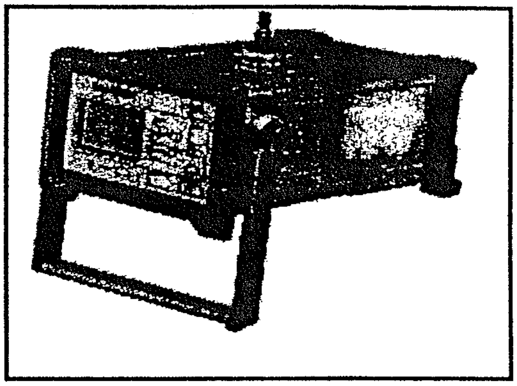
SPECIFICATIONS

Range	Resolution	Response Time	Accuracy
0 to 999 ppm	0.1 ppm	< 3 seconds	± 2 ppm or 10% of reading <2000 ppm
100 to 10,000 ppm	1 ppm	< 3 seconds	± 20% of reading > 2000 ppm
			Calibrated to 100 ppm isobutylene

Sampling Pump	Internal integrated flow rate 400 cc/minute Sample from 100' horizontally or vertically
Datalogging	15,000 points with time/date, header information
Approvals	UL and cUL Class I, Division 1, Groups A, B, C and D, EEx ia IIC T4
Battery	Rechargeable, field changeable NiMH battery pack, 10 hours operation
Dimensions (HWD)	2" x 3" x 8.2"
Weight	19.5 oz

RAE SYSTEMS MiniRAE 2000 PID rents with download cable, zero filter, probe tip, hydrophobic filter, charger, alkaline battery adapter, case and operating manual.

MIE DataRAM Aerosol Monitor
Portable Real-Time Particulate Monitor



- **Real-time measurement of particle concentrations**
- **Datalogging**

The DataRAM aerosol monitor measures concentrations of airborne dust, smoke, mists, haze and fumes with real-time readout. The instrument can be used for exposure sampling of ambient air, continuous unattended monitoring of indoor, duct or process air, as well as environmental and perimeter monitoring. The DataRAM has the widest measurement range of any real-time aerosol monitor — from 0.0001 mg/m³ to 400 mg/m³, or a total span of almost seven decades.

OPTIONAL ACCESSORIES

Respirable Cyclone Precollector, for respirable particle monitoring.
Isokinetic Sampling Probe, for isokinetic sampling within ducts.
Temperature Conditioning Heater, for monitoring above 70 percent RH.
Omnidirectional Sampling Inlet, for ambient monitoring under a variety of wind speeds and directions.
PM-10 Inlet Head, for PM-10 or PM-2.5 ambient particulate monitoring.

SPECIFICATIONS

Concentration Measurement Ranges (autoranging)	0.1 to 999.99 μg/m³, with resolution of 0.1 μg/m³ 1.00 to 39.99 mg/m³, with resolution of 0.01 mg/m³ 40.0 to 399.9 mg/m³, with resolution of 0.1 mg/m³
Accuracy	± 5% of reading ± precision
Particle Size Range of Maximum Response	0.1 to 10 μm
Sample Flow Rate	1.7 to 2.3 lpm
Datalogging	10,000 data points, with average, minimum and maximum concentrations for each point
Output	RS-232 port
Power	Sealed lead-acid battery, 24 hours operation, or AC operation with adapter
Dimensions (HWD)	5.28" x 7.25" x 13.63"
Weight	11.7 lbs

The MIE DataRAM aerosol monitor rents with an AC adapter/charger, serial download cable, software, filter cassette, soft carrying case and operating manual.

W002

ASHTEAD Technology RENTALS

www.ashtead-technology.com