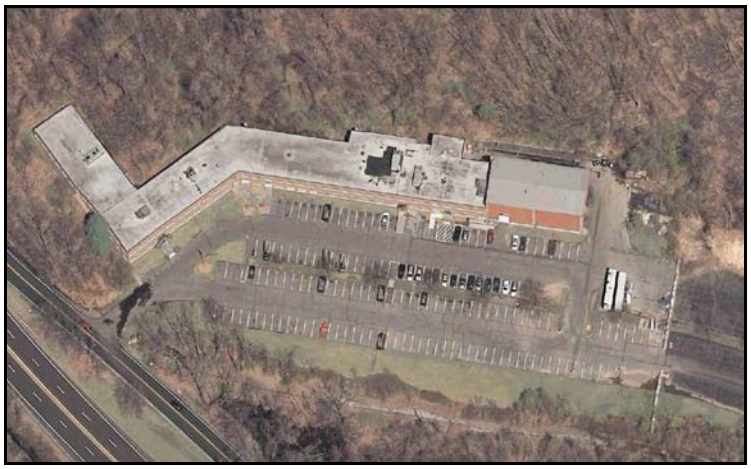
SITE CHARACTERIZATION / PRELIMINARY REMEDIAL INVESTIGATION
QUALITY ASSURANCE PROJECT PLAN
DP 16 LLC
ONE COMMERCE PARK SITE
115 WALL STREET
VALHALLA, NEW YORK



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SITE CHARACTERIZATION / PRELIMINARY REMEDIAL INVESTIGATION QUALITY ASSURANCE PROJECT PLAN

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1.0 INTRODUCTION

As required in the New York State Department of Environmental Conservation (NYSDEC) document entitled: *"Draft DER-10 – Technical Guidance for Site Investigation and Remediation,"* dated December 25, 2002, the purpose of this Quality Assurance Project Plan (QAPP) is to provide the field, office and laboratory quality assurance / quality control (QA / QC) measures and procedures to be followed during the execution of the Site Characterization / Preliminary Remedial Investigation Work Plan (SC / PRI Work Plan) being conducted at the One Commerce Park Site (hereinafter referred to as the "Subject Property") located at 115-117 Wall Street in Valhalla, New York (see ***Figure 1-1***: Site Location Map). According to the NYSDEC Order on Consent and Administrative Settlement (OC), the Subject Property is not currently listed in the New York State Registry of Inactive Hazardous Waste Sites, although the OC references Site # 360054.

It is understood that the NYSDEC is requesting completion of SC / PRI activities at the Subject Property based upon the following:

- As part of RI activities conducted circa 2000 at the southeastern adjoining property known as the Farrand Controls Site (NYSDEC Site Registry No. 3-60-046) located at 99 Wall Street, a groundwater volatile organic compound (VOC) plume possibly attributed to an upgradient site was identified; and,
- The Subject Property, which was indicated to be upgradient or crossgradient to the Farrand Controls Site, was once owned and operated by the Farrand Corporation. Therefore, there is a suspicion that halogenated solvents similar to those found at the 99 Wall Street property may have been utilized and / or disposed of on the Subject Property historically.

1.1 Project Goals and Scope

The SC / PRI Program has been designed to meet the following goals:

- Evaluate on-site, historic and current chemical use and waste disposal practices in order the identify specific areas of concern (AOCs) for subsequent evaluation;
- Investigate and confirm the presence of on-site source areas of contamination at AOCs indentified above, if any, via the collection and analyses of soil vapor samples;
- Confirm the presence of impacted groundwater, if any, observed on the Subject Property;
- Collect sufficient elevation data to confirm the potentiometric surfaces of the shallow, intermediate and deep portions of the aquifer and the inferred groundwater flow direction;
- Investigate possible historic and current upgradient groundwater contaminants migrating onto the Subject Property;
- Complete sufficient groundwater flow direction, topographic data, and preferential pathway analyses on the Subject Property to evaluate the possibility of a relationship between contamination found underlying the Subject Property, if any, and the adjacent Farrand Controls property. This objective will focus on the impacted areas on the Farrand Controls property specifically attributed by the NYSDEC to be the result of an "*apparent upgradient source*;" and,
- Meet the RI requirements set forth by the NYSDEC and USEPA to limit the need for additional investigation activities to the degree practical given the information available at this time.

The scope of the planned investigation includes the following:

- A comprehensive document review, employee interview task, and site inspection task to evaluate the Subject Property with respect to the presence and locations where hazardous materials are or may have been stored, handled and / or disposed of on-site;
- Completion of a geophysical survey to determine if reported, historic sanitary waste disposal system(s) is / are still present on the Subject Property and if so, to determine their configuration. The geophysical survey will also assist in the mark out of on-site, sub-surface utilities (that are not addressed through public mark out call-in numbers);
- Collection and analyses of soil vapor samples from beneath and adjacent to the building envelope utilizing the GORE survey technique. Soil vapor sample results will provide a good overview of AOCs, if any, and will allow the completion of a more focused soil and groundwater investigation program;

- Collection and analyses of multi-depth soil and groundwater samples from possible on-site AOCs identified during the document review and soil vapor sampling phase of the project. Multi-depth soil and groundwater grab samples are to be collected utilizing the Geoprobe drilling technique; and,

- The installation and sampling of permanent groundwater monitoring wells at multiple depths within the overburden deposits. Well locations will be optimized based upon Geoprobe and soil vapor data.

QA / QC controls are required to prevent, identify, and correct errors that may occur at any point during the investigation process including surveys; monitoring well installation; sampling and handling; sample analysis; and final reporting.

1.2 Data Quality Objectives and Criteria

The overall data quality objectives (DQOs) of this QAPP are to ensure the appropriate development and implementation of procedures for field sampling, chains-of-custody (COC) protocol, laboratory analyses and reporting that will yield reliable data that can easily be verified and defended. Specific procedures to be utilized for sampling, COC tracking, instrument calibration, laboratory analyses, field procedures / reporting, internal QC audits, and corrective actions are described in this QAPP. The purpose of this section is to define goals for data completeness, accuracy, precision, bias, representativeness and comparability. The QA requirements for each parameter are contained in the EPA guidance document *Test Methods for Evaluating Solid Waste*, *Physical/Chemical Methods (SW-846)* and *NYSDEC DER-10*.

1.2.1 Data Accuracy

Accuracy is defined as the degree of agreement of a measurement X, with an accepted reference value T, and is measured by calculating percent recovery. The difference between X and T will be expressed as a percentage of the reference value according to the formula:

- Percentage Difference = 100 (X-T)/T

Accuracy is influenced by random error or precision and systematic error or bias that may occur during sampling and analysis. External accuracy audits will be conducted by the quality assurance officer (QAO) with the support of the approved laboratory by submitting blind standards, spikes and field blanks to the laboratory. The analytical results must meet acceptable accuracy objectives.

Each laboratory utilized for the project must participate in ongoing performance audit programs administered by Apex and / or by New York State. Reference or spiked samples will be used where appropriate.

1.2.2 Data Precision

Precision is defined as a measure of mutual agreement among individual measurements of the same property. External precision audits will be conducted by submitting blind duplicate samples to the laboratory and comparing the results with the acceptance criteria.

1.2.3 Data Bias

Bias is the measure of the systematic variance in the expected sample measurement from the samples true value.

1.2.4 Data Completeness

Completeness is defined as the percentage of measurements made which are judged to be valid measurements. The completeness goal is to generate a sufficient amount of valid data. A 90 percent completeness value will be utilized as a guideline.

1.2.5 Data Representativeness

Representativeness is the assurance that analytical data are derived from sampling techniques and laboratory procedures that achieve a characteristic sample. To assure representativeness, all sampling will be conducted according to the protocols set forth in **Section 3.0**, below.

1.2.6 Data Comparability

Comparability is a qualitative parameter expressing the confidence with which one data set can be compared with another. The data generated will be reported in units consistent with other laboratories that report similar analyses and data QA / QC protocols. This will allow comparison of data among organizations and the adjacent Farrand Controls Site. Standardized data formats for calculating and reporting of analytical results will facilitate the process. The standard operating procedures (SOPs) for sampling described in **Section 3.0** will be followed for each sampling event.

2.0 PROJECT ORGANIZATION

Since the key element of any remedial investigation is the project team, a project team with extensive NYSDEC Inactive Hazardous Waste Disposal Site and USEPA Superfund site experience has been assembled. The proposed project team organization is presented in ***Figure 2-1***.

The project manager for this project will be ***Richard J. Baldwin, C.P.G., P.G.*** Mr. Baldwin is a hydrogeologist with more than 20 years of experience in the fields of environmental consulting, hydrogeology and geology with particular experience in conducting and supervising environmental investigations and remedial actions at industrial, private, Federal and publicly-owned facilities and sites. Additionally, Mr. Baldwin has experience in evaluating potential environmental impacts of projects including golf courses, housing developments, senior housing, schools and retail shopping centers. For the last several years, Mr. Baldwin's work has focused primarily on sites and facilities located in the Long Island, New York City and Upstate New York areas. A copy of Mr. Baldwin's resume is included in ***Appendix A***.

The Apex Project Director and Quality Assurance Officer (QAO) for the project will be **Daniel J. Smith, P.E.** Mr. Smith is a New York State-licensed Professional Engineer (PE) in Chemical Engineering with over 20 years of experience in the environmental consulting industry. Mr. Smith has been responsible for the implementation of investigations and remedial actions at numerous NYSDEC-regulated sites under the SPILLS and IHWDS program. The QAO's project responsibilities include conducting site audits to ensure that the QA/ QC procedures included in this Work Plan are being implemented. Additionally, the QAO will review all of the analytical data collected as part of the investigation to assure that the data are of sufficient quality to support the goals of the investigation. Mr. Smith is an officer of Apex and the Project Director for this project; thus he has the authority to ensure that appropriate staffing is available to complete the investigation. A copy of Mr. Smith's resume is included in ***Appendix A***.

Field sampling and oversight of contractors will be performed by personnel experienced in proper field sampling techniques. All analytical work will be performed by a NYSDOH ELAP-certified analytical laboratory and drilling services will be performed by experienced contractors with extensive NYSDEC regulated site experience.

3.0 MEASUREMENT/DATA ACQUISITION

Previous soil and groundwater analytical results for the adjacent Farrand Controls Site indicated that the potential contaminants of concern were limited to VOCs. Therefore, the SC / PRI Work Plan, to which this QAPP was prepared in support of, has been specifically designed to evaluate for the presence of VOCs in soil vapor, soil and groundwater matrices.

3.1 Field Documentation and Records

Project-dedicated field notebooks will be initiated at the start of on-site work and will be maintained with information pertinent to site activities. The field notebook will include the following daily information for all site activities:

- Date;
- Meteorological conditions (temperature, wind, precipitation, etc.);
- Site conditions (e.g., dry, damp, dusty, etc.);
- Identification of crew members and other personnel (e.g., agency or site owner) present;
- Description of field activities;
- Location(s) where work is performed;
- Problems encountered and corrective actions taken;
- Records of field measurements or descriptions recorded; and,
- Notice of modifications to the scope of work.

During soil vapor sampling, field samplers will include the following information:

- Sampling point locations and PID results;
- Information about sample collection (depth, odors, etc.); and,
- COC information.

During soil sampling activities, field samplers will add the following:

- Sampling point locations and photo-ionization detector (PID) results;
- Description of soil in sample (color, odor, consistency, etc.);
- Information about sample collection;

- COC information;
- Field equipment calibration; and,
- Equipment decontamination.

During sampling of wells, field samplers will add the following:

- Sampling point locations and test results such as pH, conductance, etc.;
- Information about sample collection;
- COC information, and,
- Field equipment calibration.

Each sample must have a COC record that includes sample identification number, date and time of collection, place of collection, environmental matrix, sample container, preservation method, signature of the collector, and signature and dates of persons involved in the transportation and handling of the sample. Further, for tasks requiring repetitive note taking such as groundwater sampling, pre-printed field forms will be utilized to ensure that the appropriate data are collected and recorded.

Final reports including technical review documentation, raw data, data collection sheets (as specified above), calculations, instrument calibration records and QA information will be maintained.

As discussed in the SC / PRI Work Plan, soil vapors will be collected and analyzed utilizing the GORE methodology to evaluate soil conditions underlying the building envelope, as well as adjacent to the building. The purpose of the soil vapor sampling / analysis is to evaluate for the presence of undocumented impacted soils not associated with previously-identified on-site AOCs.[1] Groundwater and soil samples will be collected from multiple depths from across the Subject Property to evaluate unsaturated soil / aquifer contaminant conditions associated with specific AOCs from across the Subject Property.

[1] *It should be noted that the location of soil vapor sample points was selected to allow mathematical modeling of data as well as to identify specific, potential AOCs. Therefore, it is critical that locations selected contain non-contaminated locations as well as possibly contaminated locations to complete statistically significant, data evaluation of soil vapor data. This approach also helps to bound areas of potential concern to better focus subsequent soil and groundwater sampling events.*

3.2 Sampling Methods

Environmental sampling will be conducted in general accordance with the appropriate techniques presented in the following guidance documents:

- Sampling Guidelines and Protocols, NYSDEC, Division of Water, March 1991;
- Compendium of Superfund Field Operations Methods, US EPA, December 1987 (EPA/540/P-87/001);
- RCRA Ground-Water Monitoring: Draft Technical Guidance, US EPA, November 1992 (EPA/530-R-93-001);
- Soil Sampling Quality Assurance User's Guide (Second Edition), US EPA, March 1989, (EPA/600/8-89/046);
- USEPA Region II CERCLA Quality Assurance Manual, Revision 1, USEPA Region II, October 1989;
- NYSDEC Draft DER-10 – Technical Guidance for Site Investigation and Remediation, December 25, 2002; and,
- NYSDEC New York State Brownfield Cleanup Program Development of Soil Cleanup Objectives Technical Support Document, September 2006.

Samples must be collected using equipment that has been properly decontaminated and procedures appropriate to site-specific factors including the matrix, the parameters to be analyzed, and the DQOs of specific tasks.

The volume of the sample collected must be sufficient to perform the analyses requested, as well as the QA / QC requirements. Sample volumes, container types, and preservation techniques will also be confirmed with the approved laboratory.

Before leaving the facility, the sampler will:

- Check all paperwork for accuracy and completeness;
- Match the physical samples with the associated paper work. The sampler will check for proper samples in the correct containers and that the field number on the samples correspond with the numbers on the completed COCs;
- Verify that samples are properly stored and secure for transport;
- Clean and package all non-disposable equipment;

- Make sure the items on the sample tags, request forms, COC record, and log book match;
- Bag all disposable items that need to be discarded; and,
- Ensure that all sample containers are free of any debris.

To avoid cross contamination, sampling equipment (defined as any piece of equipment which may contact a sample) will be decontaminated according to the following procedures outlined below.

Decontamination of non-dedicated reusable equipment will include scrubbing/washing with a laboratory-grade detergent (e.g., alconox) to remove visible contamination, followed by potable (tap) water and analyte-free water rinses. Tap water may be used from any treated municipal water system; the use of an untreated potable water supply is not an acceptable substitute. Equipment should be allowed to dry prior to use. Spent decontamination liquids will be discharged to the ground surface adjacent to the sampling points where they were generated.

Disposable sampling equipment (e.g., bailers, tubing, gloves, etc.) will be acquired from sources with strict factory-decontamination protocols; therefore, field contamination will not be required.

3.3 Sample Handling and Custody

In order for analytical results to be defensible, a COC must be established for all samples collected which demonstrates that samples have not been tampered with during collection, transfer, storage or analysis. This requires custody of the samples to be documented from the time the samples are collected through final analyses. A sample is considered as under custody if:

- It is in the person's (e.g., field sampler, laboratory technician, etc.) possession;
- It is in the person's view, after being in the person's possession;
- It was in the person's possession and then it was locked up or placed in a sealed container to prevent tampering; and / or,
- It is in a designated secure area.

3.3.1 Coordination with the Analytical Laboratory

The sampler will contact a State-approved laboratory before sampling to verify that the lab is capable of conducting the sample analysis within the holding time specified in SW 846, 3rd

edition. Further, the sampler will coordinate with the soil vapor analytical laboratory for the same purpose.

3.3.2 Preservation and Shipping Procedures

Samples for soil and groundwater VOC analyses must be placed on ice immediately after collection to minimize the loss of volatiles. Once the sampling is complete and the sampler has left the site, chain-of-custody must be maintained and properly documented. Preferably, soil and groundwater samples should be transported directly to the laboratory by the sampler or a laboratory representative. When shipping is required, the samples will be placed in a container acceptable to both the laboratory and the carrier.

The GORE sample collection methodology selected for the soil vapor evaluation portion of the SC / PRI Work Plan does not require the samples to be placed on ice. Rather, a GORE Module is removed from the pilot hole, placed back in its original container and shipped via overnight carrier to the laboratory for analyses.

3.4 Analytical Methodologies

The matrices to be sampled and analyzed as part of the SC / PRI Work Plan include soil vapor, soil and groundwater. The following provides summaries of the analytical methodologies and associated laboratories which will conduct the analyses. A list of the samples and analytical methodologies per matrix is included in **Table 3-1**. **Table 3-2** provides a summary of container, preservation and holding time requirements. The sampling locations proposed in the SC / PRI Work Plan are included in **Figures 3-1 and 3-2**.

Analytical method selection is based on whether the method provides comparable, representative, complete, precise, and accurate data for the sample matrix and the range of expected values for constituents for which the samples are being analyzed.

3.4.1 Soil Vapor Analysis

The soil vapor samples collected as part of this work will be analyzed by GORE for TCL VOCs and Freon 113 by modified EPA Method 8260. This is considered a high-quality, field-screening technique and is being conducted for the purposes of evaluating for the presence of VOC-impacted soils. It is not designed to address the issue of soil vapor intrusion. Therefore, these data will not be analyzed in strict accordance with NYSDEC Analytical Services Protocols (ASP). It should be noted that this is a sample collection / analyses technique which is utilized across the Country and in New York. In fact, the NYSDEC Hazardous Waste Group utilizes the GORE sampling and analytical methodologies as part of many NYSDEC-led investigations. Information regarding this sampling and analyses methodologies is included in ***Appendix B***.

3.4.2 Soil and Groundwater Analyses

The soil and groundwater samples collected as part of this work will be analyzed by a New York State Department of Health (NYSDOH) Environmental Laboratory Accreditation Program (ELAP)-certified laboratory (with appropriate chain-of-custody) for NYSDEC target compound list (TCL) VOCs, Freon 113 and ten (10) tentatively-identified compounds (TICs) by EPA Method 8260.[2] In order to evaluate whether former activities conducted at the Subject Property could potentially have resulted in the release(s) of other contaminants, a selected number of the soil samples will also be analyzed for TCL semi-volatile organic compounds (SVOCs) plus 20 TICs by EPA Method 8270, Target Analyte List (TAL) metals by the EPA 6010 / 7471 Series, TCL pesticides by EPA Method 8081 and TCL polychlorinated biphenyls (PCBs) by EPA Method 8082. The rationale for selecting the soil samples for the additional analyses is included in ***Table 2-2*** of the SC / PRI Work Plan

The samples will be analyzed in accordance with NYSDEC ASP Category B laboratory data deliverable format. Information regarding the analytical methodologies, including the specific analyte lists are included in ***Appendix C***.

3.5 Quality Control Protocols

This section provides details with respect to both field and laboratory QC protocols.

3.5.1 Field Quality Control Samples

Field QC samples will be submitted to a NYSDOH-approved laboratory as appropriate and as often as reasonably practical during field investigations. The PM, working in coordination with the QAO and the laboratory, will select the appropriate field-originated QC samples. The approach outlined in this document for QC samples represent standard SOPs and may need to be change or varied under some circumstances. The following QC samples will be utilized in support of the soil and groundwater sampling program to support the preparation of ASP Category B deliverables.

3.5.1.1 Duplicates

Field duplicate samples are used to assess the variability of a matrix at a specific sampling point and to assess the reproducibility of the sampling method. For soil samples, these samples are

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[2] In order to minimize for the potential of reporting high-biased data due to high turbidity matrix conditions, the grab groundwater samples collected during Geoprobe sampling will be lab filtered using a 0.45 micron filter. Laboratory filtering will not be performed for groundwater samples collected from permanent monitoring wells that allow for proper well development and purging prior to sample collection.

separate aliquots of the same sample; prior to dividing the sample into "sample" and "duplicate" aliquots. Aqueous field duplicate samples are second samples collected from the same location, at the same time, in the same manner as the first, and placed into a separate container (technically, these are co-located samples) and are submitted to the laboratory with a fictitious identifier. Each duplicate sample will be analyzed for the same parameters as the original sample collected that day. The blind field duplicate Relative Percent Difference (RPD) objective will be +50% percent RPD. Field duplicates will be collected at a frequency of 1 per 20 environmental samples for both matrices (aqueous and non-aqueous) and all test parameters.

3.5.1.2 Trip Blanks

Trip blanks are prepared in the laboratory for TCL VOC analysis only prior to sampling by filling the appropriate container with distilled / deionized water and are utilized to determine if VOC cross contamination occurs during sample shipment. The trip blank is transported to the field, handled in the same manner as the other VOC samples, and submitted to the lab with the other samples for analysis. There will be a minimum of one trip blank per shipment containing samples for TCL VOC analyses. Criteria for acceptance are below detection limits of any analyte being tested for at the site.

3.5.1.3 Rinsate Blanks

Rinsate blanks are prepared in the field by collecting distilled / deionized water in sample containers after the water has been used to rinse decontaminated equipment (e.g., bailers, macrocore samplers, etc.) prior to sampling. One rinsate blank will be collected for each type of sampling apparatus during sampling events. Criteria for acceptance are below detection limits of any analyte being tested for at the site.

3.5.1.4 Spiked Samples:

Matrix spikes and matrix spike duplicates (MS / MSD) blanks are site-specific samples which are “spiked” in the laboratory with a known concentration of a known chemical. The laboratory then analyzes the samples for the spiked chemical to evaluate whether matrix interferences are either resulting on a loss of the spiked chemical (i.e., results in low-biased data) or an increase in the spiked chemical (i.e., results in high-biased data). MS / MSD will be analyzed at a frequency of one (pair) for every 20 samples.

3.5.2 Laboratory QC Procedures

Internal QC procedures for sample analysis are the responsibility of the laboratory and are dependent upon the project DQOs and the level of NYSDEC ASP being employed. These procedures include the use of duplicate analysis, spikes, calibration standards, internal standards, blanks, QC charts, standard reference materials, reagent checks, and sample splits.

All of the soil and groundwater samples will be analyzed by a NYSDOH-approved laboratory in accordance with NYSDEC ASP Category B protocols.

3.5.2.1 GORE Laboratory QC Procedures

As discussed above, the soil vapor samples, which represent high-quality field screening data, will be analyzed by GORE for TCL VOCs and Freon 113. Analytical instrumentation consists of gas chromatographs and mass selective detectors, as well as automated thermal desorption units. Sample preparation involves cutting the tip off the bottom of the GORE Module which has been exposed to soil vapors at the site and transferring an exposed sorber to a thermal desorption tube for analysis. No further sample preparation is required. The replicate samples are retained in the laboratory for approximately two weeks after the initial analysis.

Before each sequence, two (2) instrument blanks, a sorber containing 5 micrograms (ug) of bromofluorobenzene (BFB), and a method blank are analyzed. The BFB mass spectra must meet the criteria set forth in the method before GORE Modules are analyzed. A method blank and a sorber containing BFB are also analyzed after every 30 Modules and/or trip blanks. Standards containing the selected target compounds at five (5) calibration levels are analyzed at the beginning of each sequence. The criterion for each target compound is less than 25% relative standard deviation (RSD). If this criterion is not met for any target compound, the laboratory analyst has the option of generating second or third order standard curves, as appropriate. A second source reference standard, at a level of 10 ug per target compound, is analyzed after every ten (10) modules and/or trip blanks, and at the end of the sequence. To minimize handling of the field exposed modules, no surrogates or internal standards are used. Positive identification of target compounds is determined by: 1) the presence of the target ion and at least two secondary ions; 2) retention time versus reference standard; and, 3) the analyst's judgment.

3.5.2.2 ELAP-Certified Laboratory QC Procedures

The following provides a brief summary of the USEPA analytical methodologies for the project-specific parameters. Full details are provided in **Appendix C**.

TCL VOCs

The USEPA Method 8260 used to analyze for TCL VOCs and Freon 113 employs the technique of purge and trap, coupled with a gas chromatograph/mass spectrometer analysis. An aliquot of sample, usually 5 milliliters (ml) or 25 ml of water, 5 grams (g) of soil for low level soil method, and 5 g of soil collected with methanol and extracted for medium-level soil method, is purged in a gas tight chamber with UHP-grade helium to remove the VOCs. The vapor is swept through a sorbent column where the VOCs are trapped. Next the sorbent trap is heated and back flushed, thereby desorbing the VOCs onto the analytical column within the gas chromatograph. The

fused silica capillary column is then temperature programmed to separate the volatiles prior to detection by the mass spectrometer.

TCL SVOCs

The USEPA Method 8270 used to analyze for TCL SVOCs employs the technique of solvent extraction, followed by gas chromatograph/mass spectrometer analysis. An aliquot of sample extract is injected onto a gas chromatograph. The fused silica capillary column is then temperature programmed to separate the semi-volatiles organics prior to detection by the mass spectrometer. This method is based on SW-846 Method 8270C and Method 8000B. The concentration of the solution used for the initial demonstration of precision and accuracy varies from that listed in the method. The lab uses a concentration of 40 ug/l, and a of 100 ug/l. The lab has generated limits based on in house data. These limits are updated on an as needed basis.

TCL Pesticides

The USEPA Method 8081 used to analyze for TCL pesticides employs the gas chromatographic procedure for the detection of organochlorine pesticides. Samples are extracted using the proper extraction technique. The extracts are analyzed by gas chromatography with an electron capture detector.

TCL PCBs

The USEPA Method 8082 used to analyze for TCL PCBs employs the gas chromatographic procedure for the detection of PCBs. Samples are extracted using the proper extraction technique. The extracts are analyzed by gas chromatography with an electron capture detector.

TAL Metals

The USEPA requires the use of a variety of methods to analyze for TAL metals. Soil samples are first extracted by EPA Method 3050 in which a soil sample digested in nitric acid and hydrogen peroxide. For a wipe, the whole wipe sample is digested in nitric acid and hydrogen peroxide. The digestate is then refluxed with hydrochloric acid and brought to 50 ml final volume with deionized water. Water samples are digested utilizing a mixture of HNO_3 and material to be analyzed is heated in a 100 ml tube until the volume is reduced to 5-10 ml. After cooling, the digestion is refluxed with HCl and brought to 50 ml. The samples are then analyzed either by EPA Method 6010 via Inductively coupled plasma-atomic emission spectrometry to determine trace elements, including metals; or by EPA Method 6020 via inductively coupled plasma-mass spectrometry to the determination of sub part per billion concentrations of a large number of elements. Mercury is analyzed in soils and groundwater by USEPA Methods 7041 (i.e., by cold-vapor atomic absorption for soils, sediments, bottom deposits and sludge–type

materials, the samples are digested and then analyzed for mercury by reduction with stannous chloride, which is added in-line by a mercury analyzer) and 74710 (i.e., by cold-vapor atomic absorption, samples are digested and then analyzed for mercury by reduction with stannous chloride, which is added in-line by a mercury analyzer), respectively.

3.5.3 Performance, System Audits, and Corrective Action

The QAO, or designee, will monitor and audit performance of the QA procedure to ensure that all sampling activities are performed in accordance with approved QA procedures. Performance audits by the staff-sampling activities will be conducted periodically to evaluate whether samplers are adhering to the QA / QC controls identified herein, including the proper execution and use of sample identification, sample control, COC procedures, documentation and sampling procedures.

Analytical results meeting DQOs of completeness, accuracy, and precision will be accepted. If QC samples are outside acceptance criteria, they will be evaluated by including field audit sets with internal laboratory QC samples. If combined sets meet acceptance criteria the data will be accepted. All analyzed data still not meeting acceptance criteria will be referred for corrective action. The corrective action may entail reanalysis of the sample(s) or QC, recalibration and reanalysis of the sample batch, re-prepping and analyzing the sample batch.

3.6 Instrument / Equipment Testing, Inspection, and Maintenance

A PID will be used during on-site sampling activities to screen ambient indoor air and soils for the presence of VOCs. Any additional instrument / equipment needs will be determined on site. Preventative maintenance tasks and schedules recommended by the manufacturers will be conducted and followed for all field instrumentation. Records of preventative maintenance will be maintained. The QAO and the field staff will ensure that the prescribed maintenance on field instrumentation is conducted.

Preventative maintenance procedures for laboratory equipment are the responsibility of the laboratories and must be documented in logbooks that will be monitored periodically.

3.7 Instrument / Equipment Calibration and Frequency

Laboratory calibrations will be conducted according to the 1995 Revised NYSDEC ASP Superfund Contract Laboratory Program and DER-10 for each parameter or group of similar parameters, and maintained following professional judgment and the manufacturer's specifications. Equipment used for field measurements will be calibrated according to manufacturer's specifications. The field staff is responsible to record calibration procedures for each sampling event.

3.8 Inspection / Acceptance of Supplies and Consumables

Sample containers and all other sampling supplies and consumables will be inspected by the sampler prior to use for any defects. Any defective supplies or consumables will be discarded or returned to the laboratory and acceptable replacements will be obtained.

3.9 Data Management

ASP Category B deliverable format for all soil and groundwater analytical results will be obtained from the laboratory and maintained along with daily logs, calibration records, etc. Any data which is transferred into reports and/or spreadsheets will be reviewed by the QAO or designee to ensure the accuracy of data transcription.

4.0 ASSESSMENT / OVERSIGHT

4.1 Assessments and Response Actions

Data will be evaluated using accuracy, precision, and completeness criteria as detailed in **Section 1.2**. Approved laboratories will report only data that meet those criteria.

If the samples meet the criteria above, the reported data will be accepted. If not, the laboratory QAO will be consulted to evaluate which lab QC samples were included. These samples will be included with the field audit set and reevaluated. If the combined set meets the acceptance criteria, the reported data will be accepted. If not, the data from analyzing the sample set will be used as a basis for a corrective action referral.

4.2 Reports to Management

Site-specific QA / QC information will be included in the appropriate facility files from each sampling event. The final summary of reported data from the laboratory will reflect all laboratory QA / QC measures taken. If further reporting and clarification is necessary, the laboratory QA chemist will prepare a report detailing recommendations and submit the report with the data to the project director and the QAO. These individuals will review the QA recommendations and take necessary corrective actions.

5.0 DATA VALIDATION AND USABILITY ELEMENTS

5.1 Data Review, Verification and Validation

Data validation procedures will focus on determining if the data were generated according to USEPA and NYSDEC protocols. Specifically, the QAO will audit sampling, calibration, field measurement, field logging and COC procedures. Where possible, generated data sets will be compared with previous data sets to evaluate consistency. Any data generated outside standard protocols will be either rejected or identified with the inconsistency.

All data review, validation, and verification requirements, other than the ones described above, are the responsibility of the approved laboratory.

5.2 Data Summary Usability Report

In accordance with DER-10 requirements, the QAO will prepare a Data Summary Usability Report (DUSR) for the project.[3] The DUSR evaluation will be conducted to:

- Ensure that all holding times were met;
- Confirm that all the QC data (e.g., blanks, instrument tunings, calibration standards, calibration verifications, surrogate recoveries, spike recoveries, replicate analyses, laboratory controls and sample data) fall within the protocol-required limits and specifications;
- Confirm that all of the data have been generated using established and agreed upon analytical protocols;
- Confirm an evaluation of the raw data;
- Confirm the results provided in the data summary sheets and quality control verification forms; and,
- Confirm that correct data qualifiers have been used.

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[3] *According to DER-1, the purpose of the DUSR process is to provide a thorough evaluation of analytical data without the costly and time consuming process of third party data validation. The primary objective of a DUSR is to determine whether or not the data, as presented, meets the site / project-specific criteria for data quality and data use. If data validation is found to be necessary, this can be carried out at a later date on the same data package used for the development of the DUSR as all of the soil and groundwater samples will be collected and analyzed in accordance with NYSDEC ASP Category B protocols.*

5.3 Reconciliation with User Requirements

If a QC audit results in detection of unacceptable conditions or data, the PM and the QAO will be responsible for developing and initiating corrective action(s), as warranted. If the unacceptable conditions indicate a project difficulty or if corrective action is likely to require expertise not immediately available to the project team, the appropriate resource will be notified.

Corrective action may include:

- Reanalyzing the samples, if holding time criteria permit;
- Re-sampling and analysis of the samples;
- Evaluating and amending sampling and analytical procedures; and,
- Acceptance of data, with an acknowledgment of the level of uncertainty surrounding the analytical results.

The selected corrective action will depend on how critical the samples are and the range of the reported values. If reported data is not adequate to determine whether contamination is present or not, the samples will be reanalyzed or retaken.

TABLES

Table 3-1
One Commerce Park Site
Labratory Analyes and QA / QC Requirments Matrix

Sample				Analyte					QA/QC Samples					
Type/ Methodology	**Identification Number**	**Matrix**	**Depth Interval**	**TCL VOCs plus Freon 113**	**TCL SVOCs**	**TCL Pesticides**	**TCL PCBs**	**TAL Metals**	**TB**	**RB**	**BD**	**MS**	**MSD**	**Total QA/QC**
Soil Vapor	SV-1	Soil Vapor	Shallow	1										
	SV-2	Soil Vapor	Shallow	1										
	SV-3	Soil Vapor	Shallow	1										
	SV-4	Soil Vapor	Shallow	1										
	SV-5	Soil Vapor	Shallow	1										
	SV-6	Soil Vapor	Shallow	1										
	SV-7	Soil Vapor	Shallow	1										
	SV-8	Soil Vapor	Shallow	1										
	SV-9	Soil Vapor	Shallow	1										
	SV-10	Soil Vapor	Shallow	1										
	SV-11	Soil Vapor	Shallow	1										
	SV-12	Soil Vapor	Shallow	1										
	SV-13	Soil Vapor	Shallow	1										
	SV-14	Soil Vapor	Shallow	1										
	SV-15	Soil Vapor	Shallow	1										
	SV-16	Soil Vapor	Shallow	1										
	Total	Soil Vapor	Shallow	**16**	**0**	**0**	**0**	**0**	**0**	**0**	**0**	**0**	**0**	**0**
Direct Push Soil and Groundwater	SB/GW-1	Soil	Shallow	1										
			Deep	1										
		Groundwater	Shallow	1										
			Intermediate	1										
			Deep	1										
	SB/GW-2	Soil	Shallow	1	1	1	1	1						
			Deep	1	1	1	1	1						
		Groundwater	Shallow	1										
			Intermediate	1										
			Deep	1										
	SB/GW-3	Soil	Shallow	1										
			Deep	1										
		Groundwater	Shallow	1										
			Intermediate	1										
			Deep	1										
	SB/GW-4	Soil	Shallow	1	1	1	1	1						
			Deep	1										
		Groundwater	Shallow	1										
			Intermediate	1										
			Deep	1										
	SB/GW-5	Soil	Shallow	1										
			Deep	1	1	1	1	1						
		Groundwater	Shallow	1										
			Intermediate	1										
	SB/GW-6	Soil	Shallow	1	1	1	1	1						
			Deep	1										
		Groundwater	Shallow	1										
			Intermediate	1										
			Deep	1										
	SB/GW-7	Soil	Shallow	1										
			Deep	1	1	1	1	1						
		Groundwater	Shallow	1										
			Intermediate	1										
			Deep	1										
	SB/GW-8	Soil	Shallow	1	1	1	1	1						
			Deep	1										
		Groundwater	Shallow	1										
			Intermediate	1										
			Deep	1										
			Total Soil (All Analytes):	**16**	**7**	**7**	**7**	**7**		**1**	**1**	**1**	**1**	**4**
			Total Groundwater (TCL VOCs Only):	**23**	**0**	**0**	**0**	**0**	**3**	**1**	**2**	**2**	**2**	**8**
Permanent Groundwater Monitoring Wells / Piezometers	MW-1	Groundwater	Shallow	1	1	1	1	1						
			Intermediate	1	1	1	1	1						
			Deep	1										
	MW-2	Groundwater	Shallow	1	1	1	1	1						
			Intermediate	1	1	1	1	1						
			Deep	1	1	1	1	1						
	MW-3	Groundwater	Shallow	1										
			Intermediate	1										
			Deep	1										
	P-1	Groundwater	Shallow	1										
	P-2	Groundwater	Shallow	1										
			Total Groundwater:	**11**	**5**	**5**	**5**	**5**	**2**	**1**	**1**	**1**	**1**	

Notes:
SB/GW-1 - Proposed sampling location for Geoprobe multi-depth soil and groundwater samples.
MW-1 - Proposed location for permanent multi-cluster groundwater monitoring well.
P-1 - Proposed location of permanent shallow groundwater piezometer.
Trip Blank - One per sample cooler
RB - Rinsate / Field Blank - One per sample equipment type
BD - Blind Duplicate one in 20
MS / MSD - Matrix Spike / Matrix Spike Duplicate one in 20

Table 3-2
One Commerce Park Site
Summary of Container, Preservation and Holding Time Requirements

Matrix	Analytical Parameter	USEPA Method	Sample Container		Sample Preservative	Holding Time
			Type	Number		
Soil Vapor	TCL VOCs and Freon 113	modified 8260	GORE Module	1	NA	NA
Soil	TCL VOCs, Freon 113 and 10 TICs	8260B	4 oz glass jay w/ septum	1	4^{o} C	14 days
	TCL SVOCs and 20 TICs	8270	8 oz glass jar	1	4^{o} C	5 / 35 days*
	TCL PCBs	8082	8 oz glass jar	1	4^{o} C	7 / 40 days
	TCL Pesticides	8081	8 oz glass jar	1	4^{o} C	7 / 40 days
	TAL Metals	6010 / 6020 / 7471	8 oz glass jar	1	4^{o} C	6 months
Groundwater	TCL VOCs, Freon 113 and 10 TICs	8260B	40 ml glass vial w/ septum	2	4^{o} C and HCl	14 days
	TCL SVOCs and 20 TICs	8270	One l amber glass	1	4^{o} C	5 / 35 days
	TCL PCBs	8082	One l amber glass	1	4^{o} C, pH 5 - 8	7 / 40 days
	TCL Pesticides	8081	One l amber glass	1	4^{o} C, pH 5 - 8	7 / 40 days
	TAL Metals	6010 / 6020 / 7470	One 500 ml plastic	1	HNO_3 to pH <2	6 months / 28 days for Mercury

Notes:
NA - not applicable.
* 5 / 35 - days to extract / days to analyze extract.

FIGURES

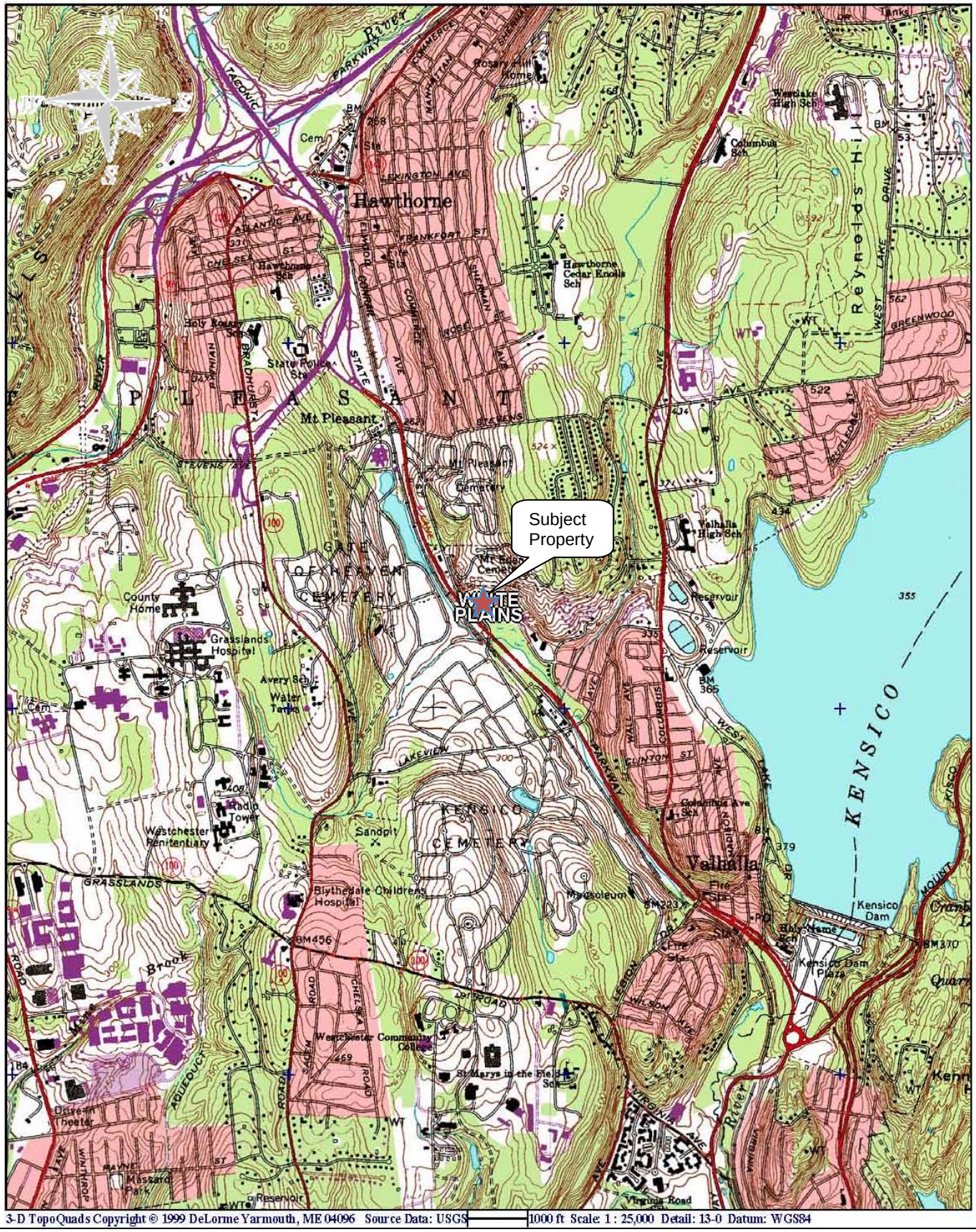


Figure 1-1
One Commerce Park Site
Site Location Map

Client:	DP 16, LLC
Project No.:	85144.003
Project Location:	Valhalla, NY
Date:	June 5, 2009

Figure 2-1
Project Organization Chart
SC / PRI Team Organization Chart

New York State Department of Environmental Conservation

Quality Assurance Officer
Project Director
Daniel J. Smith, P.E.

- **Principal-in-Charge**
- **Quality Assurance / Quality Control**
- **Data Usability Summary Report**

Project Manager
Richard J. Baldwin, P.G., C.P.G.

- **In-Charge of Field Implementation**
- **Ensure Appropriate Data Collection**
- **Report Preparation**
- **NYSDEC Coordination**

Field Team Leader
Thomas Stolworthy

- **Implement SC / RI Work Plan**
- **Ensure Quality Data Acquisition**
- **Contractor Coordination**

Project Staff
Robert Bennett

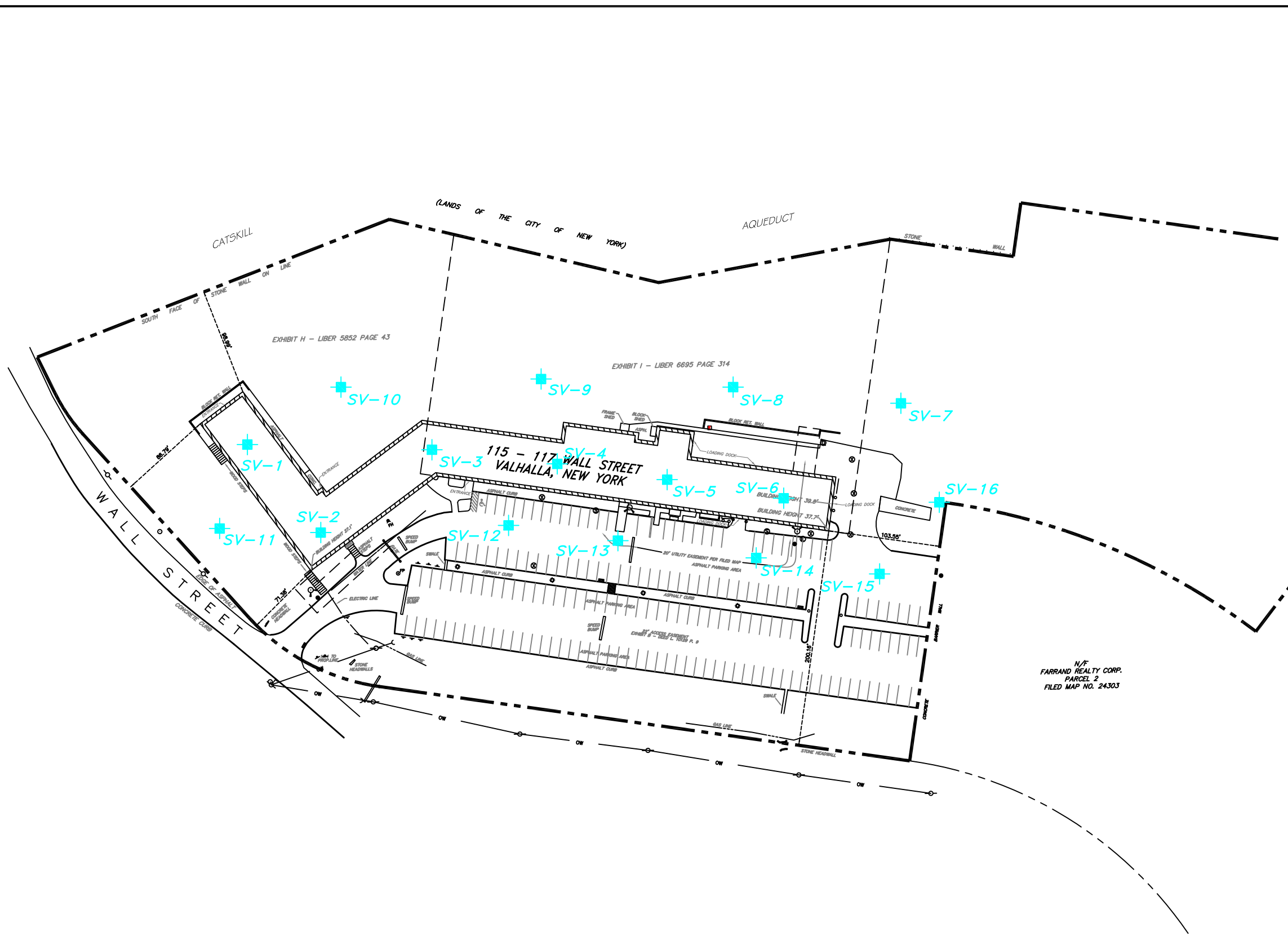
- **Oversee Contractors**
- **Collection of Samples**
- **Data Summary Tables**

Project Staff
Greg Mendez-Chicas

- **Oversee Contractors**
- **Collection of Samples**
- **Field Data Tabulation**

Project Support
Susan Russo

- **Contractor Coordination**
- **Data Management**
- **Data Table QA / QC**



LEGEND

- PROPOSED SOIL VAPOR SAMPLE LOCATION
- UTILITY POLE
- OW — OVERHEAD WIRE
- PROPERTY LINE

SOURCE: DP ALTA SURVEY CAD VERSION
DATE: MAY 22, 2008

DRAWING FILE LOCATION: P:\ACTIVE PROJECTS.../AIG/SIVANT X-REF:

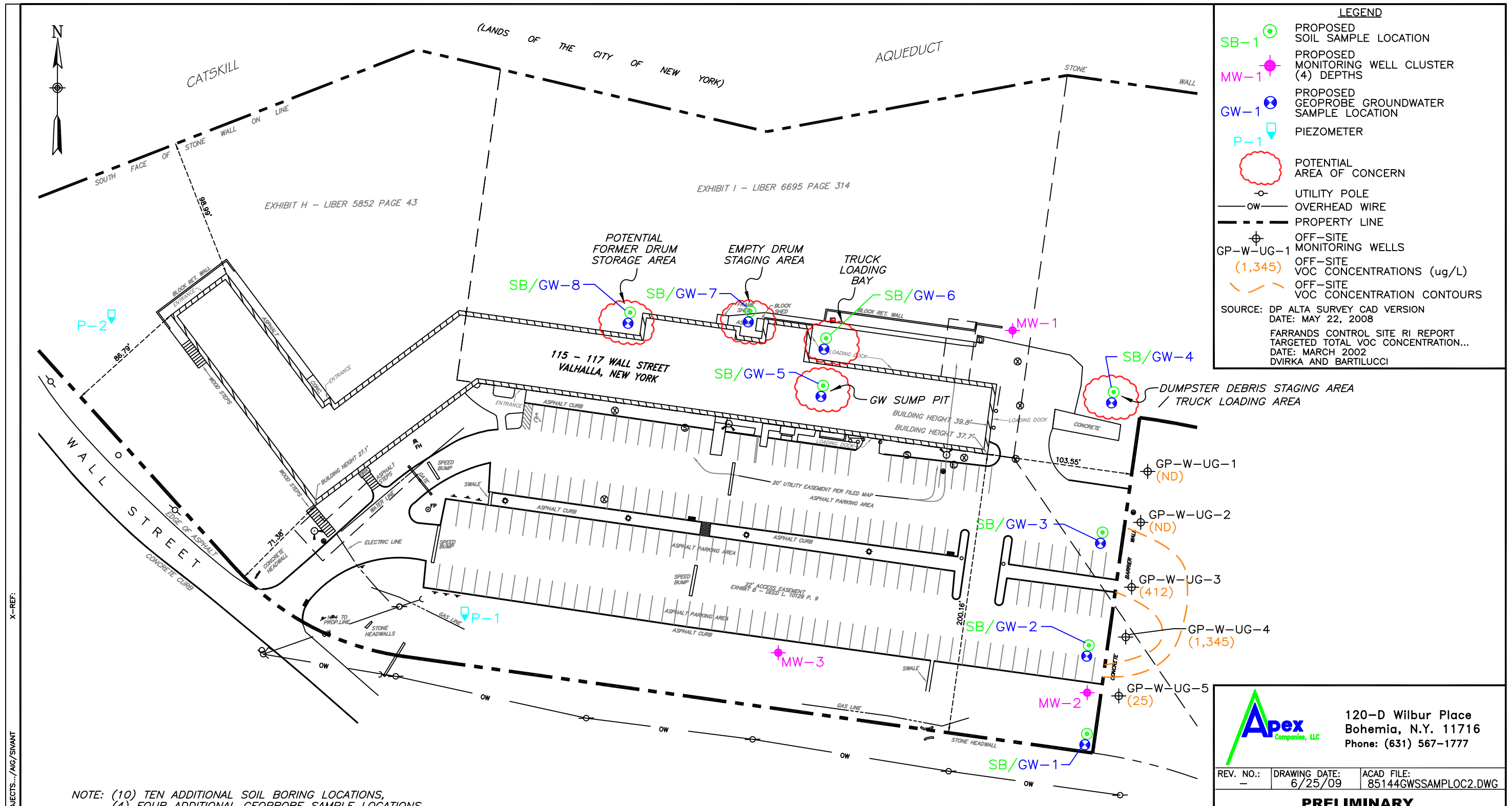
Apex Companies, LLC
120–D Wilbur Place
Bohemia, N.Y. 11716
Phone: (631) 567–1777

REV. NO.:	DRAWING DATE:	ACAD FILE:
–	7/16/09	85144PROPSVSL–2

PROPOSED SOIL VAPOR SAMPLE LOCATIONS

CLIENT:	PM:
SP&R	RB
LOCATION: 115–117 WALL STREET VALHALLA, NEW YORK	PE/RG:

DESIGNED:	DETAILED:	PROJECT NO.:	FIGURE:
SEE SOURCE	TS	85144	3-1



LEGEND

- SB–1 — PROPOSED SOIL SAMPLE LOCATION
- MW–1 — PROPOSED MONITORING WELL CLUSTER (4) DEPTHS
- GW–1 — PROPOSED GEOPROBE GROUNDWATER SAMPLE LOCATION
- P–1 — PIEZOMETER
- POTENTIAL AREA OF CONCERN
- UTILITY POLE
- OW — OVERHEAD WIRE
- PROPERTY LINE
- GP–W–UG–1 — OFF–SITE MONITORING WELLS
- (1,345) — OFF–SITE VOC CONCENTRATIONS (ug/L)
- OFF–SITE VOC CONCENTRATION CONTOURS

SOURCE: DP ALTA SURVEY CAD VERSION
DATE: MAY 22, 2008

FARRANDS CONTROL SITE RI REPORT
TARGETED TOTAL VOC CONCENTRATION...
DATE: MARCH 2002
DVIRKA AND BARTILUCCI

NOTE: (10) TEN ADDITIONAL SOIL BORING LOCATIONS, (4) FOUR ADDITIONAL GEOPROBE SAMPLE LOCATIONS, AND (1) ONE ADDITIONAL INTERIOR SAMPLE LOCATION TO BE FINALIZED AFTER SOIL VAPOR STUDIES.

Apex Companies, LLC
120–D Wilbur Place
Bohemia, N.Y. 11716
Phone: (631) 567–1777

REV. NO.:	DRAWING DATE:	ACAD FILE:
–	6/25/09	85144GWSSAMPLOC2.DWG

PRELIMINARY SOIL AND GROUNDWATER SAMPLE LOCATIONS

CLIENT: SP&R			PM: RB
LOCATION: 115–117 WALL STREET VALHALLA, NEW YORK			PE/RG:
DESIGNED: SEE SOURCE	DETAILED: TS	PROJECT NO.: 85144	FIGURE: 3-2

X–REF:

DRAWING FILE LOCATION: P:\ACTIVE PROJECTS.../AIG/SIVANT

APPENDICES

Appendix A

Resumes of Key SC / RI Personnel

Richard J. Baldwin, Baldwin, C.P.G., P.G
Apex Companies, LLC
Senior Project Director

Mr. Baldwin is a hydrogeologist/environmental scientist with over twenty years of experience in the fields of environmental consulting, hydrogeology and geology with particular experience in conducting and supervising environmental investigations and remedial actions at industrial, private, Federal and publicly-owned facilities and sites. Mr. Baldwin has extensive experience in evaluating and remediating gasoline and fuel oil releases, many of which included the contaminant methyl tertiary-butyl ether (MTBE). Additionally, Mr. Baldwin has experience in evaluating potential environmental impacts of projects including golf courses, housing developments, senior housing, schools and retail shopping centers. For the last several years, Mr. Baldwin's work has focused primarily on sites and facilities located in the Long Island, New York City and Upstate New York areas. He has extensive knowledge and experience pertaining to Long Island's federally-designated sole-source drinking water aquifer system.

Education

- *Graduate Course Work, San Jose State University, 1985-1988*
- *BA Geology, San Francisco State University, 1982*

Professional Registrations

- *Professional Geologist, PG-000552-G, Commonwealth of Pennsylvania*
- *Certified Professional Geologist, CPG #9158, Amer.Inst. of Prof. Geologists*
- *OSHA Certification, 40-hour Health and Safety Training at Hazardous Waste Sites*
- *OSHA Certification, 8-hou Refresher Health and Safety Training at Hazardous Waste Sites*
- *OSHA Certification, 8-hour Management Training*
- *OSHA Certification, 8-hour Radiation Safety Training*

Continuing Education

- *Princeton Groundwater Hydrogeology and Pollution course*
- *Environmental Law and Regulations Course, U.C. Berkeley Extension*
- *NGWA MODFLOW and MODPATH Modeling Course*
- *NGWA Visual MODFLOW Modeling Course*

General Project Experience

Mr. Baldwin has extensive experience in the selection, design, installation and maintenance of a wide range of soil and groundwater remediation systems. Remedial systems have included both active and passive free-product recovery, traditional groundwater pump and treat, soil-vapor extraction, air sparging, bioventing, bioremediation, excavation, impacted-soil management and natural attenuation. MTBE and other petroleum-related volatile organic compounds (VOCs) and semi-volatile organic compounds (SVOCs) were the contaminants of concern in many of these project sites.

Mr. Baldwin has been involved in hundreds of subsurface soil and groundwater investigations ranging from Phase I & II Environmental Site Assessments (ESAs) to Remedial Investigations. Investigation and delineation techniques have included soil borings, groundwater monitoring well networks, Hydropunch / GeoProbe sampling, surface and bore-hole geophysical methods, soil-gas surveys, aquifer testing, surface water and sediment sampling, waste characterization (soils piles, drums, USTs, aboveground storage tanks (ASTs), landfills, etc), test pits, and computer fate and transport modeling. Materials investigated have included petroleum products (heating/fuel oil and gasoline), PCB oils, coal tar, heavy metals, chlorinated solvents, explosives, pesticides, herbicides and buried medical waste.

Mr. Baldwin has evaluated the potential environmental impacts of proposed projects including golf courses, housing developments, senior housing, schools, automobile repair facilities and retail shopping centers. The potential impacts included those to groundwater quality from herbicide/pesticide application, disposal of sanitary waste and school laboratory waste and the impacts to soil quality from handling and disposal of hazardous materials, leaking petroleum underground storage tanks (USTs), historic disposal of hazardous waste and pesticide/herbicide application. These impacts were evaluated through a variety of means including the collection and analysis of soil and groundwater samples, geo- and organic-chemistry modeling, groundwater fate and transport modeling and basic research of materials, their uses and their potential migration pathways. Mr. Baldwin has provided expert witness services for various venues ranging from New York State Department of Environmental Conservation (NYSDEC) spill and hazardous waste sites to potential noise impacts.

Mr. Baldwin works closely with the U.S. Environmental Protection Agency (EPA), NYSDEC Region 1, Region 2, Region 3 and Central Office, New York State Department of Health (NYSDOH), Suffolk County Department of Health Services (SCDHS) and Nassau County Department of Health (NCDOH). Mr. Baldwin also works with local planning and review boards including the Town of East Hampton, Town of Southampton, Town of Babylon, Town of Brookhaven, Village of

Patchogue, Village of Great Neck and New York City on issues ranging from groundwater quality to historic resources to noise impacts.

Mr. Baldwin has been in the forefront of both evaluating and addressing shallow soils on Long Island which have been impacted by pesticides (particularly arsenic) and herbicides. This important issue is particularly of concern due to the re-development of agricultural lands for residential and educational end uses. Mr. Baldwin has work closely with the SCDHS and Town of Brookhaven to develop effective and easily implementable Soil Management Plans.

Mr. Baldwin's projects include supervising and performing Remedial Investigations/Feasibility Studies (RI/FSs), Interim Remedial Actions (IRMs), and implementation of selected remedies at NYSDEC Class 2 and 2a Inactive Hazardous Waste Disposal sites. Other work, conducted with the NYSDEC, includes evaluating and implementing large-scale groundwater and soil in-situ and ex-situ treatment systems to remediate MTBE.

Mr. Baldwin also has extensive experience in conducting other types of environmental work ranging from NYSDEC spill sites to remediating buried medical waste at a Long Island psychiatric center. Mr. Baldwin has extensive experience providing expert testimony/meeting presentation services in various venues. He has provided same in support of work being conducted in the Village of Greenport, Village of Lake Success, New Hyde Park, Dutchess County Supreme Court, South Farmingdale, Bay Shore, Brookhaven, Wassaic, Central Islip, Plainview and Amityville, New York. Before moving to the East Coast in 1993, Mr. Baldwin worked in the environmental industry in California. His work in the environmental industry consisted primarily of conducting large-scale environmental investigations at United States military and Department of Energy facilities

Selected Project Experience

Groundwater Evaluation and Treatment, Taconic Developmental Disabilities Services Office, Wassaic, NY

Worked on a public water supply site in New York conducting a full-scale groundwater investigation in the vicinity of the facility's supply wells which have been impacted by MTBE. Multiple well clusters were installed surrounding the high-capacity wells to evaluate subsurface conditions. One impacted well was converted to a remediation well to provide hydraulic capture of the MTBE plume prior to its impacting the remaining downgradient wells. A large-scale granulated-activated carbon (GAC) system was installed to treat the water extracted from the well. A 40,000-pound GAC unit was also installed in standby mode to address the facility's drinking water should the concentrations of MTBE ever warrant treatment. Several rounds of groundwater investigation were also conducted to confirm the MTBE source area as a nearby gasoline service station. Pilot testing was conducted and an on-site groundwater treatment system was being designed to provide source area remediation. Part of the pilot testing including evaluating specialized GAC manufactured specifically to address MTBE. Drawdown data associated with a long-term pumping well was utilized to accurately characterize aquifer/hydrogeologic conditions in order to evaluate well capture zones.

Potable Water Treatment System, Village of Brewster, NY

Designed and constructed a supplemental water treatment system at a public water supply plant to address MTBE contamination in the system prior to its distribution. The treatment system consisted of a large air stripping tower, installed in line with an existing air stripper to remove the MTBE to non-detectable concentrations. Additionally, a source area investigation was being conducted to determine the potential source(s) of the MTBE contamination.

Potable Water Treatment System, Sullivan Correctional Facility, Fallsburg, NY

Worked with the NYSDEC to evaluate, design and install a supplemental water treatment system to address MTBE present in a New York State Correctional Facility's drinking water. All four of the facility's wells were impacted. Several remedial options including utilizing GAC or air strippers were evaluated. The selected alternative was a 20,000-pound GAC system which was installed inline and in standby mode.

Former Fuel Terminal, Patchogue River, Patchogue, NY

Conducted a site investigation program at this former major fuel oil terminal site to evaluate the efficacy of same for residential re-development, which would have included a residence-use only marina. The site had been the subject of previous site remediation activities, and the NYSDEC had closed its spill file assuming that the site would only be utilized for commercial or industrial purposes. Soil, groundwater, soil vapor and outdoor ambient air samples were collected and analyzed as part of this evaluation. The results of the investigation indicated that, in part due to the presence of MTBE and other gasoline-related VOCs, additional soil remediation would have been required to make the property suitable for residential re-development. Additionally, the NYSDEC would have likely required the installation and operation of sub-slab depressurization systems for all on-site residential buildings prior to their approving the plans for the site.

Active Marina Facility, Hampton Bays, NY

The owner of this active marina facility was served with a Notice of Violation (NOV) by the NYSDEC for various environmental issues, mostly related to on-site petroleum storage/delivery systems, as well as impacts potentially associated with marine-activity uses such as vessel bottom paint removal and application, use of preserved woods, vessel maintenance activities, housing-keeping issues, etc. Apex was responsible, with input from the NYSDEC, for developing and implementing a Site Investigation Program to investigate potential soil and groundwater impacts associated with the aforementioned on-site practices. Based upon the results of the investigation, Apex was able to conclude that the fuel distribution system was not leaking and that groundwater was not deleteriously impacted. Minor concentrations of MTBE in groundwater were thought to represent ambient conditions typical for an active marina. Minor areas of impacted soil, likely from vessel bottom cleaning activities, were identified. Apex is currently assisting with negotiations with the NYSDEC to potentially allow for the implementation of engineering controls to address the impacted soils.

Aerospace Facility Superfund Site, Lake Success, NY

Managed large-scale site activities at a major Long Island aerospace facility. Activities included operations of on-going IRMs (soil vapor extraction and groundwater extraction and treatment systems); citizen participation activities; design and implementation of on-site remedies (drywell removal and soil excavation, installation of fencing and an 1,800 gallon per minute groundwater extraction and treatment system); on- and off-site RIs; regulatory compliance activities; client interactions; multi-task, multi-contractor scheduling and management; and general project management. As part of the RI, prepared a large three-dimensional groundwater flow and particle model utilizing Visual MODFLOW and MODPATH. The model was then utilized to design an optimum groundwater treatment system.

Prepared a scoping plan and RI report for an Inactive Hazardous Waste Disposal site in New York under the NYSDEC Superfund program

The work involved evaluating the nature and extent of halogenated solvents in soil and groundwater both on and off of the site. Was responsible for overseeing all phases of the report preparation, including communications with the NYSDEC and for implementing the citizen participation program. Also involved in the preparation of the FS report and selection of the final remedy which included the use of an innovative groundwater treatment technology, in-well air stripping

Former Manufacturing Facility Superfund Site, Central Islip, NY

Prepared an RI report for a Class 2 Inactive Hazardous Waste Disposal site under the NYSDEC Superfund program. The work involved evaluating the nature and extent of 1,2,3-trichloropropane (1,2,3-TCP) in soil, soil vapor and groundwater. Additionally, was responsible for evaluating the physical characteristics of this uncommon contaminant and determining potential human health effects as part of the Human Health Evaluation. Oversaw all phases of the report generation, including communications with the NYSDEC and for implementing the citizen participation program, including preparing and presenting the results of the RI at a public meeting.

Former Manufacturing Facility Superfund Site, Plainview, NY

Designed and managed targeted on-and off-site groundwater investigations, reporting and remedial design activities for a Class 2a Inactive Hazardous Waste site under the NYSDEC Voluntary Cleanup Program (VCP). By utilizing existing and recently acquired data, that resulted in a significant cost savings to the client. Oversaw the design of an air sparge/soil vapor extraction system to remediate halogenated volatile organic compounds in the site's source area unsaturated soils and underlying groundwater.

Psychiatric Facility, Islip, NY
Conducted all phases of an expedited buried medical waste program at a large New York State psychiatric hospital. Upon discovery of buried medical waste during the installation of a sewer main, a site investigation program was designed and implemented for the purpose of determining the extent of the buried waste. A successful remediation program was then implemented, which included a project-specific Health and Safety Plan dealing with medical "sharps" and potential blood-borne pathogens. The work was conducted under NYSDEC oversight.

Psychiatric Facility, Middletown, NY
Designed and implemented a subsurface investigation and oxygen release compound (ORC)/bio-venting pilot testing program at an upstate New York State psychiatric facility to remediate a No. 6 fuel oil spill. Due to the existence of on-site infrastructure, in-situ bioremediation techniques were required to remediate the petroleum without disrupting facility operations.

Former Marina Facility, Greenport, NY
Managed one of the few active NYSDEC Brownfield sites on Long Island utilizing New York State Environmental Bond Act funding. The work included evaluating the presence of undocumented USTs utilizing surface geophysical techniques, removing the USTs and associated impacted soils and preparing Site Investigation and Remedial Action reports. Responsible for all regulatory interactions, subcontractor management and Citizen Participation Plan implementation. The work was conducted concurrently with the redevelopment of the site for use as a public park.

General Environmental Planning Experience, NY
Responsible for preparing various chapters of Environmental Impact Statements (EISs) including Geology, Soil and Topography; Groundwater; Utilities, Open Space and Recreational Resources; and Project Alternatives. Reviewed other consultants' EISs for local municipalities to determine compliance with the State Environmental Quality Review Act (SEQRA) and to evaluate the potential impacts of proposed projects. Prepared potential environmental impact sections (e.g., groundwater, wetlands, air quality, visual quality, zoning, etc.) of New York Public Service Commission Article X pre-application packages for four proposed power plants.

General Military Base Experience, Nation-wide
Conducting large-scale environmental investigations at United States Military and Department of Energy facilities. Assignments included: evaluating the nature and extent of soil and groundwater contamination associated with landfills, fire training facilities and miscellaneous disposal areas on several military bases for the United States Army Corps of Engineers and the United States Navy; characterizing the nature and extent of unexploded ordnance; obtaining and interpreting surface and borehole geophysical surveys; conducting large-scale aquifer pumping tests; preparing Remedial Investigation and Site Investigation reports

Appendix B

Supporting Information for TCL VOC plus Freon 113 Soil Vapor Analysis

UNITED STATES ENVIRONMENTAL PROTECTION AGENCY
Office of Research and Development
Washington, D.C. 20460

ENVIRONMENTAL TECHNOLOGY VERIFICATION PROGRAM
VERIFICATION STATEMENT

TECHNOLOGY TYPE:	**PASSIVE SOIL GAS SAMPLER**
APPLICATION:	**SUBSURFACE SOIL GAS SAMPLING**
TECHNOLOGY NAME:	**GORE-SORBER® SCREENING SURVEY PASSIVE SOIL GAS SAMPLING SYSTEM**
COMPANY:	**W.L. GORE & ASSOCIATES, INC.**
ADDRESS:	**100 CHESAPEAKE BOULEVARD ELKTON, MARYLAND 21921**
PHONE:	**(410) 392-7600**

ETV PROGRAM DESCRIPTION

The U.S. Environmental Protection Agency (EPA) created the Environmental Technology Verification (ETV) Program to facilitate the deployment of innovative technologies through performance verification and information dissemination. The goal of the ETV Program is to further environmental protection by substantially accelerating the acceptance and use of improved and cost-effective technologies. The ETV Program is intended to assist and inform those involved in the design, distribution, permitting, and purchase of environmental technologies. This document summarizes the results of a demonstration of the W.L. Gore & Associates, Inc., GORE-SORBER® Screening Survey passive soil gas sampling system.

PROGRAM OPERATION

Under the ETV Program and with the full participation of the technology developer, the EPA evaluates the performance of innovative technologies by developing demonstration plans, conducting field tests, collecting and analyzing demonstration data, and preparing reports. The technologies are evaluated under rigorous quality assurance (QA) protocols to ensure that data of known and adequate quality are generated and that the demonstration results are defensible. The EPA's National Exposure Research Laboratory, which demonstrates field characterization and monitoring technologies, selected Tetra Tech EM Inc. as the verification organization to assist in field testing various soil and soil gas sampling technologies. This demonstration was conducted under EPA's Superfund Innovative Technology Evaluation Program.

DEMONSTRATION DESCRIPTION

In May and June 1997, the EPA conducted a field test of the GORE-SORBER® Screening Survey passive soil gas sampling system along with one other soil gas and four soil sampling technologies. This verification statement focuses on the GORE-SORBER® Screening Survey passive soil gas sampling system; similar statements have been prepared for each of the other technologies. The performance of the GORE-SORBER® Screening Survey passive soil gas sampling system was compared to the reference sampling method, active soil gas sampling (which provides a snapshot of the soil gas environment at the time the sample is collected). The comparison addressed three parameters: (1) volatile organic compound (VOC) detection and quantitation, (2) sample retrieval time, and (3) cost. Data quality indicators for precision, accuracy, representativeness, completeness, and comparability were also assessed against project-specific QA objectives to ensure the usefulness of the data.

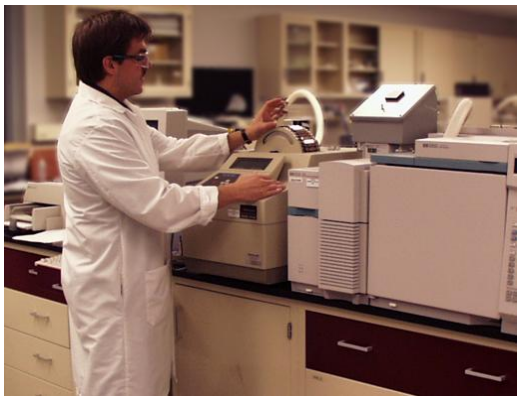
Analytical Method Summary & QA Procedures

Analytical Method Summary

Instrumentation consists of state of the art gas chromatographs equipped with mass selective detectors, coupled with automated thermal desorption units. Sample preparation involves cutting the tip off the bottom of the GORE™ Module and transferring one or more exposed sorbent containers (sorbers) to a thermal desorption tube for analysis. The adsorbent remains clean and requires no further sample preparation. The remaining unanalyzed sorbers are archived for a minimum of 15 days.

Quality Assurance Procedures

The analytical method employed is a modified US EPA method 8260/8270. Before each sequence, two instrument blanks, a sorber containing 5µg BFB (Bromofluorobenzene), and a method blank are analyzed. The BFB mass spectra must meet the criteria set forth in the method before GORE™ Modules can be analyzed. A method blank and a sorber containing BFB are also analyzed after every 30 Modules and/or trip blanks. Standards containing the selected target compounds at five calibration levels are analyzed at the beginning of each sequence. The criterion for each target compound is less than 25% RSD (relative standard deviation). If this criterion is not met for any target compound, the analyst has the option of generating second- or third-order standard curves, as appropriate. A second-source reference standard, at a level of 10µg per target compound, is analyzed after every ten modules and/or trip blanks, and at the end of the sequence. To minimize handling of the field-exposed modules, no surrogates or internal standards are used. Positive identification of target compounds is determined by 1) the presence of the target ion and at least two secondary ions; 2) retention time versus reference standard; and, 3) the analyst's judgment. As an option, data deliverables can be provided for all samples and blanks analyzed.



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The GORE-SORBER® Screening Survey passive soil gas sampling system was demonstrated at two sites: the Small Business Administration (SBA) site in Albert City, Iowa, and the Chemical Sales Company (CSC) site in Denver, Colorado. These sites were chosen because each site exhibited a wide range of VOC concentrations and a distinct soil type. The VOCs detected at the sites include vinyl chloride; cis-1,2-dichloroethene (cis-1,2-DCE); 1,1-dichloroethane (1,1-DCA); 1,1,1-trichloroethane (1,1,1-TCA); trichloroethene (TCE); and tetrachloroethene (PCE). The SBA site is composed primarily of clay soil, and the CSC site is composed primarily of medium- to fine-grained sandy soil. A complete description of the demonstration, including a data summary and discussion of results, is available in the report titled *Environmental Technology Verification Report: Passive Soil Gas Sampler, W.L. Gore & Associates, Inc., GORE-SORBER®* Screening Survey, EPA 600/R-98/095.

TECHNOLOGY DESCRIPTION

The GORE-SORBER® Screening Survey uses GORE-SORBER® modules to collect soil gas samples. The GORE-SORBER® module is a passive soil gas sampler that is designed to collect a broad range of VOCs and semivolatile organic compounds (SVOC), including halogenated compounds, petroleum hydrocarbons, and polynuclear aromatic hydrocarbons. A typical GORE-SORBER® module contains two or more passive collection units called sorbers. Each sorber contains an equal amount of sorbent materials (polymeric and carbonaceous resins). These granular adsorbent materials are used because of their affinity for a broad range of VOCs and SVOCs. The sorbers are sheathed in the bottom of a 4-foot-long, vapor-permeable retrieval cord. The cord and the sorbers are constructed of inert, hydrophobic, microporous GORE-TEX® expanded polytetrafluoroethene (ePTFE). The microporous structure of ePTFE allows vapors to move freely across the membrane and onto the sorbent material. This microporous structure also protects the granular adsorbents from physical contact with soil particulates and water. The GORE-SORBER® module is installed to a depth of 2 to 3 feet. A pilot hole is created using a slide hammer and tile probe or hand drill (in paved areas). The sampler is then manually inserted into the hole using push rods. The module is retrieved by hand and must be analyzed by the developer.

VERIFICATION OF PERFORMANCE

The demonstration data indicate the following performance characteristics for the GORE-SORBER® Screening Survey passive soil gas sampling system:

VOC Detection and Quantitation: The GORE-SORBER® Screening Survey detected the same compounds in each sample as the reference soil gas sampling method, as well as several VOCs that the reference method did not detect. This performance characteristic suggests that the GORE-SORBER® Screening Survey may detect VOCs that are at lower concentrations in the subsurface than the reference soil gas sampling method can detect. The results also indicate a general correlation between the GORE-SORBER® Screening Survey and reference method data. However, at high contaminant levels, the ratio between the mass of contaminant in soil gas detected using the GORE-SORBER® module and the concentration of contaminant in soil gas detected using the reference soil gas sampling method decreases, suggesting that sorbent saturation may have occurred. The GORE-SORBER® Screening Survey and reference method are field screening techniques that provide only an estimate of the actual concentration of contaminants in soil gas. Because the GORE-SORBER® Screening Survey and reference method use different techniques to collect soil gas samples, it is not expected that the two methods will provide the same response or that the data will be directly comparable. In addition, the GORE-SORBER® Screening Survey yields results in micrograms per sample and the reference soil gas sampling method reports results in nanograms per liter. Therefore, a statistical analysis of the data was not performed, and interpretation of the chemical concentration data for this demonstration is limited to qualitative observations.

Sample Retrieval Time: Installation of the GORE-SORBER® modules averaged 8.0 minutes per sampler at the SBA site and 7.4 minutes per sampler at the CSC site. For the demonstration, the modules were left in place for approximately 10 days. Collection of the modules required an average of 1.9 minutes per sampler at the SBA site and 2.4 minutes at the CSC site. Overall, installation and collection of 35 GORE-SORBER® modules at the SBA site required 346 minutes, an average of 9.9 minutes per sample and installation and collection of 28 GORE-SORBER® modules at the CSC site required 274 minutes, an average of 9.8 minutes per sample. The analysis and reporting by the technology developer required 14 to 18 days from the time samples were collected until the laboratory report was delivered. The reference soil gas method required 458 minutes to collect 35 samples at the SBA site, an average of 13.1 minutes per sample, and 183 minutes to collect 28 samples at the CSC site, an average

of 6.5 minutes per sample. One day was required per site to analyze the samples and report the results. Based on the demonstration results, the average sample retrieval times for the GORE-SORBER® modules were quicker than the reference soil gas sampling method in the clay soils at the SBA site and slower than the reference sampling method in the sandy soils at the CSC site. The results also indicate that the sample retrieval time for the GORE-SORBER® modules may be less susceptible to variations in soil type than the sample collection times for the reference method. During sample collection using the reference active soil gas sampler, the clay soil at the SBA site caused the system to hold its vacuum at several sampling locations; therefore, soil gas was not completely drawn into the system for sampling. In these cases, the rod was withdrawn in additional 6-inch increments until the vacuum was broken and the system's pressure reached equilibrium with atmospheric pressure. The vacuum problem was not encountered in the sandy soil at the CSC site. A two-person sampling crew retrieved soil gas samples using the GORE-SORBER® Screening Survey at both the SBA and CSC sites, and a three-person sampling and analysis crew collected and analyzed the soil gas samples using the reference soil gas sampling method at both sites.

Cost: Based on the demonstration results, the GORE-SORBER® Screening Survey cost $125 to $225 per sample plus equipment costs of $25 to $85 per day and mobilization/demobilization costs of $200 to $600 per day. Operating costs for the GORE-SORBER® Screening Survey ranged from $810 to $1,540 at both the clay soil site and the sandy soil site. For this demonstration, the active soil gas sampling method was procured at a lump sum of $4,700 per site for the collection and analysis of 40 soil gas samples at each site. Oversight costs for the active soil gas sampling method ranged from $680 to $1,260 at the clay soil site and $480 to $910 at the sandy soil site. A site-specific cost and performance analysis is recommended before selecting a subsurface soil gas sampling method.

A qualitative performance assessment of the GORE-SORBER® Screening Survey indicated that (1) all 63 modules installed at the SBA and CSC sites were retrieved without sample loss, resulting in 100 percent completeness;(2) the sampler is easy to use and requires minimal training (a 10-minute training video is available from the developer); (3) logistical requirements for the GORE-SORBER® Screening Survey require that the samplers be installed using a manual push tool, left in place for several days, retrieved by hand, and sent to the developer for analysis; and (4) sample handling in the field requires that sorbent be properly containerized and shipped to the developer. Other factors that may affect the performance range of the GORE-SORBER® Screening Survey but that were not evaluated during the demonstration are sampling depth, time allowed for sampling, type and amount of sorbent material placed in the GORE-SORBER® module, and ability of vapors to move across the module membrane.

The demonstration results indicate that the GORE-SORBER® Screening Survey can provide useful, cost-effective data for environmental problem-solving. The GORE-SORBER® modules successfully collected soil gas samples in clay and sandy soils. The sampler provided positive identification of target compounds and may detect lower concentrations of VOCs in the soil gas than can the reference soil gas sampling method. Based on the results of this demonstration, there appears to be a general correlation between the GORE-SORBER® Screening Survey and reference method data. However, at higher contaminant levels, the ratio between the mass of contaminant detected in the soil gas using the GORE-SORBER® module and the concentration of contaminant detected using the reference method decreases. As with any technology selected, the user must determine what is appropriate for the application and the project data quality objectives.

Gary J. Foley, Ph.D.
Director
National Exposure Research Laboratory
Office of Research and Development

GORE™ SURVEYS ENVIRONMENTAL SITE ASSESSMENT

FOCUSING YOUR REMEDIATION EFFORTS.

Commonly Requested Analytes

Volatiles
Vinyl Chloride
Methyl t-butyl ether
Benzene
Toluene
Ethylbenzene
o-Xylene
m,p-Xylene
Octane
1,1-Dichloroethane
1,2-Dichloroethane
1,1,1-Trichloroethane
1,1,2-Trichloroethane
1,1,1,2-Tetrachloroethane
1,1,2,2-Tetrachloroethane
1,1-Dichloroethene
trans-1,2-Dichloroethene
cis-1,2-Dichloroethene
Trichloroethene
Tetrachloroethene
Chloroform
Carbon Tetrachloride
Chlorobenzene

Semi-volatiles
1,3,5-Trimethylbenzene
1,2,4-Trimethylbenzene
1,2-Dichlorobenzene
1,3-Dichlorobenzene
1,4-Dichlorobenzene
Undecane
Tridecane
Pentadecane
Naphthalene
2-Methyl naphthalene
Acenaphthene
Acenaphthylene
Fluorene
Phenanthrene
Anthracene
Fluoranthene
Pyrene

Explosives
Nitrobenzene
2-Nitrotoluene
3-Nitrotoluene
4-Nitrotoluene
1,3-Dinitrobenzene
2,4-Dinitrotoluene
2,6-Dinitrotoluene
1,3,5-Trinitrobenzene
2,4,6-Trinitrotoluene

Chemical Agents/Breakdown Products
Mustard (as a TIC)
1,4-dithiane
1,4-oxathiane
Benzothiozole
p-Chlorophenylmethylsulfide
p-Chlorophenylmethylsulfoxide
p-Chlorophenylmethylsulfone
Dimethyldisulfide
DIMP (Diisopropyl methylphosphonate)
DMMP (Dimethyl methylphosphonate)
4-chloroacetophenone
2-chloroacetophenone

Mercury (elemental),
Pesticides/Herbicides & PCB Cogeners
Capabilities demonstrated.

NOTE:
This is not a comprehensive list of detection or analytical capabilities.

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Analytical Options

The GORE™ Survey service includes:

- GORE™ Modules
- Analysis
- Data reporting
- Up to three contour maps (where applicable)
- Final Report (in duplicate)
- Pre- and post-survey consultation (as needed)

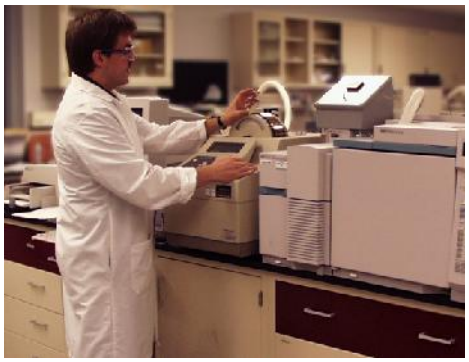
– Analysis is by thermal desorption, gas chromatography, and mass spectroscopy via modified US EPA methods 8260/8270.
– Contour maps in paper and PDF formats.
– The Final Report is issued in paper format (electronic format available upon request).
– The survey does not include field installation and retrieval costs, or shipping costs.

Standard (A1)	Fuels (A2)	Chlorinateds (A10)	VOCs, SVOCs, PAHs (A4)
MtBE	MtBE	1,1-DCE	*Standard (A1) List plus*
Benzene	Benzene	trans-1,2-DCE	Acenaphthylene
Toluene	Toluene	cis-1,2-DCE	Acenaphthene
Ethylbenzene	Ethylbenzene	TCE	Fluorene
m,p-xylene	m,p-xylene	PCE	Phenanthrene
o-xylene	o-xylene	1,1-DCA	Anthracene
Octane	Octane	1,2-DCA	Fluoranthene
Undecane	Undecane	1,1,2-TCA	Pyrene
Tridecane	Tridecane	1,1,1-TCA	
Pentadecane	Pentadecane	1,1,2,2-TetCA	
1,3,5-TMB	1,3,5-TMB	1,1,1,2-TetCA	
1,2,4-TMB	1,2,4-TMB	Chloroform	
Naphthalene	Naphthalene	Carbon tetrachloride	
2-Methylnaphthalene	2-Methylnaphthalene	Chlorobenzene	
1,1-DCE	TPH	1,2-DCB	
trans-1,2-DCE		1,3-DCB	
cis-1,2-DCE		1,4-DCB	
TCE			
PCE			
1,1-DCA			
1,2-DCA			
1,1,2-TCA			
1,1,1-TCA			
1,1,2,2-TetCA			
1,1,1,2-TetCA			
Chloroform			
Carbon tetrachloride			
Chlorobenzene			
1,2-DCB			
1,3-DCB			
1,4-DCB			
TPH			

Explosives (A6)	Chemical Agents (A8)
Standard (A1) List plus	*Standard (A1) List plus*
Nitrobenzene	1,4-Dithiane
2-Nitrotoluene	1,4-Oxathiane
3-Nitrotoluene	Thiodiglycol
4-Nitrotoluene	Benzothiozole
1,3-Dinitrotoluene	2-Chloroacetophenone
2,6-Dinitrotoluene	4-Chloroacetophenone
2,4-Dinitrotoluene	p-chlorophenylmethylsulfide
1,3,5-Trinitrotoluene	p-chlorophenylmethylsulfone
2,4,6-Trinitrotoluene	p-chlorophenylmethylsulfoxide
	Dimethyldisulfide
	Diisomethylphosphonate (DIMP)
	Dimethylmethylphosphonate (DMMP)

OPTIONS

- Custom analyte suites
  - A3 – any eight compounds from A1
  - A7 – custom list
- Vinyl chloride
- TPH only*
- GRO and DRO only*
- Expedited TAT (data table only)
  - 25%, ten business day
  - 50%, five business day
- QA Deliverables
  - BFB tune data
  - Initial calibration data
  - quantitation reports
  - extracted ion chromatograms
- Library searches
- Tentatively identified compounds



* - Compound specific reporting for the Fuels analyte list (A2) available – no additional sampling or analysis required.

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Appendix C

Supporting Information for Soil and Groundwater Analysis

Appendix C-1

Supporting Information for TCL VOC Analysis

Volatile Organic Compounds (GC/MS)	**8260B**

Analyte Description	CAS Number
Dichlorodifluoromethane	75-71-8
Chloromethane	74-87-3
Vinyl chloride	75-01-4
Bromomethane	74-83-9
Chloroethane	75-00-3
Trichlorofluoromethane	75-69-4
1,1-Dichloroethene	75-35-4
1,1,2-Trichloro-1,2,2-trifluoroethane	76-13-1
Acetone	67-64-1
Carbon disulfide	75-15-0
Methyl acetate	79-20-9
Methylene Chloride	75-09-2
trans-1,2-Dichloroethene	156-60-5
Methyl tert-butyl ether	1634-04-4
1,1-Dichloroethane	75-34-3
cis-1,2-Dichloroethene	156-59-2
Methyl Ethyl Ketone	78-93-3
Chloroform	67-66-3
1,1,1-Trichloroethane	71-55-6
Cyclohexane	110-82-7
Carbon tetrachloride	56-23-5
Benzene	71-43-2
1,2-Dichloroethane	107-06-2
Trichloroethene	79-01-6
Methylcyclohexane	108-87-2
1,2-Dichloropropane	78-87-5
Bromodichloromethane	75-27-4
cis-1,3-Dichloropropene	10061-01-5
methyl isobutyl ketone	108-10-1
Toluene	108-88-3
trans-1,3-Dichloropropene	10061-02-6
1,1,2-Trichloroethane	79-00-5
Tetrachloroethene	127-18-4
2-Hexanone	591-78-6
Dibromochloromethane	124-48-1
1,2-Dibromoethane	106-93-4
Chlorobenzene	108-90-7
Ethylbenzene	100-41-4
m&p-Xylene	136777-61-2
o-Xylene	95-47-6
Xylenes, Total	1330-20-7
Styrene	100-42-5
Bromoform	75-25-2
Isopropylbenzene	98-82-8
1,1,2,2-Tetrachloroethane	79-34-5
1,3-Dichlorobenzene	541-73-1
1,4-Dichlorobenzene	106-46-7
1,2-Dichlorobenzene	95-50-1
1,2-Dibromo-3-Chloropropane	96-12-8
1,2,4-Trichlorobenzene	120-82-1

Title: SOP for GC/MS Volatiles
[Method SW846 8260B]

Approvals (Signature/Date):

Technical Manager	Date	Health & Safety Manager / Coordinator	Date
Quality Assurance Manager	Date	Laboratory Director	Date

Facility Distribution No. _____ **Distributed To:_____**

1.0 APPROVALS

The approval of the Laboratory Director, the Facility QA Manager, Group Leader and Health and Safety Officer signify that the document has been prepared in accordance with the format requirements that have been established in this document. In addition, if the document is a Method SOP the signature signifies that it meets the specified requirements.

2.0 SCOPE AND APPLICATION

2.1 The objective of this document is to outline the techniques for determining the presence and concentration of various volatile organic target and non-target compounds in multi-media, multi-concentration samples. The 8260B compounds for this method are listed in Table 1.0. Tables 1.1, 2.0 and 2.1 lists the expanded Appendix IX/additional target compounds which are applicable to this method. The extraction method used in this procedure is purge and trap which is coupled with a gas chromatograph/mass spectrometer analysis.

2.2 It is the policy of TESTAMERICA and of the GC/MS Group to ensure that we administer contracts and orders for goods and services in a manner that is fully compliant with governmental laws and regulations, as well as the TESTAMERICA Policy Statement on Business Ethics and Conduct.

2.3 The document control number for this SOP is CT-MSS-28, Rev 9.

3.0 Terms and Definitions

3.1 There are many definitions used within the laboratory, which may be generic to all Laboratory analyses, or more specific for certain methods. For the most recent terms and definitions used within the laboratory, reference the SOP of Terms and Definitions.

4.0 SUMMARY OF METHOD

4.1 This method employs the technique of purge and trap, coupled with a gas chromatograph/mass spectrometer analysis. An aliquot of sample, usually 5 ml or 25 ml of water, 5 g of soil for low level soil method, and 5 g of soil collected with methanol and extracted for medium level soil method, is purged in a gas tight chamber with UHP grade helium to remove the volatile compounds. The vapor is swept through a sorbent column where the volatiles are trapped. Next the sorbent trap is heated and back flushed, thereby desorbing the volatiles onto the analytical column within the gas chromatograph. The fused silica capillary column is then temperature programmed to separate the volatiles prior to detection by the mass spectrometer.

4.2 This SOP is based on USEPA SW846 5030B/5035/8260B Methods.

4.3 The following deviations from the method are noted:

. The selected internal standards for this 8260B Method are:

- Fluorobenzene
- 1,4-dichlorobenzene-d4
- Chlorobenzene-d_5

. The selected surrogates for this 8260B Method are:

- Dibromofluoromethane
- 1,2-Dichloroethane-d_4
- Toluene-d_8
- Bromofluorobenzene

. The routine 8260B target list is the TCL list per Table 1.0.
Additional Analyzed that may be analyzed by 8260B are listed inTables 1.1, 2.0 and 2.1.

. Method 8260B can be either 5 ml or 25 ml purge per methodology. This must be specified by the client prior to sample analysis. In many cases the detection limits requested can be achieved by 5 ml sample volume using the Agilent 5973 with a lower calibration range.

5.0 INTERFERENCES

5.1 Method interferences may be caused by contaminants in solvents, reagents, out-gassing from equipment plumbing, and laboratory solvent vapors. This can lead to discrete artifacts and/or elevated baseline in the gas chromatograph. All these materials must be demonstrated to be free from interferences by the running of laboratory reagent blanks.

5.2 Interferences may also be caused by the diffusion of volatiles through the septum seal during storage and handling. A holding blank prepared from reagent water is stored with the samples and analyzed to serve as a check. Holding blanks are prepared weekly for each volatile sample storage unit. The results for the holding blank are reported by GC/MS and submitted to the QA/QC Manager to be filed for future reference.

5.3 Only Approved lots of Purge and Trap grade methanol shall be used for standards and sample dilutions.

5.4 Only reagent water free from Volatile organics can be used in the volatiles laboratory for sample dilutions, reagent blanks, and aqueous standards.

6.0 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

The gas chromatograph contains 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.

There are areas of high voltage in both the Gas Chromatograph and 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.

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.

6.1 Analysts shall treat all samples as if they are hazardous and take all appropriate safety precautions. Analysts shall wear, if needed:

. lab coats
. safety glasses with side shields and
. chemical resistant gloves

when handling samples or preparing standards.

6.2 Solvents and all standards shall be used in the fume hoods to minimize environmental exposure to solvent vapors.

6.3 Material Safety Data Sheets for all chemicals used in the operation are present in the laboratory for immediate access.

7.0 SAMPLE PRESERVATION AND STORAGE

7.1 All samples for volatile analysis must be protected from light and refrigerated at 4°C from

the time of receipt until analysis.

7.2 All HCL preserved samples for volatile analysis shall be analyzed within 14 days of sample collection, or seven days unpreserved.(the laboratory must be notified if samples are unpreserved to ensure unpreserved samples can be run within holding time) NYSDEC 8260B samples must be analyzed within ten days of receipt.

7.3 Refer to the sample control processing, sample removal, and log-in SOP's in section 15.0.

7.4 Low level soil analysis will be preserved with reagent grade Sodium bisulfate or Reagent water will be used if the sample effervesces when it comes in contact with the Sodium Bisulfate solution. If the soil is placed in water, this sample will be run that day, or frozen at a slight 30-45 degree angle to prevent breakage, for up to fourteen days. Client must specify at time of bottle order if no preservative is required. Encore containers or equivalent may be used for low level soil analysis. If these are used the soil plug must be transferred within 48 hours from collection.

7.5 Methanol preserved vials will be sent out for projects requesting the high concentration soil analysis. Encore containers or equivalent may be used for medium level soils. If these are used the soil plug must be transferred into methanol within 48 hours from collection. A 14 day hold from collection is used for methanol. Client must also specify what volume of methanol is required, if surrogates need to be pre-spiked prior to being collected in the field, and what soil volume will be sampled. Laboratory default is 5mls of un-spiked methanol to accept 5 grams of soil in the field.

8.0 APPARATUS AND MATERIALS

8.1 Purge and trap concentrator : Encon

8.2 Purge and trap autosampler - or Archon multiple position autosampler

8.3 Volatile Trap - VOCARB 3000 traps packed with: Carbopack B/Carboxen 1000 and 1001

8.4 GC/MS/DS System

8.4.1 Hewlett-Packard Model, 5975, 5973, 5972, 5971 or 5970 GC/MS with jet separator, or direct capillary interface, capable of scanning from 35 to 300 amu every two seconds or less, utilizing 70 volts (nominal) electron energy in the EI ionization mode, and producing a mass spectrum which meets all the instrument performance criteria when 50 ng of BFB is injected through the GC inlet. Refer to Table 3.0 for the performance criteria. and section 12.1.1 for the instrumental conditions.

8.4.2 Direct interface, or split injection port from purge and trap transfer line to GC column

8.4.3 GC Column – Restek RTX-VMS 20 meter 0.18 mm ID 1.00 micron film thickness or 75 m

x 0.53 mm ID x 3.0 um film thickness Supelco 624 fused silica widebore capillary column, or equivalent narrow bore such as 0.18 or 0.25 are used with split injection ports.

8.4.4 Chemstation / Target software capable of continuous acquisition and storage, on machine readable media, of all mass spectra obtained throughout the duration of the chromatographic program. The computer has software that allows searching any data file for ions of a specified mass and plotting such ion abundances versus time or scan number (EICP). Software also allows integrating the abundance in any EICP between specified time limits. Also, software allows for the comparison of sample non-target spectrum against reference library spectra. The most recent release of the NIST/EPA/MSDC mass spectral library shall be used as the reference library. The data system flags all manual edits with "M" qualifier.

8.4.5 Target network tape backup system.

8.4.6 Syringes – Gas-tight microsyringes 25 ul and larger, 0.006 inch ID needle; 5 ml gas-tight syringes with shut off valve, all syringes used shall be gas-tight

8.4.7 Balance – top loading balance capable of weighing +/-0.1 g, and an analytical balance capable of weighing +/-0.0001 grams. This balance must be checked for calibration once, prior to use with NIST weights. The range will be from 1gram to 100grams to bracket the working range.

8.4.8 Methanol – Purge and Trap Grade

8.4.9 Fritted sparger , culture tube, or 40ml Voa vial.

8.4.10 5ml syringe –with Luer ends, if applicable to the purging device

8.4.11 Glassware

- Bottle – 15 ml, screw cap, with Teflon cap liner
- Volumetric flasks – class A with ground glass stoppers
- Vials – various for standards

8.4.12 A heater or heated water bath capable of maintaining the purge device at 40°C +/- 1°C attached to a stir plate or sonicator that will agitate the low level soil sample, but not for waters or medium level soils

8.4.13 pH paper – narrow range (0-6pH units) and wide (0-14pH units)- All aqueous samples will have a pH taken and recorded in the injection logbook. Any pH readings above 2 will be addressed in a corrective action, if samples are analyzed more than seven days from collection.

8.4.14 Magnetic stir bars

8.4.15 Polyethylene glycol (PEG)

9.0 REAGENTS AND STANDARD PREPARATION

9.1 Reagent Water - Laboratory certified water, free from contaminants.

9.2 Dilution Methanol - Approved lots of Purge and Trap Grade or equivalent.

9.3 Stock Standards - certified standards purchased from commercial sources containing ampulated mixes of target compounds, matrix spike compounds, surrogates, and internal standards are used by the laboratory as stock standards. New ampules are opened every few months, or sooner, if the standard has degraded, evaporated, or the manufactures' date has expired. Ampules containing Gases and Ketones may need to be opened more often.

9.4 Working Standards

9.4.1 Instrument performance check solution - 4-Bromofluorobenzene (BFB) 25 ng/ul solution of BFB in methanol is prepared every six months, or sooner if the solution has degraded or evaporated. Add 2 ul of this solution into 5ml of reagent water for a 50ng concentration. Or can be directly injected into the injection port.

Compound Standard	Initial Concentration	Amount Used	Final Volume	Final Concentration
4-Bromofluorobenzene Sigma-Aldrich #48800	25,000ppm	5 ul	5 mL	25 ppm

9.4.2 Calibration Standard Solution

A 50ppm working calibration standard containing all the volatile target compounds in methanol is prepared monthly, or sooner, if the solution has degraded or evaporated. (125ppm or other levels can be made at the discretion of the analyst. The attached chart is presented as an example)

Full Cal std	Initial concentration	Amount added in ul's	Final Volume	Final concentration
Custom Mix Sigma Aldrich	2000ppm	625 ul	25 mL	50ppm

2026610				
502/524 Sigma-Aldrich-502111				
Mix 6 SigmaAldrich48799-U				
Chloroprene Sigma-Aldrich 861145				
Ketones Sigma-Aldrich 861149				
High Concentration in house supelco / ultra	10,000ppm (varied)	125 ul		

High Concentration	Initial concentration	Amount added in ul's	Final Volume	Final concentration
Acrolein SigmaAldrich 48501	Neat	59.6 ul	1 mL	50,000 ppm
2- Chloroethylvinylether SigmaAldrich 48516		9.5 ul		10,000 ppm
Vinyl Acetate SigmaAldrich 48486		10.7 ul		10,000 ppm
1,4-Dioxane SigmaAldrich 442251		96 ul		100,000 ppm
Acetonitrile SigmaAldrich 48484		140 ul		100,000 ppm
Isobutanol UltraScient RCC-183		124 ul		100,000 ppm

9.4.3 Internal Standard (IS) Spiking Solution

A 125 ppm IS spiking solution containing fluorobenzene, chlorobenzene-d_5, and 1,4-dichlorobenzene-d4 in methanol is prepared as needed, unless the solution has degraded or evaporated. One microliter (ul) is automatically added by the Archon to give a concentration of 25 ug/L.

Compound **Standard**	Initial **Concentration**	Amount **Used**	Final **Volume**	Final **Concentration**
Internal Standard Restek-30241	2500ppm	250 ul	5 mL	125 ppm

9.4.4 System Monitoring Compound (SMC) Spiking Solution

A 125 ppm SMC spiking solution containing Dibromofluoromethane, 1,2-dichloroethane-d_4, toluene-d_8, and 4-bromofluorobenzene in methanol is prepared weekly, or sooner, if the solution has degraded or evaporated. One microliter (ul) is automatically added by the Archon to give a concentration of 25 ug/L.

Compound Standard	Initial Concentration	Amount Used	Final Volume	Final Concentration
Surrogate Standard Restek 30240	2500ppm	250 ul	5 mL	125 ppm

9.4.5 Full Matrix Spiking (MS) Solution

A 50 ppm FMS solution containing the target compounds. (same solution as calibration standard see section 9.2.2).

9.4.6 LCS Spiking Solution (LCS)(20ppb_QCS)

A 25 ppm LCS solution containing all TCL, along with other compounds is prepared as needed from a source independent of the calibration standard in methanol.

LCS spiking standard	Initial concentration	Amount added in ul's	Final Volume	Final concentration
Custom cal Mix Restek 552187	2000-40,000ppm	62.5 ul	5 ML	25ppm
502.2 2000mega mix Restek 30431	2000ppm	62.5 ul		
Voa Cal mix 1 Restek 30006	5000ppm	25 ul		
502.2 Cal Mix Restek 30042	2000ppm	62.5 ul		

9.5 The standard coding system is detailed below:

A. The first letter of the code is a group identifier (ex. V is Volatiles), the next nine numbers/letters are the identifier of the std, the next five numbers is the counter the LIMS applies to each. An example of a standard code is VW8260IS_00023.

B. A label is printed and affixed to the standard.

9.6 Storage of all working standards for volatile analysis must be refrigerated at 4°C +/- 2°C degrees. Stocks are stored according to manufacturer recommendations.

10.0 CALIBRATION

10.1 Calibration Standards

Five or (six point) aqueous initial calibration standards containing all the volatile target compounds and SMC's are prepared at the 5, 20, 50, 100, and 200 ug/L levels.

(0.5,2,5,20,50,100 ug/l on Agilent 5973 GC/MS) These standards are prepared from working standards in section 9.0.

The methanol purged in the aqueous calibration standards must not exceed 1% by volume.

10.2 The working calibration of this method is defined by the initial calibration curve, 5 ug/L to 200 ug/L. All samples with target compounds exceeding the upper curve point must be diluted to within the upper half of the calibration range. In the analysis immediately following a sample that requires a dilution, results cannot be over the RL (or PQL) for the particular compound that required dilution.Therefore eliminating the possibility of carryover from the high concentration sample. Archon / Encon auto-samplers / Concentrators must be deemed free of carry-over prior to continuing sample analysis.

10.2.1 The low level calibration range of the method is .5ug/L-100ug/L.

10.3 Calibration curve preparation

See Table 9.0 for calibration curve standard preparation details.

One microliter (ul) of internal standard is automatically added by the Archon to give a concentration of 25 ug/l to all calibration standards for a final concentration of 25 ug/L. For the 25ml purge the IS spiking solution is at 5ug/l.

An initial calibration must be analyzed on each GC/MS system upon column installation, source cleaning, or whenever corrective action is taken which may affect the initial calibration criteria, or if the continuing calibration criteria can not be met.

Separate initial and continuing calibration must be analyzed for water samples, and low level soil samples (unheated versus heated/agitated purge). Extracts of medium level soil samples may be analyzed using the calibrations for water.

Quantitation is based on the average response factor from the initial calibration.

10.4 Acceptance criteria for the Initial Calibration

The initial calibration criteria must meet the following:

. Calibration Check Compounds (CCC) must have RF's whose percent relative standard deviations are less than 30 percent.

. System Performance Check Compounds (SPCC) Average RF must be equal to or greater than 0.100 except for chlorobenzene and 1,1,2,2- tetrachloroethane must be 0.30 or higher.

10.4.1 The percent relative standard deviation (%RSD) should be less than 15 % for each

compound of interest.

1. If the %RSD is less than 15 % then the average RF from the initial calibration curve is used for quantitation of the compound.

2. If the **mean** %RSD is below 15% for **all** compound in the initial calibration, then average RF can be used, if not, then linear regression is used for quantitation for those compounds above 15%. The minimum correlation coefficient for any compound using linear regression must also be 0.99.

3. There will be other clients that request particular criteria that is above and beyond the scope of this S.O.P. In these cases the client's criteria must be taken into consideration. Their criteria may enhance the basic requirements of this S.O.P. However; all criteria described in this S.O.P. must be followed. Additional client requests must be agreed upon prior to sample analysis.

10.5 Continuing calibration preparation

The continuing calibration standard is the 50 ug/L for 5971 instruments, and 20 ug/L for 5973 instruments. This standard can be part of the initial calibration or is run as a separate standard prepared as above at 50 ug/L / 20 ug/L.

The continuing calibration standard must be analyzed every 12 hours to verify that the initial calibration is still valid. Periodically the continuing calibration level should vary in concentration as a system check standard to satisfy NELAC requirements. This should be done during a 12 hour calibration, but not used as the continuing calibration for the associated samples.

If a continuing calibration fails for any reason, note the reason for failure in the comments' section of the logbook. After two attempts the analyst must determine if an initial calibration needs to be analyzed, or if an educated decision can be made to meet the continuing calibration with the analysis of further calibration checks. One steadfast rule is the last calibration check that is analyzed must be the one used.

Note: Method 8000 and TESTAMERICA policy recommends running an initial calibration after two calibration checks. Currently analysts will note reasons for failures in the comment section of the logbook. After running two calibration checks, the instrument shall be re-tuned, if in the expert opinion of the analyst that a calibration curve is not needed, but either maintenance, or standard preparation will allow for acceptable continuing calibration verification, then this procedure will be acceptable.

10.6 Continuing Calibration Acceptance criteria

The internal standard areas are compared to the mid-point of the initial calibration standard. If the internal standard areas are within 50-100% Difference and the

requirements below are met then the calibration check is successful.

. Calibration Check Compounds (CCC) - percent RSD 20 percent maximum
. System Performance Check Compounds (SPCC) - 0.1minimum RF (0.3 for Chlorobenzene and 1,1,2,2-Tetrachloroethane)

All other compounds shall be reviewed for accuracy. Various clients request different criteria to be set for non-CCC compounds. This may be present is special instruction at login, or a QAPP.

11.0 QUALITY CONTROL

11.1 Method detection limit determination is required by this method annually. An MDL check is also required to be analyzed on each instrument running 8260.

11.2 Quantitation limits or practical quantitation limits (PQL) for this method are defined by the lowest calibration point in the calibration curve as well as meeting a 2 to 3 times the MDL criteria. RL's can vary with MDL updates.

11.3 Daily Performance Tests

11.3.1 Prior to initiating any data collection activities it is necessary to establish that a given GC/MS system meets the instrument performance criteria. This is accomplished through the analysis of 50 ng of p-bromofluorobenzene (BFB).

11.3.1.1 BFB must be analyzed at the start of every 12 hour sequence. 50 ng of BFB may be directly injected onto the GC column or purged in 5.0 ml of reagent water. BFB may be analyzed simultaneously with a calibration standard.

11.3.1.2 The key ions produced during the analysis of BFB and their respective ion abundance criteria are given in Table 3.0. This criteria must be met before any calibration standards, blanks, or samples may be analyzed.

11.3.1.3 Use the target program to verify that the BFB spectrum is within criteria. If it is not within criteria, the analyst may use enhancing or other acceptable practices to put BFB within criteria.

11.3.1.4 If the criteria is not met, the BFB must be reanalyzed. Repeated failure shall require the instrument to be manually tuned. After manual tuning, the BFB must be re-injected and the abundance criteria must be met before proceeding.

11.3.2 After the instrument performance criteria is met, the initial calibration curve must be verified through the analysis of a continuing calibration at 50 ug/L or 20 ug/L. The continuing calibration criteria must be met before any method blank or sample analyses may proceed.

11.3.3 A method blank consisting of 5ml of reagent water spiked with 25 ug/L of IS and SMC's must be analyzed every 12 hours after calibration criteria has been met. An acceptable method blank must meet the following criteria:

PQL's can vary on each project. The PQL's must be reviewed prior to sample analysis It is essential that the analyst is aware of the specific requirements for each project.

The method blank can contain analytes up to the PQL for the target compounds, except methylene chloride and acetone which must be less than or equal to three times the PQL. Special projects may require lower detection limits than the PQL. In these cases the method blank must not contain compounds over the client requested detection limit.
.
Sample analysis may not proceed until the above method blank criteria have been met.

All volatile analyses associated with a method blank that does not meet the above requirements must be repurged, reanalyzed, and reported.

11.4 Matrix Spike, Matrix Spike Duplicates and Matrix Spike Blanks

11.4.1 An MS/MSD must be analyzed for each group of samples of a similar matrix within each case, 20 samples, group of samples of a similar concentration level (soils only), or each 7 calendar day period; whichever is more frequent. MSB's are required for NYSDEC protocols.

11.4.2 The limits for matrix spike compound recovery and relative percent difference (RPD) are given in Table 4.0. These limits are only advisory; therefore, no further action is required if the criteria limits are not achieved. However, frequent failures shall be investigated for possible laboratory generated error. Surrogate recovery can be outside of the laboratory generated windows in the MS/MSD/020ppb_QCS.

11.5 QC Check Samples are applicable to this method. This solution is run at a concentration of 20ppb for 8260B or 10ppb for 8260LL. This QCS is run after the daily method blank and before sample analysis. The 20ppb_QCS recoveries must fall within the laboratory generated guidelines. (see table 9.0 for water limits and 9.0a for soil limits) Up to four compounds can be outside of the recovery windows. If TCLP samples are being analyzed the analyst must use the TCLP blank fluid for the 20ppb spike. This will be called a 20ppb_LCS instead of 20ppb_QCS. If a reduced compound list is being analyzed, only the target compounds of concern must meet criteria. Various miscellaneous compounds are not included in the independent source QCS and therefore not controlled.

11.6 System Monitoring Compounds

11.6.1 SMC's are added to each sample, blank, standard, QCS and MS/MSD/MSB, prior to purging or extracting at 25 ug/L for waters, 25 ug/Kg for low level soils, and 2500 ug/Kg

for medium level soils.

11.6.2 SMC recoveries must be within the QC limits given in Table 5.0. If the recovery for any one SMC is not within limits, the following are required:

. Check all calculations for accuracy, spiking solutions, and internal standards

. Reanalyze the sample if none of the above steps reveal a problem

. If an undiluted analysis with acceptable SMC recoveries is being submitted, do not reanalyze diluted samples if the SMC recoveries are outside the limits

. Never reanalyze the FMS, FMSD,or the 020ppb_QCS even if the SMC recoveries are outside the limits

. If the sample associated with the MS/MSD does not meet specifications, it should be reanalyzed only if the MS/MSD SMC recoveries are within the limits. Document in the narrative the similarity in the SMC recoveries between the sample and associated MS/MSD.

If the reanalysis of the sample solves the problem, then only submit the second analysis. If the reanalysis does not solve the problem, then submit the data from both analyses.

11.6.3 If the recovery of any one SMC in a method blank is outside limits, then the method blank and all associated samples must be reanalyzed.

11.7 Internal Standards

11.8.1 IS's are added to each sample, blank, standard, and MS/MSD/MSB, at 25 ug/L at the time of purging.

11.8.2 The retention times (RT) and extracted ion current profile (EICP) of each IS must be evaluated for all standards immediately after the data acquisition. The IS EICP areas must be monitored and evaluated for each sample, blank, MS, MSD. If the IS EICP changes by more than a factor of 2 (-50% to +100%) from the latest (12 hour) calibration standard, the GC/MS system must be inspected for malfunctions, and corrections made as required. If the RT for any IS changes by more than 30 seconds from the latest (12 hour) calibration standard, the chromatographic system must be inspected for malfunctions, and corrections made as required. For samples analyzed within the same 12 hour time period as the initial calibration standards, compare the IS responses and RT's against the 50 ug/L calibration standard. When corrections are made, reanalysis of the samples analyzed while the system was malfunctioning is necessary.

If IS criteria is not within limits, the following are required:

. Check all calculations for accuracy, spiking solutions, and internal standards

. Reanalyze the sample if none of the above steps reveal a problem

. If the sample associated with the MS/MSD does not meet specifications, it should be reanalyzed only if the MS/MSD IS criteria is within the limits

. If the reanalysis of the sample solves the problem, then only submit the second analysis. If the reanalysis does not solve the problem, then submit the data from both analyses.

11.8 Quality Control Check Points

11.8.1 Analysis quality control approval report

Specific quality control check points have been established for the analysis of samples which are monitored through a Quality Control Approval Report (QCAR). The specific check points must be initialed and dated by the analyst to ensure the consistency and accuracy of the data produced. Refer to Figure 1.0 for the QCAR and specific control points covered.

11.8.2 Specific quality control check points have been established for the preparation of data deliverables which are monitored through a Quality Control Approval Report (QCAR). The specific check points must be initialed and dated by the analyst to ensure the consistency and accuracy of the data produced. Refer to Figure 1.1 for the QCAR and specific control points covered.

11.9 Analytical Documentation Procedures

11.9.1 Instrument batches

An instrument batch is created for each analytical sequence to organize all the associated data. Batch designations are of the format:

Xyynnnn.b

Where:

X = instrument identifier
Yy= last two digits of year

nnnn = file number of first calibration check standard in the batch for that day.

(i.e. T030012.b)

Instrument batches are number according to daily calibration check standard file. Therefore, the unique batch identifier can identify each analytical sequence. The batch consists of a file folder with all the associated QC information for the analytical sequence. The raw data is then bound together with the file folder to complete the batch. The batch, including the electronic logbook page will be scanned and electronically stored for future retrieval.

11.9.2 Filing system

All active batches are filed chronologically according to instrument. The batches are transferred to file boxes for long-term storage once all the associated data within a batch has been completed.

11.9.3 Data archiving

All data files, including the BFB analysis data file, are archived daily using the Target server back-up system. Care shall be exercised when purging data off the hard drives. To ensure that all data being purged has been archived, check latest archive date prior to purging any data.

11.9.4 Instrument run logs

It is TESTAMERICA's policy that all measurement data be recorded in logbooks with black ink, or electronic logbooks that are bound after analysis. Transcriptions shall be avoided whenever possible. The record shall reflect the measurement performed and all appropriate details for conclusions related to the measurement. The record shall be signed and dated by the individual performing the measurement on the day the measurement is performed. Corrections shall be made by drawing a single line through the error, and initialing and dating the correction. A secondary authorization of the logbook is required and shall be performed by the department's manager or designee.

Currently in Volatiles, each instrument has its own three ring binder to maintain the electronic logbook pages until 90 pages are accumulated, then the logbook pages are given to QC to bind. All pages are sequentially numbered and paginated and secondary review done prior to daily scanning. Current run logs are held in the laboratory until they have been filled, for future reference. Older run logs shall be given to the QC officer for archiving. Each analytical sequence shall be started on a new page of the log and continued on the next page, if necessary. The header information designating the standard codes used shall be completed for each sequence. All standards used are recorded in this field for future traceability. The data file, job number, sample number, quantitation factor, dilution factor, analyst's signature, and date are recorded. Refer to Figure 2.0.

11.9.5 Initial data review sheet (short exception report on the target system)

The initial data review sheet (IDRS) is a computerized review sheet, which is used to check the key quality control criteria for compliance. The IDRS is used to check that all samples have been analyzed with the required calibration time frame. The IDRS is also used as the initial data review tool. Each sample is listed on the sheet and it is either accepted or rejected in the right hand column, by the analyst performing the data review. If reruns are required for dilutions, then the analyst shall indicate the proper dilution required for reanalysis. The data reviewer initial's and dates the IDRS. The batch is then filed when completed for use during deliverables preparation. Refer to Figure 3.0 for an example of the IDRS.

11.9.6 Electronic Corrective action reports need to be issued in a timely fashion. Non Conformance Module in the Lims system is used for quality control issues that need to be addressed in the Sample delivery group Narrative.

The Non - conformance report (NCM) is issued when a problem is encountered during analysis, data reduction or deliverables preparation, data validation, or when any deviations from this SOP occur. The NCM is prepared by the analyst first identifying the problem and is then electronically submitted to the department's manager for approval. The manager will review the NCM via e-mail.

Currently, for items beyond the realm of the NCM will require a corrective action report (CAR) this is for miscellaneous items the can not be covered by an NCM. The CAR is then redistributed to all the departments and individuals involved. Refer to Figure 4.0.

11.9.7 Chain of custody record

When samples are removed from storage for preparation or analysis they must be signed out utilizing the chain of custody record (COC). The samples shall then be signed back in on the COC upon their return to storage or designated "used" if the sample volume is consumed during the preparation or analysis.

11.9.8 Sample tracking record

Samples are tracked on the TestAmerica Lims Status reports. These reports are generated by the analyst as needed. Sample backlogs are generated often to ensure all samples are accounted for by each protocol. When samples are analyzed the analyst will update the Status report with the corresponding data files for each sample, and any relevant comments needed to assist reporting the data. NCM numbers will be recorded on the Status sheets to ensure all information is passed on to the client correctly. Samples requiring reanalysis are also documented as to the reanalysis dilution required, if any.

12.0 SAMPLE PREPARATION AND INSTRUMENTAL PROCEDURES

12.1 Instrumental Conditions

Instrument conditions will vary instrument to instrument, below is an example of the conditions. If the information is in bold print, then this condition will not vary due to method restrictions.

12.1.1 Purge & Trap Device

Purge Conditions:

	(VOCARB)
Purge Gas:	Helium
Purge Time:	11.0 min
Purge Flow:	40 ml/min
Dry Purge:	0.5 min
Purge Temp:	Ambient for LLW and MLS

Desorb Conditions:

	(VOCARB)
Desorb Temp:	250°C
Desorb Flow:	15 ml/min
Desorb Time:	0.5 min

Trap Reconditioning Conditions:

	(VOCARB)
Reconditioning Temp:	270°C
Reconditioning Time:	8.0 min

12.1.2 Gas Chromatograph

Carrier Gas:	Helium
Flow Rate:	5 ml/min
Initial Temp.:	30°C
Initial Hold:	4 min
Ramp Rate 1:	5°C/min
Second Temp.:	100°C
Ramp Rate 2:	120°C/min
Final Temp.:	200°C
Final Hold:	1.0 min
Transfer Temp:	185°C

12.1.3 Mass Spectrometer

Electron Energy:	70 eV
Mass Range:	35 - 300 amu
Scan Time:	less than 1 sec/scan

The mass spectrometer must be tuned to meet the instrument performance check criteria for 50 ng of BFB listed in Table 3.0.

12.2 Sample Analysis Procedures

12.2.1 Water Samples (using guidance from method 5030B)

Samples are removed from Sample Control storage and are signed out in the chain of custody form. The chain of custody is a legal document, therefore there ensure all samples are signed out prior to leaving sample control. If a sample is brought to you directly from sample control ensure that the sample was signed out at that time.

All samples are allowed to warm to room temperature.

Make sure all instrumental operating conditions are correctly set and BFB, calibration and blank criteria have been met.

In a gas tight 5 ml syringe, load a 5 ml aliquot of sample (or 25 ml depending on request) by drawing up past the 5ml line on the syringe, then bring to volume. The sample that is expelled from the syringe to waste can be used to check and record the pH in the logbook. This procedure destroys the integrity of the sample for future analysis, therefore, if there is only one vial, the analyst shall fill a second gas tight syringe in the same manner. This second syringe is maintained only until such time as the analyst has determined that the first sample has been properly analyzed. If an analysis is required from the second syringe, it must be performed within 24 hours. Care must be taken to prevent air from leaking into the syringe during storage.

If the sample is being loaded on a Archon auto sampler, just remove excess label material from vial so the vial slips into the auto sampler position with ease, remove any label that may be on the cap to ensure the robotic arm won't stick to the label. Load vials and record client id's from actual vial into the logbook. Then program the Archon for correct method to run sequence. Internal standards and surrogates can be added automatically by Archon, or spiked at correct concentration into each vial. Record pH in logbook .

For 5ml purge using a syringe to load sample, spike the sample with 5 uL of the IS and SMC 25ug/ml spiking solution utilizing a 25 uL gas-tight, through the open syringe valve, then load the syringe contents into the sparging vessel. If an autosampler is utilized, set up the autosampler start and stop positions for the sequence being purged.

Inject the sample into the purging chamber and purge at ambient temperature. After purging, the sample is thermally desorbed onto the GC column.

While the trap is being desorbed into the GC, empty the purging chamber either manually or with the autodrain option. Wash the chamber with a minimum of two 5 ml flushes of reagent water to avoid carryover of target compounds.

The trap is then reconditioned while the sample is temperature programmed on the GC to separate the volatile organics.

When a sample has been analyzed that has saturated ions from a compound, sample analysis may not resume until a blank has been analyzed that demonstrates that the system is free of interferences. Once the system is free of interferences, the sample that saturated the detector must be diluted and reanalyzed.

If a sample is analyzed which contains target compounds at concentrations greater than the initial calibration upper limit, but not saturated, then the sample must be reanalyzed at an appropriate dilution. The purge and trap system shall be demonstrated to be free from carry-over through the subsequent analyses of blanks and/or samples which do not contain the target compound at a concentration greater than the PQL.

When the system is run unattended, using the autosampler, if a sample is analyzed which contains target compounds at concentrations greater than the initial calibration upper limit, then the sample must be reanalyzed at an appropriate dilution. The samples analyzed subsequently shall be carefully evaluated. If any subsequent analyses contains the target compounds which were at concentrations greater than the initial calibration upper limit in the previous sample, at a concentrations greater than the PQL, then those samples must be reanalyzed once the system has been decontaminated and shown to free of interferences.

A water FMS/FMSD (FMSB-NYSDEC) is prepared by spiking the sample aliquot with 44 uL of calibration solution using a 50 uL gas-tight syringe. The spiked sample is then analyzed as previously described.

A method blank must be analyzed every 12 hours after the calibration criteria has been achieved. The method blank consists of 5 ml reagent water spiked with 5 uL of IS and SMC, and carried through the analytical procedure. Method blank criteria is defined in section 11.3.3.

12.2.2 Low Level Soil Samples (using guidance from method 5035)

The type of sample container that was used by the client must be determined at time of sample receipt. A hermetically sealed voa vial with preservative can be held for 14 days from collection. A sealed soil plug sample such as the EnCore sampler must be either transferred to a voa vial with 5mls of a Sodium Bisulfate solution, (**Note: using Sodium Bisulfate as a preservative has caused a "false" elevated level of acetone to be present due to a reaction between the soil and sodium Bisulfate. It is recommended to use reagent water if Acetone is a compound of concern on the site**) or reagent water

and a magnetic stir bar, or analyzed within 48 hours from collection. If reagent water is used the vial must be frozen at a 45 degree angle to avoid cracking of the glass vial. This can be held for 14 days from collection. Ensure vial comes to room temperature prior to analysis. TESTAMERICA Connecticut prefers to use reagent water, since reactions can occur when adding soil sample to Sodium Bisulfide that cause moderate levels of Acetone to be falsely detected.

Samples are removed from Sample Control storage and are signed out on the chain of custody form. If the soil is received in a sealed plug type sampler such as the EnCore sampler, then the plug is transferred to a pre-weighed voa vial containing 5mls of the Sodium Bisulfate solution, or regent water, and a magnetic stir bar. The vial is sealed and not opened until time of disposal. The vial is then re-weighed and the weight is recorded on the vial along with the subtracted weight that determines the soil weight. The client ID is transferred along with the laboratory ID onto the voa vial. If QC has been requested on a particular sample the vials are labeled "MS" or "MSD". Once the soil is transferred and the above steps have been performed, the soil is placed in the volatile refrigerator for storage (freezer at 45 degree angle for reagent water vials).

Reagent water vial with stir bar will be used if the soil sample effervesces when it comes in contact with the Sodium Bisulfate solution. If the soil is place in water, this sample will either be run that day, or frozen at a 45 degree angle to prevent breakage, for up to 14 days.

The client / client service, must specify at time of bottle order if no preservative is required. Encore containers or equivalent may be used for low level soil analysis. If these are used the soil plug must be transferred within 48 hours from collection. Arrangements must be made prior to shipping Encores to the laboratory for weekend transferring of the soil plugs.

All samples are allowed to warm to room temperature. All soils must be re-weighed prior to analysis to determine weight of sample.

Make sure all instrumental operating conditions are correctly set and BFB, calibration and blank criteria have been met.

The sample consists of the entire contents of the sample container. The voa vial is not to be opened. This sample is then loaded on the Archon autosampler. The internal standards and surrogates along with 5mls of reagent water will be added automatically by the Archon, or by analyst, prior to the sample pre-heat mode. Then the contents of the voa vial will be purged onto the Voacarb 3000 trap, and analyzed by the Mass Spectrometer. Then the trap is reconditioned by heating.

Surrogates are spiked in all curve points, continuing calibrations, Laboratory control spikes (LCS's) method blanks and samples at 25ppb for low level soils.

A percent moisture determination is performed by weighing out 5 grams soil from a separate container that has been submitted for percent solid determination. The soil shall dry overnight at 105 degrees Celsius. Allow to the sample to cool before weighing the sample back. The percent moisture is determined according to the following equation:

$$\frac{\text{g of wet sample - g of dry sample}}{\text{g of wet sample}} \times 100$$

If a sample is analyzed which contains target compounds at concentrations greater than the initial calibration upper limit, then the high concentration method must be utilized, unless the client sent other sealed voa vials with a lower sample volume that would be within the linear calibration range. Then the sample must be reanalyzed at an appropriate dilution. No less than 1.0 gram of sample may be analyzed by the low level method (i.e. a 5 fold dilution) utilizing the low level soil method. If a larger dilution is required, then the medium level soil method must be employed. With Encore type samplers, dilutions can not easily be made. Most analyses that have compounds recovering over 200ppb will have to be run as medium level soils, unless a portion of sample, no less than one gram was transferred from the Encore type sampler within the 48 hour holding time and analyzed as a low level soil.

The purge and trap system shall be demonstrated to be free from carryover through the subsequent analyses of blanks and/or samples which do not contain the target compound at a concentration greater than the PQL.(see section 9.2 for detail)

A low level soil FMS/FMSD (FMSB-NYSDEC) is prepared by spiking 50ppb of the calibration standard into two separate sample vials. The voa vials are not to be opened. This sample is then loaded on the Archon autosampler. The internal standards and surrogates along with 5mls of reagent water will be added automatically by the Archon prior to the sample pre-heat mode. Then the voa vial will be purged onto the Voacarb 3000 trap. Then analyzed by the Mass Spectrometer.

A low level soil method blank must be analyzed every 12 hours after the calibration criteria has been achieved. The method blank consists of 5.0 grams of a purified solid matrix added to 5.0 ml of reagent water and a magnetic stir bar. The method blank is then loaded on the Archon and automatically spiked with internal standards , surrogates ,and 5mls of reagent water then carried through the analytical procedure. Method blank criteria is defined in section 11.3.3.

12.2.3 Medium Level Soil Samples

Samples are removed from the Sample Control storage area and signed out on the chain of custody form.

All samples are allowed to warm to room temperature.

Make sure all instrumental operating conditions are correctly set and BFB, calibration and blank criteria have been met.

12.2.3.1 Laboratory Preserved

More than one scenario may take place with high concentration soil samples. Review the client's Quality Assurance Plan prior to preparing samples to ensure there are no other site requirements for preparing the methanol extracts. If the client expected high levels of volatiles in the field, then the samples were collected in the field preserved in methanol. The client may use the EnCore sampler or equivalent, therefore the plug of soil from the EnCore sampler is placed into the pre-weighed voa jar containing 10mls of purge and trap grade methanol. Record the weight of the jar, soil and methanol. Record the soil weight on the jar. Shake the jar for 2 minutes, then let the contents settle and transfer 1-2mls of the methanol extract into a extract vial. Store extract until time of analysis. It is also possible that a low level soil is actually a high level soil. Therefore the methanol extraction is performed at the laboratory. If the sample is extracted at the laboratory, then extract as follows:

The sample consists of the entire contents of the sample container. Do not discard any supernatant liquids. Mix the contents of the sample container with a narrow metal spatula. A medium level soil extract is prepared by weighing out 5.0 grams of sample into a 20 ml extraction vial, record the weight to the nearest 0.1 gram. Determine the percent moisture as in the low level method. Quickly add 10.0 ml of purge and trap grade methanol to the vial. Cap and shake for 2 minutes.

Using a disposable pipette, transfer about 1 ml of sample extract to a GC vial for storage. The remainder of the extract may be discarded. Transfer about 1 ml of the reagent methanol to a GC vial for use as the method blank. These extracts may be stored in the dark at 4 degrees Celsius (+/- 2 degrees) prior to analysis.

In a gas tight 5 ml syringe, load a 5 ml aliquot of reagent water and spike with 5 uL of IS/Surrogate spiking solution (25ppb on column concentration) using a 25 uL gas-tight syringe, and 100 ul of sample using a 250 uL gas tight syringe, extract and purge for 11.0 minutes at ambient temperature. If an autosampler is utilized, set up the autosampler start and stop positions for the sequence being purged. After purging, desorb onto the GC column.

The medium level soil extract is analyzed under a water initial and continuing calibration.(using guidance form method 5030B)

The trap is then reconditioned at 270 degrees Celsius for 6-8 minutes while the sample is temperature programmed on the GC to separate the volatile organics.

If an extract is analyzed which contains target compounds at concentrations greater than the initial calibration upper limit, then the extract must be reanalyzed at an appropriate

dilution. Volumes of less than 10 ul (i.e. 10 fold MLS dilution) shall be prepared diluting an aliquot of the methanol extract and then taking 100 uL for analysis. Add the volume of methanol extract from the sample and a volume of clean methanol to total 100 ul. The total methanol volume added shall be 100 ul, excluding the methanol in the standards.

The purge and trap system shall be demonstrated to be free from carry-over through the subsequent analyses of blanks and/or samples which do not contain the target compound at a concentration greater than the PQL.

A medium level soil FMS/FMSD is prepared by spiking a 5.0 gram sample with 10.0 mls of purge and trap methanol. 880ul of the sample is added to the 44ml voa vial and then spiked with 44ul of calibration check standard. The spiked sample extract is then analyzed as previously described.

A method blank must be analyzed every 12 hours after the calibration criteria has been achieved. The method blank consists of 10.0 ml of reagent methanol. A 100 uL aliquot of the method blank extract is then spiked into 5 ml reagent water fortified with 5 uL IS/Surrogate solution. The method blank is then carried through the analytical procedure as described previously. Method blank criteria is defined in section 11.3.3.

12.2.3.2 Field Preserved

If high volatile concentrations are expected in the soil samples a different sampling method is required. The soil vial will contain methanol, be pre-weighed and labeled, prior to shipping to the client (Some states require surrogates to be pre-spiked in the methanol prior to sample collection. This can be done when requested. The laboratory must be notified for special requests) There must also be an empty soil vial for total solids determination in the laboratory. Five grams of soil will be added to the methanol vial in the field and weights recorded on the chain of custody, vials or other appropriate locations.

Oily waste samples will be collected in empty vials (unless known to be soluble in methanol or polyethylene glycol (PEG) * PEG must be requested when placing bottle orders otherwise vials will contain methanol. The laboratory will weigh the voa vial prior to analysis, record the weight in the logbook, check to ensure the methanol and soil mixture is still present. If not a corrective action must be written to notify client of possible re-sampling, or another acceptable solution. The sample is then shaken and allowed to settle. A portion of the diluted sample is then removed from the vial and analyzed by method 5030B.(see section 11.2.1) Total solids, sample weight, and sample volume used, all need to be recorded to determine correct dilution factor for quantitation.

The default volume of methanol for TESTAMERICA Connecticut is 10ml without surrogates if volumes are not specified by client

12.3 Qualitative Analysis

12.3.1 Target Compounds

The relative retention time of a target compound must be within +/- 0.06 RRT units of the RRT of the calibration standard for a positive identification. For reference the standard must be analyzed within the same 12 hour time period as the sample. If coelution of interfering components prohibits accurate assignment of the sample component RRT from the total ion chromatogram, the RRT shall be assigned by using the extracted ion current profiles for ions unique to the component of interest.

In addition, a comparison must be made between the mass spectrum obtained in the sample analysis and the reference mass spectrum for that compound, which was obtained on that specific GC/MS system. The requirements for qualitative verification by comparison of mass spectra are as follows:

All ions present in the reference spectrum at an intensity greater than 10% must be present in the sample spectrum.

The relative intensities of the ions above 10% must agree with 20% between the reference and sample spectra.

Ions greater than 10% in the sample spectrum but not present in the reference spectrum must be considered and accounted for by the analyst.

If a compound cannot be verified by the above criteria, but in the technical judgement of the analyst, the identification is correct, then the compound shall be reported.

12.3.2 Tentatively Identified Compounds

If requested a library search shall be performed for non-target compounds in the sample for purposes of tentative identification. For this purpose, the most recent release of the NIST mass spectral library shall be used.

Up to 10 organic compounds of apparent concentration not listed in Table 1.0, shall be tentatively identified via a forward library search. Only compounds with responses greater than 10% of the closest IS exhibiting no interference are to be searched.

The Target software is utilized to perform the automated library search. The program (TICS) is executed with the data file, method, and number of compounds to be searched, specified for each sample or blank. Prior to running the program, the analyst must delete from the quantitation file, using the Target review program, the non-TCL compounds which were identified in the quantitation file. This will facilitate their automated search using the program. If the non-TCL positive hits are not removed prior to executing the program, they would be counted as target compounds and not be searched by the program, leading to false negatives.

A tentative identification will be made after a comparison between the mass spectrum obtained in the sample analysis and the library search mass spectra found for that compound. The requirements for tentative verification by comparison of mass spectra are as follows:

Ions present in the reference spectrum at an intensity greater than 10% should be present in the sample spectrum.

The relative intensities of the ions above 10% should agree with 20% between the reference and sample spectra.

Ions present in the sample spectrum but not in the reference spectrum should be reviewed for possible background contamination or coeluting compounds.

Ions present in the reference spectrum but not in the sample spectrum should be reviewed for possible background subtraction by the data system.

If in the technical judgement of the mass spectral interpretation specialist, no valid tentative identification can be made, the compound shall be reported as unknown. Additional classification shall be made if possible (i.e. Unknown hydrocarbon).

12.4 Quantitative Analysis

12.4.1 Target Compounds

Target compounds are quantitated by the internal standard technique. The associated internal standard used is listed in Table 6.0. The EICP area of the quantitation ions of compounds listed in Tables 8.0 are used. The quantitation ion for the SMC compound bromofluorobenzene can be m/z 174 instead of m/z 95 due the co-elution of the target compound 1,1,2,2-tetrachloroethane, which interferes with m/z 95. Other co-eluters that must be inspected are listed in Figure 7.0.

The relative response factor (RRF) from the initial calibration standard or the equation for linear regression is used to calculate the concentration in the sample depending on the percent RSD in the calibration curve. When compound concentrations are below the PQL, but the compound meets identification criteria, report the concentration with a "J" qualifier.

Water Samples

Concentration

$$ug/L = \frac{(Ax)\,(Is)\,(Df)}{(Ais)\,(RRF)\,(Vo)}$$

where,

Ax = area of the compound quantitation ion

Ais = area of IS quantitation ion

Is = IS amount in nanograms

RRF = Average Relative response factor from the ambient temperature purge of the Initial calibration curve (Linear Regression may replace this equation if the mean response for all the compounds in the method is above 15% RSD in initial calibration)

Vo = volume of water purged in ml's

Df = Dilution factor. The dilution factor for analysis of water samples for volatiles by this method is defined as the ration 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 2.5 ml of sample is diluted to 5.0 ml with reagent water and purged, DF=5.0 ml/2.5 ml = 2.0. If no dilution is performed, Df = 1.0.

Low Level Soil Samples

Concentration
(dry weight basis)

$$ug/Kg = \frac{(Ax)(Is)}{(Ais)(RRF)(Ws)(D)}$$

where,

Ax, Is, and Ais are as given for water.

$$D = \frac{100 - \%\ moisture}{100}$$

Ws = weight of sample added in grams

RRF = Average Relative response factor from the heated temperature purge of the Initial calibration curve. (Linear Regression may replace this equation if the mean response for all compounds in the method is above 15% RSD in initial calibration)

Medium Level Soil Samples

Concentration
(dry weight basis)

$$ug/Kg = \frac{(Ax)(Is)(Vt)(1000)(Df)}{(Ais)(RRF)(Va)(Ws)(D)}$$

where,

Ax, Is, Ais and RRF are as given for water.

$$D = \frac{100 - \%\ moisture}{100}$$

RRF = Average Relative response factor from the ambient temperature purge of the initial calibration curve. (Linear Regression may replace this equation if the mean response for all compounds in the method is above 15% RSD in initial calibration)

Ws = weight of sample extracted in grams

Vt = total volume of methanol extract in ml

Va = volume of the methanol extract added to the reagent water for purging in ul

Df = Dilution factor. The dilution factor for medium level soils is defined as the ratio of the number of microliters (uL) of methanol added to the reagent water for purging (i.e. Va above) to the number of uL of the methanol extract of the sample contained in that volume Va. The dilution factor is equal to one in all cases other than those requiring dilution of the methanol extract.

12.4.2 Linear Fit

$$Conc = A + B\frac{(R_x C_{is})}{R_{is}}$$

Conc = Concentration in ug/l
R_x = Response for analyte(area of quantitation ion)
R_{is} = Response for internal standard(area of quantitation ion)
C_{is} = Concentration of internal standard
A = Intercept
B = Slope

The corresponding Target software calculation is as follows:

$$Conc = C_{is}\left(b + \frac{1}{m1} \times \frac{R_x}{R_{is}} \right)$$

b = Concentration Ratio Intercept
m1 = Inverse of slope

12.4.3 Quadratic fit

$$Conc = A + B\left(\frac{R_x C_{is}}{R_{is}}\right) + C\left(\frac{R_x C_{is}}{R_{is}}\right)^2$$

C = Curvature

The corresponding Target software calculation is as follows:

$$Conc = C_{is}\left(b + m1 \times \frac{R_x}{R_{is}} + m2 \times \left(\frac{R_x}{R_{is}}\right)^2 \right)$$

m1 = First order coefficient
m2 = Curvature(Second order coefficient)

12.4.4 The concentration in the sample is then calculated.

12.4.5 Tentatively Identified Compounds

An estimated concentration for non-target compounds tentatively identified in the sample shall be determined by the internal standard method. For quantitation, the nearest IS free of interferences shall be used.

The equation for calculating concentrations are the same as in 12.4.1. Total area counts from the total ion chromatograms are used for both the IS and compound. A RRF of 1.0 is assumed and the resulting concentration shall be qualified as "J" (estimated), indicating the quantitative and qualitative uncertainties associated with this non-target compound.

12.4.6 The three Xylene isomers are to be reported as Xylenes (total). The meta and para isomers coelute on the capillary column, therefore, special attention must be given to their quantitation. The two reponse factors (RF) for the three Xylene isomers are totaled divided by two and an average RF is used for quantitation.

12.4.7 The cis and trans isomers of 1,2-Dichloroethene are to be reported as 1,2-Dichloroethene (total). The two isomers do not coelute on the capillary, therefore, the RRF is determined by summing the two isomer areas and then dividing by the total isomer concentrations. The area from both peaks and this RRF are then used to quantitate the 1,2-Dichloroethene (total) concentration. Both isomers must be present in the initial and continuing

calibration standards.

12.4.8 If the on-column concentration of any compound in any sample exceeds the initial calibration range, a new aliquot of that sample must be diluted and purged. Guidance in performing dilutions, and exceptions to this requirement are as follows:

12.4.9 Use the results of the original analysis to determine the approximate dilution factor required to get the largest analyte peak within the initial calibration range.

12.4.10 The dilution factor chosen shall keep the response of the largest analyte peak for a target compound in the upper half of the initial calibration range of the instrument.

12.4.11 Data for more than two analyses shall not be submitted.

12.4.12 Run associated sample at correct dilution prior to running FMS/FMSD. It is not necessary to re-run a FMS/FMSD, or 020ppb_QCS if internal standards and or surrogates are out of criteria. Or spike compounds are outside of recovery windows. However; FMSB's Must meet Internal standard and surrogate criteria. Since a Full Matrix spike is used, the likely hood of all compounds being within the acceptable range are slim. Therefore, note the compounds outside of the laboratory windows in the SDG narrative.

12.5 Instrument Maintenance

12.5.1 Preventative maintenance

TestAmerica retains an outside service vendor, and utilizes personnel trained in preventative maintenance. Preventative maintenance is performed at scheduled intervals on all equipment. All instrument preventative maintenance is performed according to the manufacturers recommended procedures, by trained personnel. All preventative maintenance shall be thoroughly documented in the maintenance log, as to a description of the maintenance performed, the date performed, and the personnel performing the maintenance. Upon returning the system into control, a cross-reference batch number shall be recorded in the maintenance log. This batch number will demonstrate that a successful repair has been achieved.

12.5.2 Corrective maintenance determinants and procedures

Corrective maintenance is deemed necessary when the analytical system, after reanalysis, cannot meet tune, calibration, or other protocol specific QC criteria. Corrective maintenance may include, but is not limited to, decontamination of the system, injection port cutting and cleaning, source cleaning, replacing the electron multiplier, column replacement, jet separator cleaning or replacement, or filament replacement. All corrective maintenance is performed according the manufacturers recommended procedures, by trained personnel. All corrective maintenance shall be thoroughly documented in the maintenance log, as to a description of the maintenance performed, the

date performed, and the personnel performing the maintenance.

12.5.3 Maintenance authorization

All preventative and corrective maintenance is authorized by the department's manager, or designee. When outside maintenance is deemed necessary, a service call is placed for all equipment covered under a service contract, by the department's manager, or designee.

12.6 Data System

12.6.1 Data Acquisition and System Operation

Data is acquired from sample analyses using the Target software. Analytical batches are set up with all the associated sample ID, dilution, and data file information. Automated post-acquisition quantitation is qued with the appropriate method, as well as, post-acquisition archiving of the data file. The sequence is assigned and started using the ChemStation software.

12.6.2 Instrument errors

System errors are logged to the ChemStation error logs. The system manager shall be responsible for checking and providing corrective actions for all major system errors. Minor system errors, such as insufficient disk space, are handled by trained analysts, as necessary.

12.6.3 Manual Integrations and Editing Flags

Manual integrations are required when the automated software doesn't correctly integrate extracted ion current profiles (EICP). A user shall be logged into the Target system as their own name. This name will signify who performed the manual integration. To perform a manual integration, the target compound of interest is selected and the EICPs are graphically presented. The peak can then be correctly integrated. A reason code shall be selected for the type of integration performed by selecting Review codes from the menu options.

UN = Unidentified peak based on spectra or concentration
ID = Identified a peak based on spectra
INT = Integrated a peak due to incorrect integration

A new quantitation report is produced. The manually integrated data file is the saved by exiting and saving from file edit. Manual integrations are flagged by the data system with the "M" qualifier beside any manually integrated area. The manually integrated data file is the saved by exiting from Target review. Manual integrations are flagged by the data system with the "M" qualifier beside any manually integrated area on the hardcopy quant report. The analyst name will appear on the electronic manual

integration report which is uploading to the LIMS system when data files are uploaded. A hardcopy print out of the EICP of the quant ion displaying the manual integration shall be produced for the before and after integrations and is included in the raw data to the clients when the report is generated during final packaging.

13.0 CALCULATIONS

13.1 Relative Response Factor (RRF)

$$RRF = \frac{(Ax)(Cis)}{(Ais)(Cx)}$$

where,

Ax = area of the compound quantitation ion

Ais = area of IS quantitation ion

Cis = IS concentration

Cx = compound concentration

An average RRF is calculated for each compound and SMC from the initial calibration.

The RRF used for quantitation is based upon the ortho-Xylene isomer peak. The area from both peaks and this RRF are then used to quantitate the Xylenes (total) concentration. All three isomers must be present in the initial and continuing calibration standards.

The RRF is used for 1,2-Dichloroethene is calculated by summing the two isomer areas and then dividing by the total isomer concentrations. The area from both peaks and this RRF are then used to quantitate the 1,2-Dichloroethene (total) concentration in a sample. Both isomers must be present in the initial and continuing calibration standards.

13.2 Percent Relative Standard Deviation (%RSD)

$$\%RSD = \frac{\text{standard deviation}}{\text{mean}} \times 100$$

13.3 Percent Difference (%D)

$$\%D = \frac{(\text{average RRFi}) - (\text{RRFc})}{(\text{average RRFi})} \times 100$$

where,

average RRFi = average RRF from the initial calibration

RRFc = RRF from the continuing calibration standard

13.4 Percent Moisture

$$\% \text{ moisture} = \frac{\text{g of wet sample - g of dry sample}}{\text{g of wet sample}} \times 100$$

13.5 Target Compound Concentrations

The calculations used to determine the target compound concentrations are described in section 11.4.

13.6 SMC Percent Recovery

$$\% \text{ Recovery} = \frac{\text{concentration found}}{\text{concentration spiked}} \times 100$$

13.7 Matrix Spike Recovery

$$\% \text{ Recovery} = \frac{SSR - SR}{SA} \times 100$$

where,

SSR = spiked sample result

SR = sample result

SA = spike added

13.8 Relative Percent Difference

$$RPD = \frac{\text{absolute } (MSR - MSDR)}{(½)(MSR+MSDR)} \times 100$$

where,
MSR = matrix spike recovery

MSDR = matrix spike duplicate recovery

The absolute value of the recovery difference is used in the above equation.

13.9 Adjusted Contract Required Quantitation Limit for Samples

$$\text{Adjusted PQL} = \frac{(\text{PQL}) \times \text{Df}}{\text{D}}$$

where,

$$\text{D} = \frac{100 - \%\ \text{moisture}}{100}$$

Df = the dilution factor

14.0 ACCEPTANCE OF DATA

14.1 Method Blank

The method blank can contain analytes up to the PQL for the target compounds, except methylene chloride and acetone which must be less than or equal to three times the PQL. Special projects may require lower detection limits than the PQL. In these cases the method blank must not contain compounds over the client requested detection limit.(See Table 10.0 for various method blank criteria for special projects.)

If a method blank exceeds the limits for contamination above, the laboratory shall consider the analytical system to be out of control. The source of the contamination must be investigated and appropriate corrective actions taken and documented before further sample analysis proceeds.

14.2 System Monitoring Compounds (SMC)

All SMC's must be within the recovery criteria listed in Table 5.0. Method blanks and samples with recoveries outside the required windows must be reanalyzed. Refer to section 11.6 for SMC information.

14.3 Instrument Performance Check

The criteria for bromofluorobenzene is listed in Table 3.0 and in section 11.3.1.

14.4 Internal Standards
The IS criteria is described in section 11.7.

14.5 Full Matrix Spike/Full Matrix Spike Duplicate

The FMS/FMSD criteria is described in section 11.4.

15.0 REPORTING OF RESULTS

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."

16.0 Pollution Prevention

16.1 Pollution prevention is the reduction and/or elimination of wastes or the toxicity of wastes at the point of generation. The laboratory strives to keep all wastes at a minimum through a variety of techniques. These include the following.

16.1.1 Inventory control: Rotate stocks (first in, first out) and only purchase reasonable amounts of chemicals. Do not purchase excessive quantities, which will be discarded as wastes at some future point.

16.1.2 Material Handling and Storage: Keep containers properly labeled. Keep solvents covered. Store and handle all chemicals, reagents, standards, etc. to prevent contamination and degradation.

16.1.3 Personnel: All staff must be trained in the proper handling and disposal of waste.

16.1.4 Waste Reduction: Reduce the volume of waste generated wherever possible.

16.1.5 Chemical /material substitution: Use less toxic chemicals whenever possible. Avoid the use of chromic acid (Chromerge) solutions.

17.0 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 Tracking anc Collection of Hazardous Waste SOP. The following waste streams are produced when this method is carried out.

17.1 All waste shall be managed in accordance with all state and federal requirements, TESTAMERICA Connecticut's Hazardous waste management plan, the RCRA Contingency Plan and the Corporate Safety Manual.

17.2 All personnel who handle or generate waste must be trained prior to handling waste.

- Aqueous waste water is put in screw cap 500ml bottles with sodium bisulfate to

neutralize water. This water can be put in 50 gallon drum for neutralizing and disposal as treated waste.

- Soil waste is initially stored in satellite waste area's below desks in the volatiles laboratory. This soil is then put into the non hazardous soil waste drums in the hazardous waste room.
- Methanol waste from medium level soils samples are initially accumulated and segregated from low level soil waste in the satellite waste area below the desks in the volatiles laboratory. This soil / methanol waste and vial is disposed of in flammable waste vials drum in the hazardous waste room.
- Outdated, expired and obsolete calibration standards are laboratory packed yearly.

18.0 SUPPLEMENTAL DOCUMENTS

18.1 Standard Operating Procedure Documentation Policy/Procedures Organics

18.2 SOP for Samples Processing Methods Performed at Sample Arrival

18.3 SOP for Log-In Methods for CLP Samples

18.4 SOP for Storing Water and Soil Samples for Organic and Inorganic Sample Analysis

18.5 SOP for Documenting Sample Removal from the Laboratory

18.6 Standard Operating Procedure for Sample Tracking

19.0 REFERENCES

19.1 Purge & Trap Method EPA SW846 3rd Edition, Methods 5030B/5035.

19.2 Volatile organics by Gas Chromatography/Mass Spectrometry (GC/MS) SW846 3rd Edition, Method 8260B.

19.3 EnChem, Inc 1795 Industrial Drive, Green Bay WI 54302 (920) 469-2436
EnChem produces the EnCore sampler a tool that one manually inserts into the soil to take a plug of soil sample. The sample is then capped and shipped to the laboratory. The laboratory then must remove the soil and analyze by either the 5035 soil method or a methanol extraction method (5030B) * soil samples must be analyzed within 48 hours after collection, or transferred to vials. (section 6.2.1.8 5035-10)

19.3 Associated Design and Manufacturing Company, 814 North Henry Street, Alexandria, VA 22314 (703) 549-5999 Purge and Trap Soil Sampler - (model 3780PT) this tool allows a sampler to take a soil sample plug and through the use of various adapters for the voa vial, put the plug of soil into a standard 40ml voa vial, then ship the voa vial and voa vial adapter to the laboratory for analysis by method 5035.

19.4 Becton Dickinson & Co Franklin Lakes NJ 07417-1884 (888) 237-2762 or VWR, 800) 932-5000 Sterile syringe (not cut). The end of the plastic syringe barrel is cut off, either in the field, or by the laboratory prior to shipping. The syringe is tarred out in the field on a portable balance prior to scooping up a sample. Five grams of soil sample is then collected in the syringe and put into a voa vial that has been pre-weighed, preserved with sodium bisulfate and contains a Teflon coated stir bar(all supplied by laboratory). Vials are sealed and samples are directly loaded onto an autosampler which pierces the septa of the voa vial therefore keeping the sample sealed and not opened by the laboratory. Four other vial should be collected in the same manner to ensure enough sample volume for repeated analyses at the laboratory. One empty voa vial should also be filled with soil for total solids determination, and to be used if the soil collected must be run as a medium level soil.

20.0 SUBSTANTIVE REVISIONS

20.1 Original issue; lab update to method 8260B - January 27, 1998.

20.2 Revision on: November 17, 1998. Changed Laboratory name in entire document from AEN to STL. Section 7.0; updated instrumentation and computer systems. Section 11.0; added method 5035 low level soil procedure.

20.3 Corrected section 9.4.1 item number 2 to state “below 15%”. Corrected header problem. - January 27, 1999.

20.4 Updated to reflect A.C.O.E. modifications March 19,1999
20.5 Updated to reflect NELAP added sections 3,16,17 renumbered SOP October 6, 1999
20.6 Updated after Laboratory Manager’s review 11/05/1999
20.7 Annual review of S.O.P. updated as per requirements of STL QC program 02/26/01.
20.8 Review of S.O.P. updated as per requirements of STL QC program 06/12/2003.
20.9 Updated to reflect changes in method 5035. 9/23/2003.
20.10 Updated to reflect low level water changes for 5973, and ACOE requirements 2/11/2005
20.11 General updates 2/16/2005
20.12 General updates 4/10/2007
20.13 General corrections found during group review of 8260B sop and updates to include TestAmerica name. 8/23/2007.

20.14 Removed Std Prep SOP reference, added sect 9.5 & 9.6 for reagent coding and storage requirements. Updated Safety, Pollution control & waste mgmt sections. Updated sect 12.6.3. for manual integration. Sect 4.3, added reference to analyte list and tables. Added table 2.1. Removed ACOE references. Removed Figure 5. Corrected typo's in document. 11/26/08. Section 10.3 removed initial calibration prep info from here and added Table 9.0. Section 10.4.1 #2 removed statement on inclusion of linear regression plot included in Raw data. Section 11.0 MDL frequence requirements updated. Section 11.2 RL determination clarified. Section 11.3.3 MB acceptance criteria clarified. Section12.5.1 modified .Section 10.2 modified. Section 11.3.1.1 modified. Section 10.2 modified. Added section 10.2.1. Updated 7.5, default volume. Removed Figure 6. 2/1/09

TABLE 1.0
TARGET COMPOUND LIST (TCL) AND ESTIMATED QUANTITATION LIMITS (EQL)

Volatile Organics	Quantitation Limits*			
	Water ug/L	Low Soil ug/Kg	Med. Soil ug/Kg	On Column ng
Chloromethane	10	10	1,000	(50)
Bromoethane	10	10	1,000	(50)
Vinyl Chloride	10	10	1,000	(50)
Chloroethane	10	10	1,000	(50)
Methylene Chloride	10	10	1,000	(50)
Acetone	10	10	1,000	(50)
Carbon Disulfide	5	5	500	(50)
1,1-Dichloroethene	5	5	500	(50)
1,1-Dichloroethane	5	5	500	(50)
1,2-Dichloroethene (total)	5	5	500	(50)
Chloroform	5	5	500	(50)
1,2-Dichloroethane	5	5	500	(50)

2-Butanone	10	10	1,000	(50)
1,1,1-Trichloroethane	5	5	500	(50)
Carbon Tetrachloride	5	5	500	(50)
Bromodichloromethane	5	5	500	(50)
1,2-Dichloropropene	5	5	500	(50)
cis-1,3-Dichloropropene	5	5	500	(50)
Trichloroethene	5	5	500	(50)
Dibromochloromethane	5	5	500	(50)
1,1,2-Trichloroethane	5	5	500	(50)
Benzene	5	5	500	(50)
trans-1,3-Dichloropropene	5	5	500	(50)
Bromoform	5	5	500	(50)
4-Methyl-2-pentanone	10	10	1,000	(50)
2-Hexanone	10	10	1,000	(50)
Tetrachloroethene	5	5	500	(50)
Toluene	5	5	500	(50)
1,1,2,2-Tetrachloroethane	5	5	500	(50)
Chlorobenzene	5	5	500	(50)
Ethylbenzene	5	5	500	(50)
Styrene	5	5	500	(50
Xylene (total)	5	5	500	(50)

***Quantitation limits listed for soil/sediment are based on wet weight. The quantitation limits calculated by the laboratory for soil/sediment, calculated on dry weight basis as required by the contract, will be higher.**

TABLE 1.1
8260B COMPOUND LIST AND ESTIMATED QUANTITATION LIMITS (EQL)

Volatile Organics	Quantitation Limits*			
	Water ug/L	Low Soil ug/Kg	Med. Soil ug/Kg	25 mL Purge ug/L
Benzene	10	10	1,000	(50)
Bromobenzene	10	10	1,000	(50)
Bromochloromethane	10	10	1,000	(50)
Bromodichloromethane	10	10	1,000	(50)
Bromoform	10	10	1,000	(50)
Bromomethane	10	10	1,000	(50)
n-Butylbenzene	5	5	500	(50)
sec-Butylbenzene	5	5	500	(50)
tert-Butylbenzene	5	5	500	(50)
Carbon Tetrachloride	5	5	500	(50)
Chlorobenzene	5	5	500	(50)
Chlorodibromomethane	5	5	500	(50)
Chloroethane	10	10	1,000	(50)
Chloroform	5	5	500	(50)
Chloromethane	5	5	500	(50)
2-Chlorotoluene	5	5	500	(50)
4-Chlorotoluene	5	5	500	(50)
1,2-Dibromo-3-chloropropane	5	5	500	(50)
1,2-Dibromoethane	5	5	500	(50)
Dibromomethane	5	5	500	(50)
1,2-Dichlorobenzene	5	5	500	(50)
1,3-Dichlorobenzene	5	5	500	(50)
1,4-Dichlorobenzene	5	5	500	(50)
Dichlorodifluoromethane	5	5	500	(50)
1,1-Dichloroethane	10	10	1,000	(50)
1,2-Dichloroethane	10	10	1,000	(50)
1,1-Dichloroethene	5	5	500	(50)
cis-1,2-Dichloroethene	5	5	500	(50)
trans-1,2-Dichloroethene	5	5	500	(50)
1,2-Dichloropropane	5	5	500	(50)

1,3-Dichloropropane	5	5	500	(50)
2,2-Dichloropropane	5	5	500	(50
1,1-Dichloropropene	5	5	500	(50)
Ethylbenzene	10	10	1,000	(50)
Hexachlorobutiene	10	10	1,000	(50)
Isopropylbenzene	10	10	1,000	(50)
p-Isopropyltoluene	10	10	1,000	(50)
Methylene Chloride	10	10	1,000	(50)
Naphthalene	10	10	1,000	(50)
n-Propylbenzene	5	5	500	(50)
Styrene	5	5	500	(50)
1,1,1,2-Tetrachloroethane	5	5	500	(50)
1,1,2,2-Tetrachloroethane	5	5	500	(50)
Tetrachloroethene	5	5	500	(50)
Toluene	5	5	500	(50)
1,2,3-Trichlorobenzene	10	10	1,000	(50)
1,2,4-Trichlorobenzene	5	5	500	(50)
1,1,1-Trichloroethane	5	5	500	(50)
1,1,2-Trichloroethane	5	5	500	(50)
Trichloroethene	5	5	500	(50)
Trichlorofluoromethane	5	5	500	(50)
1,2,3-Trichloropropane	5	5	500	(50)
1,2,4-Trimethylbenzene	5	5	500	(50)
1,3,5-Trimethylbenzene	5	5	500	(50)
Vinyl Chloride	5	5	500	(50)
o-Xylene	5	5	500	(50)
m-Xylene	5	5	500	(50)
p-Xylene	10	10	1,000	(50)

***Quantitation limits listed for soil/sediment are based on wet weight. The quantitation limits calculated by the laboratory for soil/sediment, calculated on dry weight basis as required by the contract, will be higher.**

TABLE 2.0
APPENDIX IX COMPOUND LIST AND ESTIMATED QUANTITATION LIMITS (EQL)

Volatile Organics	Quantitation Limits* **		
	Water ug/L	Low Soil ug/Kg	Med. Soil ug/Kg
Chloromethane	10	10	1,000
Bromomethane	10	10	1,000
Vinyl Chloride	10	10	1,000
Chloroethane	10	10	1,000
Methylene Chloride	5	5	500
Acetone	10	10	1,000
Carbon Disulfide	5	5	500
1,1-Dichloroethene	5	5	500
1,1-Dichloroethane	5	5	500
1,2-Dichloroethene (total)	5	5	500
Chloroform	5	5	500
1,2-Dichloroethane	5	5	500
2-Butanone	10	10	1,000
1,1,1-Trichloroethane	5	5	500
Carbon Tetrachloride	5	5	500
Vinyl Acetate	10	10	1,000
Bromodichloromethane	5	5	500
1,2-Dichloropropane	5	5	500
cis-1,3-Dichloropropene	5	5	500
Trichloroethene	5	5	500
Dibromochloromethane	5	5	500
1,1,2-Trichloroethane	5	5	500
Benzene	5	5	500
trans-1,3-Dichloropropene	5	5	500
Bromoform	5	5	500
4-Methyl-2-Pentanone	10	10	1,000
2-Hexanone	10	10	1,000
Tetrachloroethene	5	5	500
1,1,2,2-Tetrachloroethane	5	5	500

Toluene	**5**	**5**	**500**
Chlorobenzene	**5**	**5**	**500**
Ethylbenzene	**5**	**5**	**500**
Styrene	**5**	**5**	**500**

***Quantitation limits listed for soil/sediment are based on wet weight. The quantitation limits calculated by the laboratory for soil/sediment, calculated on dry weight basis as required by the contract, will be higher.**

TABLE 2.0 (continued)
APPENDIX IX COMPOUND LIST AND ESTIMATED QUANTITATION LIMITS (EQL)

Volatile Organics	Quantitation Limits * **		
	Water ug/L	Low Soil ug/Kg	Med. Soil ug/Kg
Xylene (total)	5	5	500
Dibromomethane	10	10	1,000
1,2-Dibromoethane (EDB)	10	10	1,000
1,1,1,2-Tetrachloroethane	10	10	1,000
1,2,3-Trichloropropane	10	10	1,000
Dichlorodifluoromethane	10	10	1,000
Iodomethane	10	10	1,000
3-Chloro-1-Propene	10	10	1,000
2-Methyl-2-Propenenitrile	10	10	1,000
2-Chloro-1,3-Butadiene	10	10	1,000
Methyl Methacrylate	10	10	1,000
Ethyl Methacrylate	10	10	1,000
1,4-Dichloro-2-Butene	10	10	1,000
1,2-Dibromo-3-Chloropropane	10	10	1,000
Acrolein	100	100	10,000
Acrylonitrile	35	35	3,500
Trichlorofluoromethane	10	10	1,000
Pentachloroethane	5	5	500

***Quantitation limits listed for soil/sediment are based on wet weight. The quantitation limits calculated by the laboratory for soil/sediment, calculated on dry weight basis as required by the contract, will be higher.**

**** PQL's are based on a minimum of the lowest point in the curve or raised accordingly based on MDLs.**

Table 2.1 Additional Analytes analyzed by 8260B

Analyte	Quantitation Limits		
	Water (ug/L)	Low Soil (ug/Kg)	Med Soil (ug/Kg)
Iodomethane(methyl iodide)	2.0	10	1000
2-Nitropropane	4.0	20	2000
Cyclohexane	0.5	5	500
Methyl acetate	0.5	5	500
Methylcyclohexane	0.5	5	500
n-Butanol	20.0	100	10000
tert-Butyl Formate	5.0	15	1500
Ethyl Acrylate	2.0	5.0	200

TABLE 3.0
GC/MS PERFORMANCE STANDARD
BROMOFLUOROBENZENE (BFB)

m/z	Ion Abundance Criteria	% Relative Base Peak	Abundance Appropriate Peak
50	15-40% of mass 95	25.60	25.60
75	30-60% of mass 95	54.84	54.84
95	Base peak, 100% relative abundance	100.00	100.00
96	5-9% of mass 95	7.58	7.58
173	Less than 2 percent of mass 174	0.00	0.00
174	Greater than 50% of mass 95	90.01	90.01
175	5-9% of mass 174	6.66	7.40
176	95-101% of mass 1 74	88.81	98.66
177	5-9% of mass 175	6.52	7.32

TABLE 4.0
MATRIX SPIKE RECOVERY AND RELATIVE PERCENT DIFFERENCE LIMITS

Compound	% Recovery Water	RPD Water	% Recovery Soil	RPD Soil
1,1-Dichloroethane	61-145	14	59-172	22
Trichloroethene	71-120	14	62-137	24
Benzene	76-127	11	66-142	21
Toluene	76-125	13	59-139	21
Chlorobenzene	75-130	13	60-133	21

TABLE 5.0
SYSTEM MONITORING COMPOUND RECOVERY LIMITS*

Compound	% Recovery Water	% Recovery Soil
Toluene-d_8	85-125	54-93
Dibromofluoromethane	87-128	53-114
Bromofluorobenzene	83-120	50-103
1,2-Dichloroethane-d_4	78-118	50-121

*Surrogate windows will change as periodic updates of surrogate windows are performed.

TABLE 6.0
VOLATILE INTERNAL STANDARDS WITH CORRESPONDING TARGET COMPOUNDS
AND SYSTEM MONITORING COMPOUNDS ASSIGNED FOR QUANTITATION

Bromochloromethane	**1,4-Difluorobenzene**	**Chlorobenzene-d_5**
Chloromethane	1,1,1-Trichloroethane	2-Hexanone
Bromomethane	Carbon Tetrachloride	4-Methyl-2-Pentanone
Vinyl Chloride	Bromodichloromethane	Tetrachloroethene
Chloroethane	1,2-Dichloropropane	1,1,2,2-Tetrachloroethane
Methylene Chloride	trans-1,3-Dichloropropene	Toluene
Acetone	Trichloroethene	Chlorobenzene
Carbon Disulfide	Dibromochloromethane	Ethylbenzene
1,1-Dichloroethene	1,1,2-Trichloroethane	Styrene
1,1-Dichloroethane	Benzene	Xylene (total)
1,2-Dichloroethene (total)	cis-1,3-Dichloropropene	Bromofluorobenzene (smc)
Chloroform	Bromoform	Toluene-d_8 (smc)
1,2-Dichloroethane		
2-Butanone		
1,2-Dichloroethane-d_4 (smc)		

(smc) - system monitoring compound

TABLE 7.0
CHARACTERISTIC IONS FOR SYSTEM MONITORING COMPOUNDS AND INTERNAL STANDARDS FOR VOLATILE ORGANIC COMPOUNDS

System Monitoring Compounds	Primary Ion	Secondary Ion(s)
Dibromofluoromethane	111	113,192
4-Bromofluorobenzene	95	174,176
1,2-Dichloroethane-d_4	65	102
Toluene-d_8	98	70, 100

Internal Standards	Primary Ion	Secondary Ion(s)
Fluorobenzene	96	50,70
1,4-Dichlorobenzene d4	152	63, 88
Chlorobenzene-d_5	117	82, 119

TABLE 8.0
CHARACTERISTIC IONS FOR VOLATILE TARGET COMPOUNDS

Analyte	Primary Ion*	Secondary Ion(s)
Chloromethane	50	52
Bromomethane	94	96
Vinyl Chloride	62	64
Chloroethane	64	66
Methylene Chloride	84	49, 51, 86
Acetone	43	58
Carbon Disulfide	76	78
1,1-DIchloroethene	96	61, 98
1,1-Dichloroethane	63	65, 83, 85, 98, 100
1,2-Dichloroethene	96	61, 98
Chloroform	83	85
1,2-Dichloroethane	62	64, 100, 98
2-Butanone	43**	57
1,1,1-Trichloroethane	97	99, 117, 119
Carbon Tetrachloride	117	119, 121
Bromodichloromethane	83	85
1,1,2,2-Tetrachloroethane	83	85, 131, 133, 166
1,2-Dichloropropene	75	77
Trichloroethene	130	95, 97, 132
Dibromochloromethane	129	208, 206
1,1,2-Trichloroethane	97	83, 85, 99, 132, 134
Benzene	78	---
cis-1,3-Dichloropropene	75	77
Bromoform	173	171, 175, 250, 252, 254, 256
2-Hexanone	43	58, 57, 100
4-Methyl-2-pentanone	43	58, 100
Tetrachloroethene	164	129, 131, 166
Toluene	91	92
Chlorobenzene	112	114
Ethylbenzene	106	91
Styrene	104	78, 103
Total Xylenes	106	91

* The primary ion should be used unless interferences are present, in which case, a secondary ion may be used.
** m/z 43 is used for quantitation of 2-butanone, but m/z 72 must be present for positive identification.

Appendix C-2

Supporting Information for TCL SVOC Analysis

Semivolatile Compounds by GC/MS	8270C

Analyte Description	CAS Number
1,1'-Biphenyl	92-52-4
2,4,5-Trichlorophenol	95-95-4
2,4,6-Trichlorophenol	88-06-2
2,4-Dichlorophenol	120-83-2
2,4-Dimethylphenol	105-67-9
2,4-Dinitrotoluene	121-14-2
2,4-Dinitrophenol	51-28-5
2,6-Dinitrotoluene	606-20-2
2-Chloronaphthalene	91-58-7
2-Chlorophenol	95-57-8
2-Methylnaphthalene	91-57-6
2-Methylphenol	95-48-7
2-Nitroaniline	88-74-4
2-Nitrophenol	88-75-5
3,3'-Dichlorobenzidine	91-94-1
3-Nitroaniline	99-09-2
4,6-Dinitro-2-methylphenol	534-52-1
4-Bromophenyl phenyl ether	101-55-3
4-Chloro-3-methylphenol	59-50-7
4-Chloroaniline	106-47-8
4-Chlorophenyl phenyl ether	7005-72-3
4-Methylphenol	106-44-5
4-Nitroaniline	100-01-6
4-Nitrophenol	100-02-7
Acenaphthene	83-32-9
Acenaphthylene	208-96-8
Acetophenone	98-86-2
Anthracene	120-12-7
Atrazine	1912-24-9
Benzaldehyde	100-52-7
Benzo[a]anthracene	56-55-3
Benzo[a]pyrene	50-32-8
Benzo[b]fluoranthene	205-99-2
Benzo[g,h,i]perylene	191-24-2
Benzo[k]fluoranthene	207-08-9
Bis(2-chloroethoxy)methane	111-91-1
Bis(2-chloroethyl)ether	111-44-4
Bis(2-ethylhexyl) phthalate	117-81-7
Butyl benzyl phthalate	85-68-7
Caprolactam	105-60-2
Carbazole	86-74-8
Chrysene	218-01-9
Di-n-butyl phthalate	84-74-2
Di-n-octyl phthalate	117-84-0
Dibenz(a,h)anthracene	53-70-3
Dibenzofuran	132-64-9
Diethyl phthalate	84-66-2
Dimethyl phthalate	131-11-3
Fluoranthene	206-44-0
Fluorene	86-73-7
Hexachlorobenzene	118-74-1
Hexachlorobutadiene	87-68-3
Hexachlorocyclopentadiene	77-47-4
Hexachloroethane	67-72-1
Indeno[1,2,3-cd]pyrene	193-39-5
Isophorone	78-59-1
N-Nitrosodi-n-propylamine	621-64-7
N-Nitrosodiphenylamine	86-30-6
Naphthalene	91-20-3
Nitrobenzene	98-95-3
Pentachlorophenol	87-86-5
Phenanthrene	85-01-8
Phenol	108-95-2
Pyrene	129-00-0
2,2'-oxybis[1-chloropropane]	108-60-1

Title: SOP for GC/MS Semivolatiles
[Method SW846 8270C]

Approvals (Signature/Date):

Magdalena Stayniak XX/XX/09	David W. Hilfiker XX/XX /09
Technical Manager Date	Health & Safety Manager/Coordinator Date
Dawn May 10/14 /09	XX/XX /09
Quality Assurance Manager Date	Laboratory Director Date

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1.0 APPROVALS

The approval of the Laboratory Director, the Facility QA Manager, Group Leader and Health and Safety Officer signify that the document has been prepared in accordance with the format requirements that have been established in this document. In addition, if the document is a Method SOP the signature signifies that it meets the specified requirements.

2.0 SCOPE AND APPLICATION

2.1 The purpose of this SOP is to outline the techniques for determining the presence and concentration of various semivolatile organic target and non-target compounds. The standard 8270C target compounds are listed in Table 1.0. Table 1.1 lists the expanded Appendix IX target compounds applicable to this method. The method used in this procedure is solvent extraction and gas chromatograph/mass spectrometer analysis.

2.2 It is the policy of Testamerica and of the Semivolatiles Group to ensure that we administer contracts and orders for goods and services in a manner that is fully compliant with governmental laws and regulations, as well as the Testamerica Policy Statement on Business Ethics and Conduct.

2.3 The document control number for this SOP is CT-MSS-27, rev. 12.

3.0 TERMS AND DEFINITIONS

3.1 There are many terms and definitions used within the laboratory, which are listed in the latest version of the SOP for Terms and Definitions.

4.0 SUMMARY OF METHOD

4.1 This method employs the technique of solvent extraction, followed by gas chromatograph/mass spectrometer analysis. An aliquot of sample extract is injected onto a gas chromatograph. The fused silica capillary column is then temperature programmed to separate the semi-volatiles organics prior to detection by the mass spectrometer.

4.2 This method is based on SW-846 Method 8270C and Method 8000B. The concentration of the solution used for the initial demonstration of precision and accuracy varies from that listed in the method. The lab uses a concentration of 40 ug/l, and the concentration listed in Table 6 of the method is 100 ug/l. The lab has generated limits based on in house data. These limits are updated on an as needed basis.

4.3 In the instance where project plans may require lower detection limits, Selected Ion Monitoring (SIM) may analyze certain compounds. Compounds and analysis procedures are noted in Appendix A of this SOP.

5.0 INTERFERENCES

5.1 Method interferences may be caused by contaminants in solvents, reagents, and laboratory solvent vapors. This can lead to discrete artifacts and/or elevated baseline in the gas chromatograph. All these materials must be demonstrated to be free from interferences by the running of laboratory reagent blanks.

6.0 HEALTH AND 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.

6.1 SPECIFIC SAFETY CONCERNS OR REQUIREMENTS

The gas chromatograph contains 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.

There are areas of high voltage in the gas chromatograph. Depending on the type of work involved, either turn the power to the instrument off, or disconnect it from its source of power.

6.2 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 (2)	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.
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.
Acetone	Flammable	1000 ppm-TWA	Inhalation of vapors irritates the respiratory tract. May cause coughing, dizziness, dullness, and headache.
2 – Exposure limit refers to the OSHA regulatory exposure limit.			

7.0 SAMPLE PRESERVATION AND STORAGE

7.1 All sample extracts for semivolatile analysis must be protected from light and refrigerated at 4^0C from the time of extraction until analysis.

7.2 All sample extracts for semivolatile analysis shall be analyzed within 40 days of following extraction.

8.0 APPARATUS AND MATERIALS

8.1 GC/MS/DS System

8.1.1 Gas Chromatograph –Agilent Model 6890 GC, an analytical system which is temperature programmable with splitless injection and all required accessories including syringes, analytical columns, and gases.

8.1.2 GC Column – Phenomenex ZB-5ms, 30m x 0.25mm ID x 0.25um film thickness, or Restek Corporation RTX5, 30m x 0.25mm ID x 0.5um film thickness columns, or equivalent.

8.1.3 Mass Spectrometer - Agilent 5973, or 5975 capable of scanning from 35 to 500 AMU's every 1 second or less, utilizing 70 volts (nominal) electron energy in the EI ionization mode, and producing a mass spectrum which meets all the instrument performance criteria when 50 ng or less of DFTPP is injected through the GC inlet. Refer to Table 4.0 for the performance criteria. Any samples analyzed when DFTPP criteria have not been met will require reanalysis.

8.1.4 GC/MS interface - any GC to MS interface that gives acceptable calibration points, at 50 ng or less per injection, for each of the parameters of interest, and achieves all acceptable performance criteria, may be used. GC to MS interfaces constructed of all-glass or glass-lined materials are recommended. Glass can be deactivated by silanizing with dichlorodimethylsilane.

8.1.5 Data system - Agilent Chemstation / Enviroquant / Target Software, capable of continuous acquisition and storage, on machine readable media, of all mass spectra obtained throughout the duration of the chromatographic program. The computer has software that allows searching any data file for ions of a specified mass and plotting such ion abundances versus time or scan number (EICP). Software also allows integrating the abundance in any EICP between specified time limits. Also, software allows for the comparison of sample non-target spectrum against reference library spectra. The most recent release of the NIST/EPA/NIH mass spectral library shall be used as the reference library. The data systems will flag all manual edits with "M" qualifier.

8.1.6 DAT storage unit for long term, off line storage.

8.1.7 Syringes -various volumes.

8.1.8 Micropipets (Wiretrols) - various volumes.

9.0 REAGENTS AND STANDARD PREPARATION

9.1 Stock Standards - Certified standards purchased in ampules from commercial sources of target compound mixes, matrix spike compounds, surrogates, and internal standards. The laboratory utilizes the manufacturers expiration date for the stock standards. If the manufacturer provides no expiration date, the lab uses 12 months from receipt as the expiration date. The lab uses a 12-month from the

preparation date for the expiration of intermediate standards, unless the stocks used to prepare it expire earlier. In that case, the earlier date is used. However, if it appears that the solution has degraded or evaporated, it is replaced sooner. Storage of stock standards shall be stored at manufacturers recommendations. Table 10 contains a listing of typical standards used, with manufacturer and part #'s.

9.2 Working Standards

9.2.1 Calibration Standard Solutions

Calibration standards at six concentration levels are prepared from the stock solutions. There are three curves which may be used as needed and are listed in Tables 9.0, 9.1 and 9.2, listing analytes and curve concentration for each level. Table 9.0 is the normal 8270 compound list most routinely requested. Table 9.1 contains the Appendix 9 compound list including additional analytes that may be requested. Table 9.2 is a list of Zeneca analytes. Refer to Table 11 for the preparation of each standard level. Each standard contains all the target compounds and surrogates. Continuing calibration standards should be prepared weekly.

9.2.2 Internal Standard (IS) Spiking Solution

A 400 ng/ul IS spiking solution containing the internal standards 1,4-dichlorobenzene-d4, naphthalene-d8, acenaphthene-d10, phenanthrene-d10, chrysene-d12, and perylene-d12, is prepared from stock. Add 5 ul of this solution to a 100 ul aliquot of extract for a concentration of 20 ng/ul.

9.2.3 Surrogate (SU), Matrix Spike (MS), and Full Matrix Spike (FMS) Spiking Solution

The acid and base-neutral surrogate, matrix spike, and full matrix spike spiking solutions containing the appropriate compounds in methanol are prepared and utilized by the Extractions group.

9.2.4 DFTPP Tuning Mix

The DFTPP Tuning mix contains DFTPP at 25ug/ml . Alternatively, the continuing calibration standard at 40ug/ml may contain DFTPP, 4'4 DDT, pentachlorophenol, and benzidine to test tuning, breakdown and tailing.

9.2.5 Storage of all working standards for semivolatile analysis must be protected from light and refrigerated at $<10^0$C.

10.0 CALIBRATION

10.1 Calibration Standards

Six initial calibration standards containing all the semivolatile target compounds and SU's are analyzed by injecting 1ul of the calibration standards. The IS compounds are added to all calibration standards at a final concentration of 20 ng/uL.

10.2 The working calibration range of this method is defined by the initial calibration curve, 4 to 80 ng. See table 9.0 for compound specific concentrations. All extracts with target compounds exceeding 80 ng/uL, must be diluted to within the upper half of the calibration range.

10.3 Initial Calibration

The calibration curve is prepared by adding 100 ul of each of the six calibration standards and 5 ul of Internal Standard Solution to a 200 ul conical vial for a total of 105 ul per vial. An initial calibration must be analyzed on each GC/MS system upon installation, whenever corrective action is taken which may affect the initial calibration criteria, or if the continuing calibration criteria cannot be met.

The initial calibration can be performed only after the instrument performance is verified by meeting the DFTPP ion abundance criteria listed in Table 4.0. If less than 12 hours has expired since the tuning compound was injected, after meeting the initial calibration criteria, then samples may be analyzed. Quantitation is based on either the average RF in the curve or a linear regression line. If time does not remain in the 12 hour time period, a continuing calibration must be performed.

A 2nd source ICV is required following an initial calibration curve. Criteria is +/- 25% from the initial calibration with random exceedance criteria of 5% of the analytes up ±50% difference from the initial calibration standard

.

The response factors of the system performance check compounds (SPCC's) listed in Table 5.1 must have average relative response factors which are greater than or equal to 0.05, and the continuing calibration check compounds (CCC's) listed in Table 5.0 must have percent relative standard deviations (RSD's) less than or equal to 30.0 percent for the initial calibration to be acceptable. Linear regression or a quadratic equation (must use at least 6 levels for quadratic) may be used if the % RSD does not meet criteria for any compound except a CCC, listed in Table 5.0. The coefficient for the linear regression or quadratic equation must be 0.990 or greater.

A minimum of 5 calibration points are used to calculate the % RSD's. A six-point calibration point is typically run, however, the low point for poorly responding acid compounds may be removed. This removal of the low point is acceptable as long as the reporting limit can still be met by the lowest standard remaining in the curve. A five-point calibration curve is required for all compounds.

There are three options that can be used to determine the acceptability of an initial calibration. They are listed below;

1) If the %RSD of any compound is ≤15%, the average RF from the curve for that compound may be used for quantitation.

2) If the %RSD for one or more analytes is greater than 15%, the mean %RSD for all compounds will be calculated. If the mean %RSD is ≤15%, the average RF may be used for all compounds.

3) If the mean %RSD is greater than 15%, quantitation from the regression line must be used for those compounds whose %RSD is greater than 15%. Quadratic fit may also be used if there is a 6-point curve. The coefficient must be >= 0.990 for both linear regression and quadratic fit.

10.4 Continuing Calibration

The continuing calibration can be performed only after the instrument performance is verified by meeting the DFTPP ion abundance criteria listed in Table 4.0. The calibration is verified by injecting a standard containing 40ng/uL of each compound and 50ng or less of DFTPP. The standard must be analyzed every 12 hours.

The response factors of the system performance check compounds (SPCC's) listed in Table 5.1 must have response factors greater than 0.05, and the continuing calibration check compounds (CCC's) listed in Table 5.0 must be less than or equal to 20.0 percent difference (%D) for the continuing calibration to be acceptable. The calculations are listed in Section 13.0. If the continuing calibration does not meet the above criteria the standard must be re-injected. If in the judgment of the analyst routine maintenance will solve the problem it can be performed prior to re-injection of the standard. Examples of routine maintenance include cutting the column, cleaning the injection port, and changing the injection port liner. Repeated failure may require corrective actions and reanalysis of the initial calibration.

11.0 QUALITY CONTROL

11.1 Demonstration of Analyst Capability

11.1.1 Prepare four aliquots of the QC Check Standard at 40 ug/l in reagent water. Process the samples through the whole analytical procedure.

11.1.2 Calculate the average recovery (x) and the standard deviation (s) for each analyte from the four results. Compare the s and x with the criteria generated from the laboratory control charts for each compound for each matrix. The limits are derived from laboratory-generated data, and are updated as needed. If all analytes meet the acceptance criteria, analysis of samples can begin. If any analyte fails, the cause for the failure must be determined and the test must be repeated for that analyte.

11.1.3 The demonstration of analyst capability will be verified on an annual basis.

11.2 Method detection limits (MDL's) for this method will be verified on an annual basis as detailed in the latest version of the corporate SOP on MDL's. The MDL will be verified on each instrument on a yearly basis by the analysis of an MDL check sample. The MDL check sample is an extracted sample containing each target analyte at a concentration close to the MDL. Each analyte must be detected in order for the instrument to be considered capable of reporting estimated result to the calculated MDL. If an analyte cannot be detected at the given concentration, instrument maintenance should be performed and the MDL check sample re-analyzed. If the compound is still not detected, a new MDL should be prepared and analyzed at a higher concentration. The new MDL should be used in reporting results.

11.3 Method Performance Tests

11.3.1 Prior to initiating any data collection activities it is necessary to establish that a given GC/MS system meets the instrument performance criteria. This is accomplished through the analysis of 50ng or less of decafluorotriphenylphosphine (DFTPP).

11.3.1.1 DFTPP must be analyzed at the start of every 12-hour sequence and can be a component in the 40ng/ul calibration standard.

11.3.1.2 The key ions produced during the analysis of DFTPP and their respective ion abundance criteria are given in Table 4.0. This criteria must be met before any calibration standards, blanks, or samples may be analyzed.

11.3.1.3 If the criteria is not met, the DFTPP must be reanalyzed. Repeated failure shall require the

instrument to be manually tuned. After manual tuning, the DFTPP must be re-injected and the abundance criteria must be met before proceeding.

The 12-hour time period for a GC/MS system instrument performance check and standards calibration (initial or continuing calibration criteria) begins at the moment of injection of the DFTPP analysis that is submitted as documentation of a compliant instrument performance check. The time period ends after 12 hours have elapsed according to the system clock

11.3.2 After the instrument performance criteria is met, an initial calibration is performed, or the initial calibration curve is verified through the analysis of a continuing calibration at 40ng/uL. The continuing calibration criteria must be met before any method blank or sample analyses may proceed.

11.3.3 A method blank spiked with surrogates is extracted with every batch of samples and must be analyzed with the sample extracts after calibration criteria has been met. An acceptable method blank must meet the following criteria:

* Less than or equal to 5X the PQL for the phthalate esters.

* Less than or equal to the PQL for each of the other target compounds

If the method blank exceeds the above criteria, the analytical system is considered to be out of control. The source of the contamination must be investigated and appropriate corrective measures must be taken and documented before further sample analysis proceeds. All samples analyzed with a method blank that is out of control must be re-extracted and reanalyzed. The problems and solutions must be addressed in the SDG narrative.

When running samples on an instrument and there is not Method blank for that analytical sequence(i.e. dilution reruns), an instrument blank must be run consisting of 100 ul of appropriate solvent with 5 ul of ISTD after initial or continuing calibration criteria and before sample extracts.

11.3.4 Structural Isomers

Structural Isomers that produce very similar mass spectra should be identified as individual isomers only if they have sufficiently GC retention times and sufficient GC resolution.

Sufficient GC resolution is achieved if the height of the valley between two isomer peaks is less than 25% of the sum of the two peak heights. Otherwise, structural isomers are identified as isomeric pairs.

11.4 Breakdown and Tailing requirements

Degradation of DDT to DDE and DDD should not exceed 20%. See 8081A SOP for the percent breakdown calculation.

Benzidine and Pentachlorophenol should be present at their normal responses, and no peak tailing should be visible.

11.5 Matrix Spike, Matrix Spike Duplicates and Matrix Spike Blanks

11.5.1 An MS and MSD must be extracted and analyzed for each group of samples of a similar matrix within each case, 20 samples, group of samples of a similar concentration level (soils only), or each 7 calendar day period; whichever is more frequent. An MSB is extracted and analyzed for NYSDEC SW846 work requiring a blank spike in the deliverables.

11.5.2 The lab generated limits for matrix spike compound recovery and relative percent difference (RPD) are given in Table 2.0. These limits are only advisory; therefore, no further action is required if the criteria limits are not achieved. However, frequent failures shall be investigated for possible laboratory generated error. The NYSDEC MSB has mandatory acceptance criteria. The MSB is evaluated against the matrix specific % Recoveries listed in Table 2.0. If the MSB does not meet criteria, a CAR is written, and the client is informed. The lab will proceed in accordance with the client's instructions.

11.6 Laboratory Control samples (LCS)

11.6.1 A LCS must be extracted with each extraction preparation batch for each group of samples of a similar matrix and concentration level (soils will require separate LCS's if they are extracted by low level or medium level extraction), and must be analyzed with the sample extracts.

11.6.2 The limits for the spike compound recoveries are based on laboratory generated data, and are updated on at least an annual basis. The number of allowable exceedances for LCS without re-extraction being required is as follows:

a) >90 analytes in LCS, 5 analytes allowed in ME of the LCS control limit:
b) 71-90 analytes in LCS, 4 analytes allowed in ME of the LCS control limit;
c) 51.70 analytes in LCS, 3 analytes allowed in ME of the LCS control limit;
d) 31-50 analytes in LCS, 2 analytes allowed in ME of the LCS control limit;
e) 11-30 analytes in LCS, 1 analyte allowed in ME of the LCS control limit;
f) <11 analytes in LCS, no analytes allowed in ME of the LCS control limit;

Marginal exceedances(ME) must be random and is +/- 4 standard deviations from the mean. If the same analyte exceeds the LCS control limits frequently, the source of the problem has to be located and corrected. Re-extraction is required when the number of failures exceeds the allowable amount.

11.7 Surrogates

11.7.1 The surrogates Phenol-d5, 2,4,6-Tribromophenol, 2-Fluorophenol, Nitrobenzene-d5, Terphenyl-d14, and 2-Fluorobiphenyl are added to each sample, blank, standard, and MS/MSD, prior to extraction. The acid surrogates are added at 75 ug and the base-neutrals at 50 ug for waters and soils.

11.7.2 Surrogate recoveries must be within the lab generated QC limits given in Table 3.0. The QC limits are periodically re-evaluated and updated as necessary. If the recovery for any two acid surrogates (phenol-d5, 2-fluorophenol, and 2,4,6-tribromophenol) or any two BN surrogates (nitrobenzene-d5, 2-fluorobiphenyl, and terphenyl-d14) are not within the QC limits defined in Table 3.0, or if any one of these surrogate recoveries are below 10%, the following actions are required:

* Check all calculations for accuracy, spiking solutions, and internal standards

* Re-analyze the sample if none of the above steps reveal a problem

* Do not reanalyze dilutions if surrogate recoveries are outside limits

* If recoveries are high and the sample does not contain targets for that fraction, i.e. acid or base, report the sample results and narrate in the report narrative.

* Never reanalyze the MS or MSD, even if the surrogate recoveries are outside the limits

* If the sample associated with the MS/MSD does not meet specifications, it should be reanalyzed only if the MS/MSD surrogate recoveries are within the limits

If the reanalysis of the sample solves the problem, then only submit the second analysis. If the reanalysis does not solve the problem, then re-extract and reanalyze the sample. If the re-extraction and reanalysis of the sample solves the problem, contact the client by a CAR and find out what they want submitted. If the re-extraction and reanalysis does not solve the problem, then both sets of results may be reported. Re-extraction and reanalysis shall be decided on a project specific basis and may not be requested by the client.

11.7.3 If the recovery of the surrogates in a method blank are not acceptable, as defined in section 11.6.2 above, then the method blank and all associated samples must be re-extracted and reanalyzed.

11.8 Internal Standards (IS or ISTD)

11.8.1 Internal standards are added to each sample, blank, standard, and MS/MSD, at 20 ng prior to injection.

11.8.2 The retention times (RT) and extracted ion current profile (EICP) of each IS must be evaluated for all standards immediately after the data acquisition. The IS EICP areas must be monitored and evaluated for each sample, blank, MS, MSD. If the IS EICP changes by more than a factor of 2 (-50% to +100%) from the latest (12 hour) calibration standard, the mass spectrometric system must be inspected for malfunctions, and corrections made as required. If the RT for any IS changes by more than 30 seconds from the latest (12 hour) calibration standard, the chromatographic system must be inspected for malfunctions, and corrections made as required. When corrections are made, reanalysis of the samples analyzed while the system was malfunctioning is necessary. If no system malfunctioning occurred, then document the matrix interference problem via a corrective action report. If the sample associated with the MS/MSD does not meet specifications, it should be reanalyzed only if the MS/MSD IS criteria is within limits.

If the reanalysis of the sample solves the problem, then only submit the second analysis.

11.8.3 The ISTD areas in the continuining calibration standard must be within (-50% to +100%) from the last Initial calibration curve. If the continuing calibration standard fails to meet this criteria, a new initial calibration must be analyzed.

11.9 Quality Control Check Points

11.9.1 Analysis Quality Control Approval Report

Specific quality control checkpoints have been established for the analysis of samples which are

monitored through a Quality Control Approval Report (QCAR). The specific check points must be initialed and dated by the analyst to ensure the consistency and accuracy of the data produced.

11.10 Analytical Documentation Procedures

11.10.1 Instrument Batches

An instrument batch is created for each analytical sequence to organize all the associated data. Batch designations are of the format:

Xnnnnnn where X = instrument identifier
and nnnnnn = number of batch

(i.e.R051234) Instrument batch numbers are created by combining the last two digits of the year with the first data file number in the batch, creating a unique batch identifier.

11.10.2 Filing System

All active batches are filed chronologically according to instrument. The batches are transferred to file boxes for long-term storage once all the associated data within a batch has been completed.

11.10.3 Data Archiving

The data files and method files on the server are archived on a daily basis by the systems group.

11.10.4 Instrument Run Logs

It is TestAmerica's policy that all measurement data be recorded in logbooks or on preprinted log sheets in permanent ink. Transcriptions shall be avoided whenever possible. The record shall reflect the measurement performed and all appropriate details for conclusions related to the measurement. The record shall be signed and dated by the individual performing the measurement on the day the measurement is performed. Corrections must be made by drawing a single line through the error, and initialing and dating the correction. A secondary authorization of the logbook is required and shall be performed by the department's manager or designee.

Each instrument has its own set of run logs which are sequentially number and paginated and bound once 90 pages is reached. Run logs are filed with the QA department once they have been filled, for future reference. Each analytical sequence shall be started on a new page of the log and continued on the next page, if necessary. The header information designating the standard codes used shall be completed for each sequence. All standards used are recorded in this field for future traceability. The data file, job and sample number, dilution factor, analyst's signature, and date are recorded.

11.10.5 Initial data review sheet (BSUM)

The initial data review sheet (IDRS) is a computerized review sheet, which is used to check the key quality control criteria for compliance. The IDRS is used to check that all samples have been analyzed with the required calibration time frame, if the IS's meet RT and EICP area criteria, and if the ID file being utilized was correctly updated. The IDRS is also used as the initial data review tool. Each sample is listed on the sheet and it is either accepted or rejected in the right hand column, by the

analyst performing the data review. If reruns are required for dilutions, then the analyst shall indicate the proper dilution required for reanalysis.

11.10.6 Corrective Action Reports

A corrective action report (CAR) is initiated when a problem is encountered during analysis, data reduction or deliverables preparation, data validation, or when any deviations from this SOP occur. The CAR is initiated by the analyst or dept. manager first identifying the problem through the NCM module in the LIMS system. It is then electronically forwarded to all appropriate departments, QA officer, and Lab Manager. Reference SOP for correction action reports.

11.10.7 Chain of Custody Record

When samples are removed from storage for preparation or analysis they must be signed out utilizing the chain of custody record (COC). The samples shall then be signed back in on the COC upon their return to storage or designated "used" if the sample volume is consumed during the preparation or analysis.

11.10.8 Sample Tracking Record

Samples are tracked on the Semivolatile Tracking Sheet (see Figure 3.0). A tracking sheet is generated by printing the LIMS prep batch sheet. The tracking sheet is updated after initial data review, designating that the sample is analyzed and complete with its associated data file, or that the sample requires reanalysis. Samples requiring reanalysis are also documented as to the reanalysis dilution required, if any. If a re-extraction is required, an NCM is issued.

12.0 SAMPLE PREPARATION AND INSTRUMENTAL PROCEDURES

12.1 Instrumental Conditions

12.1.1 Gas Chromatograph (Suggested conditions)

Carrier Gas:	Helium
Flow Rate:	30 cm/sec
Initial Temp.:	40^0C
Initial Hold:	4 min
Ramp Rate 1:	10°/min
Second Temp.:	260C
Second Hold:	12 min
Ramp Rate 2:	8^0/min
Final Temp.:	310^0C
Final Hold:	10 min
Injector Temp.:	250^0C
Transfer Temp:	310^0C
Injector:	Grob-type, splitless
Sample Volume:	1 or 2 ul
EPC/EPP	99 mls/min to 40 psi for 0.3 min then 99 mls/min to 7.1 psi (this can be used on instruments equipped with EPC)

12.1.3 Mass Spectrometer

Electron Energy:	70 eV
Mass Range:	35 - 500 amu
Scan Time:	less than 1 sec/scan

The mass spectrometer must be tuned to meet the instrument performance check criteria for 50 ng or less of DFTPP listed in Table 4.0.

12.2 Sample Analysis Procedures

12.2.1 Sample Extract Analysis

Sample extracts are removed from storage (refrigerator #36) and are signed out on the chain of custody form. All sample extracts are signed back in after they returned to storage.

Make sure all instrumental operating conditions are correctly set and DFTPP and calibration criteria have been met.

In a 200 ul shell vial, load a 100 ul aliquot of sample extract and spike with 5 ul of IS spiking solution.
The prepared extracts are then loaded into the HP auto sampler carousel tray and the sequence is set up in the software to match the injection sequence on the auto sampler tray. If a sample is analyzed which contains target compounds at concentrations greater than the initial calibration upper limit, then the sample must be reanalyzed at an appropriate dilution.

12.3 Qualitative Analysis

12.3.1 Target Compounds

The relative retention time of a target compound must be within +/- 0.06 RRT units of the RRT of the calibration standard for a positive identification. For reference the standard must be analyzed within the same 12 hour time period as the sample. If the sample is analyzed within the same 12 hour time period as the initial calibration, then use the 40 ng standard as the reference. If co-elution of interfering components prohibits accurate assignment of the sample component RRT from the total ion chromatogram, the RRT shall be assigned by using the extracted ion current profiles for ions unique to the component of interest.

In addition, a comparison must be made between the mass spectrum obtained in the sample analysis and the reference mass spectrum for that compound, which was obtained on that specific GC/MS system. The requirements for qualitative verification by comparison of mass spectra are as follows:

All ions present in the reference spectrum at intensity greater than 10% must be present in the sample spectrum.

The relative intensities of the ions above 10% must agree with 20% between the reference and sample spectra.

Ions greater than 10% in the sample spectrum but not present in the reference spectrum must be considered and accounted for by the analyst.

If a compound cannot be verified by the above criteria, but in the technical judgment of the analyst, the identification is correct, then the compound shall be reported.

The mass spectral interpretation checklist, refer to Figure 1.0, is used by the mass spectral interpretation specialists to verify the identification of the compounds with recurrent mass spectral interpretation problems. Refer to the checklist for the specific compounds reviewed.

12.3.2 Tentatively Identified Compounds

A library search may be performed upon request for non-target compounds in the sample for purposes of tentative identification. For this purpose, the most recent release of the NIST mass spectral library shall be used. Pesticide and PCB confirmation is also done by tentatively identified compound search if needed to prove the presence of these compounds from a GC analysis.

Up to 20 non-target organic compounds of highest apparent concentration shall be tentatively identified via a forward library search. Only compounds with responses greater than 10% of the closest IS exhibiting no interference are to be searched. Peaks suspected of being aldol-condensation reaction products shall be searched and reported as such, but not counted as part of the 20 most intense non-target compounds. Solvent peaks which may be detected due to an early scan start time shall not be counted as a library search compound.

A tentative identification will be made after a comparison between the mass spectrum obtained in the sample analysis and the library search mass spectra found for that compound. The requirements for tentative verification by comparison of mass spectra are as follows:

Molecular ions present in the reference spectrum should be present in the sample spectrum.

Ions present in the reference spectrum at intensity greater than 10% should be present in the sample spectrum.

The relative intensities of the ions above 10% should agree with 20% between the reference and sample spectra.

Ions present in the sample spectrum but not in the reference spectrum should be reviewed for possible background contamination or co-eluting compounds.

Ions present in the reference spectrum but not in the sample spectrum should be reviewed for possible background subtraction by the data system.

If in the technical judgment of the mass spectral interpretation specialist, no valid tentative identification can be made, the compound shall be reported as unknown. Additional classification shall be made if possible (i.e. unknown hydrocarbon).

12.4 Quantitative Analysis

12.4.1 Target Compounds

Target compounds are quantitated by the internal standard technique. The associated internal standard used is listed in Table 6.0. The EICP area of the quantitation ions of compounds listed in Tables 7.0 and 8.0 are used.

The average relative response factor (RRF) from the curve or the linear regression line from the curve 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, document the reasons in the SDG narrative. The area of a secondary ion cannot be substituted for the primary ion unless a RRF is calculated using the secondary ion.

When compound concentrations are below the PQL, but the compound meets identification criteria, report the concentration with a "J" qualifier if it is above the MDL.

Water Samples

$$\text{Concentration ug/L} = \frac{(Ax)(Is)(Vt)(Df)}{(Ais)(RRF)(Vo)(Vi)}$$

where,

Ax =area of the compound quantitation ion

Ais = area of IS quantitation ion

Is = IS amount in nanograms

RRF = average RRF from curve

Vo =volume of water extracted in ml's

Vi = volume of extract injected in ul's

Vt = volume of the concentrated extract in ul's

Df = Dilution factor. The dilution factor for the analysis of water samples for semi-volatiles organics by this method is defined as follows:

$$\frac{\text{uL most conc. extract used to make dilution + uL clean solvent}}{\text{uL most conc. extract used to make dilution}}$$

If no dilution is performed, Df = 1.0.

Soil Samples

$$\text{Concentration ug/Kg (dry weight basis)} = \frac{(Ax)(Is)(Vt)(Df)}{(Ais)(RRF)(Vi)(Ws)(D)}$$

where,

Ax, Is, Ais, Vt, Vi, Df, and RRF are as given for water.

$$D = \frac{100 - \%\ \text{moisture}}{100}$$

Ws = weight of sample extracted in grams

12.4.2 Tentatively Identified Compounds

An estimated concentration for non-target compounds tentatively identified in the sample shall be determined by the internal standard method. For quantitation, the nearest IS free of interferences shall be used.

The equation for calculating concentrations is the same as in 12.4.1. Total area counts from the total ion chromatograms are used for both the IS and compound. A RRF of 1.0 is assumed and the resulting concentration shall be qualified as "J" (estimated) and "N"(presumptive evidence of presence), indicating the quantitative and qualitative uncertainties associated with this non-target compound.

12.4.3 If the on-column concentration of any target compound in any sample extract exceeds the initial calibration range, that sample extract must be diluted, the IS concentration readjusted, and the sample extract reanalyzed. Guidance in performing dilutions, and exceptions to this requirement are as follows:

12.4.3.1 Use the results of the original analysis to determine the approximate dilution factor required to get the largest analyte peak within the initial calibration range.

12.4.3.2 The dilution factor chosen shall keep the response of the largest analyte peak for a target compound in the upper half of the initial calibration range of the instrument.

12.4.3.3 Data for more than two analyses shall not be submitted.

12.5 Instrument Maintenance

12.5.1 Preventative Maintenance

Preventative maintenance is performed at scheduled intervals on all equipment according to the manufacturers recommendations. All instrument preventative maintenance is performed according the manufacturers recommended procedures, by trained personnel. All preventative maintenance shall be thoroughly documented in the injection log with a description of the maintenance performed, the date performed, and the personnel performing the maintenance.

12.5.2 Corrective Maintenance Determinants and Procedures

Corrective maintenance is deemed necessary when the analytical system, after reanalysis, cannot meet tune, calibration, or other protocol specific QC criteria. Corrective maintenance may include, but is not limited to, decontamination of the system, source cleaning, replacing the electron multiplier, column replacement or filament replacement. All corrective maintenance is performed according the manufacturers recommended procedures, by trained personnel. All corrective maintenance shall be thoroughly documented in the maintenance log, with a description of the maintenance performed, the date performed, and the personnel performing the maintenance.

Any corrective maintenance should document a RTC (Return to Control) in the maintenance log indicating the first valid initial calibration curve following maintenance.

12.5.3 Maintenance Authorization

All preventative and corrective maintenance is authorized by the department's manager, or designee. When maintenance is deemed necessary, a service call is placed for all equipment covered under a service contract, by the department's manager, or designee.

12.6 Data System

12.6.1 Data Acquisition and System Operation

Data is acquired from sample analyses using the Agilent, ChemStation software. Analytical batches are set up with all the associated sample ID, dilution, and data file information. Automated post-acquisition quantitation performed using Target software.

12.6.2 Instrument Errors

System errors are logged to a system error file at the time of occurrence. The system manager shall be responsible for checking and providing corrective actions for all major system errors. Minor system errors, such as insufficient disk space, are handled by trained analysts, as necessary.

12.6.3 Manual Integrations and Editing Flags

Manual integrations are required when the automated software doesn't correctly integrate extracted ion current profiles (EICP). A user shall be logged into the Target system as their own name. This name will signify who performed the manual integration. To perform a manual integration, the target compound of interest is selected and the EICPs are graphically presented. The peak can then be correctly integrated. A reason code shall be selected for the type of integration performed by selecting Review codes from the menu options.

UN = Unidentified peak based on spectra or concentration
ID = Identified a peak based on spectra
INT = Integrated a peak due to incorrect integration

A new quantitation report is produced. The manually integrated data file is the saved by exiting and saving from file edit. Manual integrations are flagged by the data system with the "M" qualifier beside any manually integrated area. The manually integrated data file is the saved by exiting from Target review. Manual integrations are flagged by the data system with the "M" qualifier beside any manually integrated area on the hardcopy quant report. The analyst name will appear on the electronic manual integration report which is uploading to the LIMS system when data files are uploaded. A hardcopy print out of the EICP of the quant ion displaying the manual integration shall be produced for the before and after integrations and is included in the raw data to the clients when the report is generated during final packaging.

13.0 CALCULATIONS

13.1 Relative Response Factor (RRF)

$$RRF = \frac{(Ax)\,(Cis)}{(Ais)(Cx)}$$

where,

Ax =area of the compound quantitation ion

Ais = area of IS quantitation ion

Cis = IS concentration

Cx =compound concentration

An average RRF is calculated for each compound and surrogate from the initial calibration.

13.2 Percent Relative Standard Deviation (%RSD)

$$\%RSD = \frac{\text{Standard Deviation}}{\text{mean}} \times 100$$

13.3 Percent Difference (%D)

$$\%D = \frac{(\text{Average RRFi}) - (\text{RRFc})}{(\text{Average RRFi})} \times 100$$

where,

Average RRFi = Average RRF from the initial calibration

RRFc = RRF from the continuing calibration standard

13.4 Percent Moisture

$$\%\ \text{moisture} = \frac{\text{g of Wet Sample - g of Dry Sample}}{\text{g of Wet Sample}} \times 100$$

13.5 Target Compound Concentrations in the extract

13.5.1 The concentration of each identified analyte and surrogate in the extract is calculated from the average RF of the initial calibration or from the linear or quadratic cuve fitted to the initial calibration points.

13.5.2 Average Response factor – If the average of all the RSD's of the response factors in the initial calibration is <= 15%, the average response factor from the initial calibration may be used for quantitation.

$$Cex = \frac{RxCis}{RisAvgRF}$$

13.5.3 Linear Fit

$$Cex = A + B\frac{(RxCis)}{Ris}$$

Cex = Concentration in extract, ug/ml
Rx = Response for analyte(area of quantitation ion)
Ris = Response for internal standard(area of quantitation ion)
Cis = Concentration of internal standard
A = Intercept
B = Slope

The corresponding Target software calculation is as follows:

$$Cex = Cis\left(b + \frac{1}{m1} \times \frac{Rx}{Ris}\right)$$

b = Concentration Ratio Intercept
m1 = Inverse of slope

13.5.4 Quadratic fit

$$Cex = A + B\frac{(RxCis)}{Ris} + C\frac{(RxCis)^2}{Ris}$$

C = Curvature

The corresponding Target software calculation is as follows:

$$Cex = Cis\left(b + m1 \times \frac{Rx}{Ris} + m2 \times \left(\frac{Rx}{Ris}\right)^2\right)$$

m1 = First order coefficient
m2 = Curvature(Second order coefficient)

13.6 The concentration in the sample is then calculated.

13.6.1 Aqueous calculation

$$Concentration(ug/L) = \frac{(Cex)(DF)(Vt)}{(Vo)}$$

Vt = Volume of final extract in ul's
Vo = volume of water extracted in ml's
DF = Dilution factor

13.6.2 Sediment/soil, Sludge(on a dry weight basis) and Oil Waste(normally on a wet weight basis:

$$Concentration(ug/Kg) = \frac{(Cex)(DF)(Vt)}{(Ws)(X)}$$

Ws = Weight of Sample extracted or diluted in grams
X = (100-%moisture in sample)/100, for a dry weight basis or 1 for a wet weight basis (moisture factor applied by LIMS)

13.7 Surrogate Percent Recovery

$$\% \text{ Recovery} = \frac{\text{Concentration Found}}{\text{Concentration Spiked}} \times 100$$

13.8 Matrix Spike Recovery / Full Matrix Spike Recovery

$$\% \text{ Recovery} = \frac{SSR - SR}{SA} \times 100$$

where,

SSR = spiked sample result

SR =sample result

SA = spike added

13.9 Relative Percent Difference

$$RPDc = \frac{\text{Absolute } (Cms - Cmsd)}{(1/2)(Cms+Cmsd)} \times 100\%$$

where,

Cms = Concentration of matrix spike

Cmsd= Concentration of matrix spike duplicate

The absolute value of the concentration difference is used in the above equation.

13.9.1 Adjusted Estimated Quantitation Limit for Samples

$$\text{Adjusted EQL} = \frac{(EQL) \times Df}{D}$$

where,

$$D = \frac{100 - \% \text{ Moisture}}{100}$$

Df = Dilution Factor

14.0 ACCEPTANCE OF DATA

14.1 Method Blank

The method blank must contain less than or equal to the PQL of the target compounds, except the phthalate esters, which must be less than or equal to five times the PQL.

14.2 Surrogates (SU)

Refer to section 11.6 for surrogate acceptance criteria and corrective actions.

14.3 Instrument Performance Check

The criteria for DFTPP is listed in Table 4.0 and in section 11.3.1.

14.4 Internal Standards

The IS acceptance criteria is described in section 11.7.

14.5 Matrix Spike/Matrix Spike Duplicate

The MS/MSD acceptance criteria is described in section 11.4.

15.0 REPORTING OF RESULTS

15.1 General Information

The following samples suffixes are used:

LCS = Laboratory Control Sample
MS = Matrix Spike
MSD = Matrix Spike Duplicate
RE = Reanalysis or Re-extract
DL = Secondary Dilution
MSB = Matrix Spike Blank (used as a prefix)
FMS = Full Matrix Spike

15.2 Analysis Data Sheet

All results are reported uncorrected for blank contaminants and to one significant figure if the concentration is less than 10, and two significant figures if the concentration is greater than 10. Values below the MDL are not reported.

Data Reporting Qualifiers are as follows:

U = Analyzed for, but not detected.
J = Estimated value; used for all TIC concentrations and all targets with are less than the EQL adjusted for extraction volumes/% M.
B = Compound detected in the blank as well as in sample.
.
E = Concentration exceeds the working range of initial calibration.
D = All compounds detected in analysis at a secondary dilution.
N = Presumptive evidence of a compound; qualifiers at TIC results which have been tentatively identified with a CAS #.

Other qualifiers are defined in the report package.

15.3 Case Narrative

A case narrative is produced for every SDG. Any problems encountered during analysis, data

reduction, or any deviation from standard operating procedures will be noted here.

15.4 Forms

Necessary forms will be determined by the level of deliverables.

16.0 SUPPLEMENTAL DOCUMENTS

16.1 The analyst is referred to other departments SOP's for additional information concerning sample storage, sample removal, sample tracking and other information not included in this SOP.

17.0 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). Employees must abide by the policies in Section 13 of the Corporate Safety Manual for "Waste Management and Pollution Prevention."

17.1 Pollution prevention is the reduction and/or elimination of wastes or the toxicity of wastes at the point of generation. The laboratory strives to keep all wastes at a minimum through a variety of techniques. These include the following.

17.1.1 Inventory control: Rotate stocks (first in, first out) and only purchase reasonable amounts of chemicals. Do not purchase excessive quantities, which will be discarded as wastes at some future point.

17.1.2 Material Handling and Storage: Keep containers properly labeled. Keep solvents covered. Store and handle all chemicals, reagents, standards, etc. to prevent contamination and degradation.

17.1.3 Personnel: All staff must be trained in the proper handling and disposal of waste.

17.1.4 Waste Reduction: Reduce the volume of waste generated wherever possible.

17.1.5 Chemical/material substitution: Use less toxic chemicals whenever possible. Avoid the use of chromic acid (Chromerge) solutions.

18.0 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 Tracking anc Collection of Hazardous Waste SOP. The following waste streams are produced when this method is carried out.

18.1 All waste shall be managed in accordance with all state and federal requirements. See the TESTAMERICA-CT RCRA Contingency Plan.

18.2 All personnel who handle or generate waste must be trained within six months of employment in proper waste handling and requirements.

18.3 Solvent waste generated containing Methylene chloride, shall be disposed of in the container labeled

for Methylene chloride waste, then transferred at the end of the working shift to the waste drum located in extractions labeled Methylene chloride waste.

19.0 SUPPLEMENTAL DOCUMENTS

19.1 There is a variety of SOP's available to the analyst that document how to perform ancillary tasks that are not detailed in this SOP.

19.2 SOP for Control Limits

20.0 REFERENCES

20.1 USEPA SW-846, Method 8270C

21.0 SUBSTANTIVE REVISIONS

21.1 Original Issue - 01/06/98.

21.2 Changed AEN to STL, and minor editorial changes - 2/10/99.

21.3 Added appendix for Army Corps of Engineers specific requirements - 3/16/99.

21.4 Added new Section 3 - Terms and Definitions, Section 16 - Pollution Prevention, Section 17 - Waste Management, and Section 18 - Supplemental documents. Renumbered sections as appropriate, and minor editorial changes - 10/10/99.

21.4 Minor editorial changes – 05/15/01.

21.6 Updated to include minor procedural changes due to the implementation of Target and labnet software. Expanded the standards expiration dates section, and made minor procedural and editorial changes, including lowering the concentration used for the acid surrogates. Incorporated lab generated surrogate and matrix spike control limits into SOP, and revised Figures to current versions – 10/15/02.

21.7 Updated Section 6.0 for health and safety. Updated Section 10.0 to include 6 point calibration curves, linear regression and quadratic coefficient criteria. Updated 11.5.2 LCS criteria for PAH list. Updated references to tables and figures.

21.8 Section 9.2.4 added. Removed misleading statement from section 10.3 that 'no continuing calibration standard was required'. Added 2nd source ICV to section 10.3. Added linear regression and quadratic coefficient criteria. Referenced Appendix A for ACOE criteria. Section 11.1 and 11.1.3 reworded to analyst capability. Section 11.2 –clarified procedure of the quarterly check of MDL check samples. 11.3.3 added reference to Appendix A for ACOE criteria. Section 11.5.1 soils only statement clarified.. Section 11.5.2 added reference to Appendix A for ACOE criteria. Added Section 11.6.2.1 for reference to Appendix A for ACOE criteria. Section 12.3.2 added a TIC requirement. Section 12.4.1 removed manual integration signature requirement. Section 13.5 and 13.6 added calculations for a target compound. Section 14.1 added reference to Appendix A for ACOE criteria. Section 13.8 – changed RPD to calculate from concentration rather than recovery. Table 1 – clarified dry weight notation. Section 9.2.5 added. Updates to Appendix A – Changed initial calibration criteria. Added linear regression and quadratic fix requirements. Changed

continuing calibration criteria. Section 4.1 changed MB criteria. Added Section 5 for LCS criteria. Added Section 6 for Surrogate criteria. Changed ACOE requirements to APPENDIX A. Changed SIM analysis to APPENDIX B.

21.9 Revised section 9.2 to include weekly preparation of the continuing calibration standard. 6-24-05.

21.10 Added section 4.5 to include reference to sim/scan analysis. Added 5975 to Section 8.1.3. Std concentrations changed, referenced table 9.0 in section 9.2.1 and 10.2. Removed reference to 2ul injection from sect 10.1. Clarified use of linear regression with CCC's in Section 10.3. Updated continuing calibration concentration in section 10.4. Changed CAR to electronic submittal in section 11.9.6. 12/15/05.

21.11 Added Phenomenex column information to section 8.1.2. Added the need to run an instrument blank before sample extracts if no MB is run to section 11.3.3. Added need to log minor maintenance performed in the header section of run logs to section 11.9.4. 05/10/07

21.12 Section 11.5.2 – LCS changed from 12 out to 20% of target list can be out of criteria 5/10/07.

21.13 Added Organophosporous Pesticide, Zeneca Pesticide, and Golder compounds to Appendix A for target compound list table

21.14 Added new TestAmerica SOP header and control number, changed company name; changes Hewlett Packard to Agilent in several sections, section 9.1-added storage of stock standards, section 11.6.2 added "any 2" to BN surrogate criteria and added statement regarding surrogate that are out high, updated section 11.8.4 on electronic logs,revised section 11.9.6 to LIMS NCM process, updated section 11.9.8 for LIMS tracking, added statement on manual integration reasons in section 12.6.3; 01/21/08.

21.15 Added Table 9.0(Curve conc's each analyte). Table 10 Part #'s added. Table 11 added – prep. Updated 12.6.3 Man. Int. Removed Older instrument ref., updated MDL procedure, Added ISTD comparison of ccal to Ical. Added Return to Control Maintenance doc., Added DDT , Tailing, and Resolution requirements. Removed simult sim/scan. Removed ACOE references. Updated Safety, Pollution Control, Waste Mgmt sections. Updated QL levels in Table 1 & 1.1. 11/26/08

21.16 Sect 10.3 clarified ICV criteria. Sect 11.6.2 changed LCS criteria. 10/14/09.

TABLE 1.0
TARGET COMPOUND LIST (TCL) AND PRACTICAL QUANTITATION LIMITS (PQL)

Semi-Volatile Organics	Quantitation Limits*	
	Water	Low Soil
Phenol	4	270
bis(2-Chloroethyl)ether	4	270
2-Chlorophenol	4	270
1,2-Dichlorobenzene	4	270
1,4-Dichlorobenzene	4	270
Benzyl Alcohol	4	270
1,3-Dichlorobenzene	4	270
2-Methylphenol	4	270
2,2'-oxybis(1-Chloropropane)#	4	270
4-Methylphenol	4	270
N-Nitroso-di-n-propylamine	4	270
Hexachloroethane	4	270
Nitrobenzene	4	270
Isophorone	4	270
2-Nitrophenol	4	270
2,4-Dimethylphenol	4	270
Benzoic Acid	50	1670
bis(2-Chloroethoxy)methane	4	270
2,4-Dichlorophenol	4	1670
1,2,4-Trichlorobenzene	4	270
Naphthalene	4	270
4-Chloroaniline	4	270
Hexachlorobutadiene	4	270
4-Chloro-3-methylphenol	4	270
2-Methylnaphthalene	4	270
Hexachlorocyclopentadiene	4	270
2,4,6-Trichlorophenol	4	270
2,4,5-Trichlorophenol	10	670

Semi-Volatile Organics	Quantitation Limits*	
	Water (ug/L)	Low Soil (ug/Kg)
2-Chloronaphthalene	4	270
2-Nitroaniline	4	270
Dimethylphthalate	4	270
2,4-Dinitrotoluene	4	270
Diethylphthalate	4	270
4-Chlorophenyl-phenylether	4	270
Fluorene	4	270
4-Nitroaniline	4	270
4,6-Dinitro-2-methylphenol	10	1670
N-nitrosodiphenylamine	10	270
4-Bromophenyl-phenylether	4	270
Hexachlorobenzene	4	270
Pentachlorophenol	10	1670
Phenanthrene	4	270
Anthracene	4	270
Di-n-butylphthalate	4	270
Fluoranthene	4	270
Pyrene	4	270
Butylbenzylphthalate	4	270
3,3'-Dichlorobenzidine	4	270
Benzo(a)anthracene	4	270
Chrysene	4	270
bis(2-Ethylhexyl)phthalate	4	270
Di-n-octylphthalate	4	270
Benzo(b)fluoranthene	4	270
benzo(k)fluoranthene	4	270
Benzo(a)pyrene	4	270
Indeno(1,2,3-cd)pyrene	4	270
Dibenzo(a,h)anthracene	4	270

Benzo(g,h,i)perylene	4	270
Acenaphthylene	4	270
3-Nitroaniline	4	270
Acenaphthene	4	270
2,4-Dinitrophenol	10	1670
4-Nitrophenol	10	670
Dibenzofuran	4	270

*When a sample is reported, quantitation limits are based on wet weight. This table is to indicate the starting quantitation limit. The quantitation limits calculated by the laboratory for soil/sediment, calculated on dry weight basis as required by the contract, will be higher.

#Previously known as bis(2-chloroisopropyl)ether.

TABLE 1.1
APPENDIX IX PRACTICAL QUANTITATION LIMITS

Semi-Volatile Organics	Quantitation Limits	
	Water (ug/L)	Low Soil (ug/Kg)
1-Naphthylamine	50	1700
1,2,4-Trichlorobenzene	4	270
1,2,4,5-Tetrachlorobenzene	10	330
1,3,5-Trinitrobenzene	50	1,700
1,4-Naphthoquinone	100	3,300
2-Acetylaminofluorene	10	330
2-Chloronaphthalene	4	270
2-Chlorophenol	4	270
2-Methylnaphthalene	4	270
2-Naphthylamine	50	1,700
2,3,4,6-Tetrachlorophenol	50	1,700
2,4-Dichlorophenol	4	270
2,4-Dimethylphenol	4	270
2,4-Dinitrophenol	50	1,700
2,4-Dinitrotoluene	4	270
2,4,5-Trichlorophenol	10	670
2,4,6-Trichlorophenol	4	270
2,6-Dichlorophenol	50	1,700
2,6-Dinitroluene	4	270
3-Methylcholanthrene	10	330
3,3'-Dichlorobenzidine	4	270
3,3'-Dimethylbenzidine	50	1,700
4-Aminobiphenyl	10	330
4-Bromophenyl phenylether	4	270
4-Chlorophenyl phenylether	4	270
4,6-Dinitro-o-methylphenol	50	1,700
4-Nitroquinoline-1-oxide	50	1,700

Semi-Volatile Organics	Quantitation Limits	
	Water (ug/L)	Low Soil (ug/Kg)
Acenaphthylene	4	270
5-Nitro-o-toluidine	10	330
7,12-Dimethylbenz[a]anthracene	50	1,700
Acenaphthene	4	270
Acetophenone	4	270
alpha,alpha-Dimethylphenethylamine	100	3,300
Aniline	4	270
Anthracene	4	270
Aramite	100	3,300
Benzo[a]anthracene;Benzanthracene	4	270
Benzo[a]pyrene	4	270
Benzo[b]fluoranthene	4	270
Benzo[g,h,i]perylene	4	270
Benzo[k]fluoranthene	4	270
Benzyl alcohol	4	270
bis(2-Chloroethoxy)methane	4	270
bis(2-Chloroethyl)ether	4	270
bis(2-Ethylhexyl)phthalate	4	270
bis(2-Chloroisopropyl)ether	4	270
Butylbenzylphthalate	4	270
Chrysene	4	270
Di-n-butylphthalate	4	270
Di-n-octylphthalate	4	270
Diallate	10	330
Dibenzofuran	4	270
Dibenz[a,h]anthracene	4	270
Diethylphthalate	4	270
Dimethylphthalate	4	270
Dinoseb	10	330

Diphenylamine	10	330
Ethylmethanesulfonate	10	330
Fluoranthene	4	270
Fluorene	4	270
Hexachloroethane	4	270
Hexachlorobenzene	4	270
Hexachlorobutadiene	4	270
Hexachlorocyclopentadiene	4	270
Hexachlorophene	2000	27000
Hexachloropropene	10	330
Indeno(1,2,3-cd)pyrene	4	270
Isophorone	4	270
Isosafrole	10	330
m-Methylphenol(3&4)total	4	270
m-Dichlorobenzene (1,3)	10	330
m-Dinitrobenzene	10	330
m-Nitroaniline (3-)	4	270
Methapyrilene	10	330
Methylmethanesulfate	10	330
N-Nitroso-di-n-butylamine	10	330
N-Nitroso-diethylamine	20	670
N-Nitroso-dimethylamine	10	330
N-Nitrosodiphenylamine	10	330
N-Nitroso-di-n-propylamine	10	330
N-Nitroso-methylethylamine	10	330
N-Nitroso-morpholine	10	330
N-Nitroso-pyrrolidine	10	330
Naphthalene	4	270
Nitrobenzene	4	270
o-Toluidine	10	330
o-Dichlorobenzene (1.2-)	4	270
o-Nitroaniline (2-)	4	270

o-Nitrophenol (2)	4	270
o-Methylphenol (2)	4	270
p-Phenylenediamine	25	67000
p-Nitroaniline (4-)	4	270
p-(Dimethylamino)azobenzene	10	330
p-Nitrophenol (4-)	10	1670
p-Dichlorobenzene (1,4-)	4	270
p-Chloro-m-methylphenol	5	270
p-Chloroaniline (4-)	4	270
Pentachlorobenzene	10	330
Pentachloronitrobenzene	10	330
Pentachlorophenol	25	1670
Phenacetin	10	330
Phenanthrene	4	270
Phenol	4	270
Pronamide	10	330
Pyridine	25	830
Pyrene	4	270
Safrole	10	330

TABLE 1.2
ORGANOPHOSPOROUS PESTICIDES PRACTICAL QUANTITATION LIMITS

Semi-Volatile Organics	Quantitation Limits	
	Water (ug/L)	Low Soil (ug/Kg)
2-Picoline	10	330
o,o,o-TEPA	10	330
Thionazin	10	330
Sulfotep	10	330
Phorate	10	330
Dimethoate	10	330
Disulfoton	10	330
Methyl parathion	10	330
Parathion	10	330
Famphur	10	330

TABLE 1.3
GOLDER PRACTICAL QUANTITATION LIMITS

Semi-Volatile Organics	Quantitation Limits	
	Water (ug/L)	Low Soil (ug/Kg)
Indane	10	330
1-Propynyl Benzene	10	330
m-Toluidine	10	330
2-Chloroaniline	10	330
2-Nitrotoluene	10	330
3-Chloroaniline	10	330
Benzo (b) Thiopene	10	330
1-Chloro-2-nitrobenzene	10	330
Dichloran	10	330
Diphenamid	10	330

TABLE 1.4
TCLP PRACTICAL QUANTITATION LIMITS

Semi-Volatile Organics	Estimated Quantitation Limits ug/L
1,4-Dichlorobenzene	20
Hexachloroethane	20
Nitrobenzene	20
Hexachlorobutadiene	20
2,4,6-Trichlorophenol	20
2,4,5-Trichlorophenol	100
2,4-Dinitrotoluene	20
Hexachlorobenzene	20
Pentachlorophenol	100
2-Methylphenol	20
3&4-Methylphenol	20
Pyridine	40

TABLE 2.0
MATRIX SPIKE RECOVERY AND RELATIVE PERCENT DIFFERENCE LIMITS

Compound	% Recovery Water	RPD Water	% Recovery Soil	RPD Soil

QC limits are updated in the Laboratory Information Management System.

TABLE 3.0
SURROGATE RECOVERY LIMITS

QC limits are updated in the Laboratory Information Management System.

TABLE 4.0
DFTPP KEY IONS AND ION ABUNDANCE CRITERIA FOR
QUADRAPOLE MASS SPECTROMETERS

Mass	Ion Abundance Criteria
51	30.0 - 60 percent of mass 198
68	Less than 2.0 percent of mass 69
69	Greater than 1.00% and less than 100.00% of mass 198
70	Less than 2.0 percent of mass 69
127	40-60 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 percent of mass 198
441	Present, but less than mass 443
442	40.0 - 100.0 percent of mass 198
443	17 - 23 percent of mass 442

TABLE 5.0

Calibration Check Compounds (ccc)	
Phenol	Acenaphthene
1,4-Dichlorobenzene	N-Nitrosodiphenylamine
2-Nitrophenol	Pentachlorophenol
2,4-Dichlorophenol	Fluoranthene
Hexachlorobutadiene	Di-n-octylphthalate
4-Chloro-3-methylphenol	Benzo(a)pyrene
2,4,6-Trichlorophenol	

TABLE 5.1

System Performance Check Compounds (spcc)	
N-Nitroso-di-n-propylamine	Hexachlorocyclopentadiene
2,4-Dinitrophenol	4-Nitrophenol

TABLE 6.0
SEMI-VOLATILE INTERNAL STANDARDS WITH CORRESPONDING TARGET COMPOUNDS AND SURROGATES ASSIGNED FOR QUANTITATION

1,4-Dichlorobenzene-d_4	**Naphthalene-d_8**	**Acenaphthene-d_{10}**	**Phenanthrene-d_{10}**	**Chrysene-d_{12}**	**Perylene-d_{12}**
Phenol	Nitrobenzene	Hexachlorocyclopentadiene	4,6-Dinitro-2-	Pyrene	Di-n-octylphthalate
bis(2-Chloroethyl)ether	Isophorone	2,4,6-Trichlorophenol	N-Nitrosodiphenylamine	Butylbenzylphthalate	Benzo(b)fluoranthene
2-Chlorophenol	2-Nitrophenol	2-Chloronaphthalene*	4-Bromophenyl phenyl	3,3'-Dichlorobenzidine	Benzo(k)fluoranthene
1,3-Dichlorobenzene	2,4-Dimethyl-phenol	2-Nitroaniline	Hexachlorobenzene	Benzo(a)anthracene	Benzo(a)pyrene
1,4-Dichlorobenzene	bis(2-Chloroethoxy)methane	Dimethylphthalate	Pentachlorophenol	bis(2-Ethylhexyl)phthalate	Indeno(1,2,3-cd)pyrene
1,2-Dichlorobenzene	2,4-Dichlorophenol	Acenaphthylene	Phenanthrene	Chrysene	Dibenz(a,h)anthracene
2-Methylphenol	1,2,4-Trichlorobenzene	3-Nitroaniline	Anthracene	Terphenyl-d_{14} (surr)	Benzo(g,h,i)perylene
2,2'-oxybis-(1-	Naphthalene	Acenaphthene	Di-n-butylphthalate		
4-Methylphenol	4-Chloroaniline	2,4-Dinitrophenol	Fluoranthene		
N-Nitroso-di-n-	Hexachlorobutadiene	4-Nitrophenol			
Hexachloroethane	4-Chloro-3-methylphenol	Dibenzofuran			
2-Fluorophenol (surr)	2-Methylnaphthalene	2,4-Dinitrotoluene			
Phenol-d_5 (surr)	Nitrobenzene-d_5 (surr)	2,6-Dinitrotoluene			
		2,4,5-trichlorophenol			
		Diethylphthalate			
		4-Chlorophenyl phenyl			
		Fluorene			
		4-Nitroaniline			
		2-Fluorobiphenyl (surr)			
		2,4,6-Tribromophenol (surr)			

TABLE 7.0
CHARACTERISTIC IONS FOR INTERNAL STANDARDS
FOR SEMI-VOLATILE COMPOUNDS

Internal Standards	Primary Ion	Secondary Ions
1,4-Dichlorobenzene-d_4	152	115
Naphthalene-d_8	136	68
Acenaphthene-d_{10}	164	162, 160
Phenanthrene-d_{10}	188	94, 80
Chrysene-d_{12}	240	120, 236
Perylene-d_{12}	264	260, 265

TABLE 8.0
CHARACTERISTIC IONS FOR SEMI-VOLATILE TARGET COMPOUNDS AND SURROGATES

Parameter	Primary 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
2,2'-oxybis(1-Chloropropane)	45	77, 79
4-Methylphenol	108	107
N-Nitroso-di-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
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
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
Dibenz(a,h)anthracene	278	139, 279
Benzo(g,h,i)perylene	276	138, 277

SURROGATES

Parameter	Primary Ion	Secondary Ion(s)
Phenol-d_5	99	42, 71
2-Fluorophenol	112	64
2,4,6-Tribromophenol	330	332, 141
Nitrobenzene-d_5	82	128, 54
2-Fluorobiphenyl	172	171
Terphenyl	244	122, 212

Table 9.0
8270 Curve Concentrations

Compounds	Level 1 ug/ml	Level 2 ug/ml	Level 3 ug/ml	Level 4 ug/ml	Level 5 ug/ml	Level 6 ug/ml
Pyridine	4	10	20	40	60	80
n-nitrosodimethylamine	4	10	20	40	60	80
cyclohexanone	4	10	20	40	60	80
phenol	4	10	20	40	60	80
bis(2-chloroethyl)ether	4	10	20	40	60	80
1,3-dichlorobenzene	4	10	20	40	60	80
1,4-dichlorobenzene	4	10	20	40	60	80
1,2-dichlorobenzene	4	10	20	40	60	80
benzyl alcohol	4	10	20	40	60	80
2-methylphenol	4	10	20	40	60	80
bis(2-chloroisopropyl)ether	4	10	20	40	60	80
n-nitroso-di-n-propylamine	4	10	20	40	60	80
hexachloroethane	4	10	20	40	60	80
4-methylphenol	4	10	20	40	60	80
2-chlorophenol	4	10	20	40	60	80
nitrobenzene	4	10	20	40	60	80
bis(2-chloroethoxy)methane	4	10	20	40	60	80
1,2,4-trichlorobenzene	4	10	20	40	60	80
isophorone	4	10	20	40	60	80
2,4-Dimethylphenol	4	10	20	40	60	80
Benzoic Acid	10	25	30	40	60	80
Hexachlorobutadiene	4	10	20	40	60	80
Naphthalene	4	10	20	40	60	80
2,4-Dichlorophenol	4	10	20	40	60	80
4-chloroaniline	4	10	20	40	60	80
2,4,6-Trichlorophenol	4	10	20	40	60	80
2,4,5-Trichlorophenol	10	25	30	40	60	80
2,4,5-Trichlorotoluene	4	10	20	40	60	80
Hexachlorocyclopentadiene	4	10	20	40	60	80
2-Methylnaphthalene	4	10	20	40	60	80
2-Nitroaniline	4	10	20	40	60	80
2-Chloronaphthalene	4	10	20	40	60	80
4-Chloro-3-methylphenol	4	10	20	40	60	80
2,6-Dinitrotoluene	4	10	20	40	60	80
2-Nitrophenol	4	10	20	40	60	80
3-Nitroaniline	4	10	20	40	60	80
Dimethyl phthalate	4	10	20	40	60	80
2,4-Dinitrophenol	10	25	30	40	60	80
Acenaphthylene	4	10	20	40	60	80
2,4-Dinitrotoluene	4	10	20	40	60	80
Acenaphthene	4	10	20	40	60	80
Dibenzofuran	4	10	20	40	60	80
4-nitrophenol	10	25	30	40	60	80
Fluorene	4	10	20	40	60	80

4-Nitroaniline	4	10	20	40	60	80
azobenzene = 1,2-diphenylhydrazine	4	10	20	40	60	80
4-Bromophenyl phenyl ether	4	10	20	40	60	80
Hexachlorobenzene	4	10	20	40	60	80
Diethyl phthalate	4	10	20	40	60	80
4-Chlorophenyl phenyl ether	4	10	20	40	60	80
pentachlorophenol	10	25	30	40	60	80
n-nitrosodiphenylamine=diphenylamine	4	10	20	40	60	80
4,6-Dinitro-2-methylphenol	10	25	30	40	60	80
Phenanthrene	4	10	20	40	60	80
Carbazole	4	10	20	40	60	80
Anthracene	4	10	20	40	60	80

Table 9.0
8270 Curve Concentrations (continued)

Compounds	**Level 1 ug/ml**	**Level 2 ug/ml**	**Level 3 ug/ml**	**Level 4 ug/ml**	**Level 5 ug/ml**	**Level 6 ug/ml**
Di-n-butyl phthalate	4	10	20	40	60	80
Fluoranthene	4	10	20	40	60	80
Benzidine	4	10	20	40	60	80
Pyrene	4	10	20	40	60	80
Butyl benzyl phthalate	4	10	20	40	60	80
Benzo(a)anthracene	4	10	20	40	60	80
Chrysene	4	10	20	40	60	80
3,3-Dichlorobenzidine	4	10	20	40	60	80
Bis(2-ethylhexyl)phthalate	4	10	20	40	60	80
Di-n-octyl phthalate	4	10	20	40	60	80
Benzo(b)fluoranthene	4	10	20	40	60	80
Benzo(k)fluoranthene	4	10	20	40	60	80
Benzo(a)pyrene	4	10	20	40	60	80
Indeno(1,2,3-cd)pyrene	4	10	20	40	60	80
Dibenzo(a,h)anthracene	4	10	20	40	60	80
Benzo(ghi)perylene	4	10	20	40	60	80
Acetophenone	4	10	20	40	60	80
benzaldehyde	4	10	20	40	60	80
caprolactam	4	10	20	40	60	80
1,1'-Biphenyl	4	10	20	40	60	80
atrazine	4	10	20	40	60	80
2-Fluorophenol	4	10	20	40	60	80
Phenol-d5	4	10	20	40	60	80
Nitrobenzene-d5	4	10	20	40	60	80
2-Fluorobiphenyl	4	10	20	40	60	80
2,4,6-Tribromophenol	10	25	30	40	60	80
Terphenyl-d14	4	10	20	40	60	80

Table 9.1

Appendix 9 Curve Concentrations

Compounds	Level 1 ug/ml	Level 2 ug/ml	Level 3 ug/ml	Level 4 ug/ml	Level 5 ug/ml	Level 6 ug/ml	Level 7 ug/ml
Methyl methane sulfonate	5	10	25	40	60	80	100
n-Nitrosomethylethylamine	5	10	25	40	60	80	100
n-Nitrosodiethylamine	5	10	25	40	60	80	100
Ethyl methane sulfonate	5	10	25	40	60	80	100
n-Nitrosopyrolidine	5	10	25	40	60	80	100
p-Phenylenediamine	5	10	25	40	60	80	100
n-Nitrosopiperidine	5	10	25	40	60	80	100
Hexachloropropene	5	10	25	40	60	80	100
n-Nitrosodi-n-butylamine	5	10	25	40	60	80	100
2,6-Dichlorophenol	5	10	25	40	60	80	100
Safrole	5	10	25	40	60	80	100
1,2,4,5-Tetrachlorobenzene	5	10	25	40	60	80	100
Isosafrole	5	10	25	40	60	80	100
1,4-Naphthoquinone	5	10	25	40	60	80	100
m-Dinitrobenzene	5	10	25	40	60	80	100
1,3,5-trinitrobenzene	5	10	25	40	60	80	100
Pentachlorobenzene	5	10	25	40	60	80	100
1-Naphthylamine	5	10	25	40	60	80	100
2,3,4,6-Tetrachlorophenol	5	10	25	40	60	80	100
2-Naphthylamine	5	10	25	40	60	80	100
5-Nitro-o-toluidine	5	10	25	40	60	80	100
4-Aminobiphenyl	5	10	25	40	60	80	100
Diallate	5	10	25	40	60	80	100
Phenacetin	5	10	25	40	60	80	100
Pronamide	5	10	25	40	60	80	100
Pentachloronitrobenzene	5	10	25	40	60	80	100
3,3-Dimethylbenzidine	5	10	25	40	60	80	100
Methapyrilene (=HCL)	5	10	25	40	60	80	100
p-Dimethylaminoazobenzene	5	10	25	40	60	80	100
2-Acetylaminofluorene	5	10	25	40	60	80	100
7,12-Dimethylbenz(a)anthracene	5	10	25	40	60	80	100
3-Methylcholanthrene	5	10	25	40	60	80	100
Aniline	5	10	25	40	60	80	100
Dinoseb	5	10	25	40	60	80	100
4-nitroquinoline-1-oxide	5	10	25	40	60	80	100
n-nitrosomorpholine	5	10	25	40	60	80	100
1-methylnaphthalne	5	10	25	40	60	80	100
alpha,alpha-dimethylphenethylamine	5	10	25	40	60	80	100
aramite	5	10	25	40	60	80	100
hexachlorophene	125	250	400	500	600	800	1000
kepone	5	10	25	40	60	80	100
1-chloro-2-nitrobenzene	5	10	25	40	60	80	100
2-chloroaniline	5	10	25	40	60	80	100
3-chloroaniline	5	10	25	40	60	80	100
dichloran	5	10	25	40	60	80	100

diphenamid	5	10	25	40	60	80	100
m-toluidine	5	10	25	40	60	80	100
indane	5	10	25	40	60	80	100
1-propynyl benzene	5	10	25	40	60	80	100
2-nitrotoluene	5	10	25	40	60	80	100
benzo (b) thiopene	5	10	25	40	60	80	100

Level 7 is only run when Hexachlorophene cannot be seen at the Level 1. Level 7 will only be included in the calibration in these cases.

Table 9.1
Appendix 9 Curve Concentrations(continued)

Compounds	Level 1 ug/ml	Level 2 ug/ml	Level 3 ug/ml	Level 4 ug/ml	Level 5 ug/ml	Level 6 ug/ml
2-picoline	5	10	25	40	60	80
o,o,o-TEPA	5	10	25	40	60	80
thionazin	5	10	25	40	60	80
sulfotep	5	10	25	40	60	80
phorate	5	10	25	40	60	80
dimethoate	5	10	25	40	60	80
disulfoton	5	10	25	40	60	80
methyl parathion	5	10	25	40	60	80
patathion	5	10	25	40	60	80
famphur	5	10	25	40	60	80

Table 9.2
Zeneca Curve Concentrations

Compounds	Level 1 ug/ml	Level 2 ug/ml	Level 3 ug/ml	Level 4 ug/ml	Level 5 ug/ml	Level 6 ug/ml
EPTC	5	10	20	30	40	50
Butylate	5	10	20	30	40	50
Vernolate	5	10	20	30	40	50
Pebulate	5	10	20	30	40	50
R-25788	5	10	20	30	40	50
Molinate	5	10	20	30	40	50
Cycloate	5	10	20	30	40	50
Fonofos	5	10	20	30	40	50
Ametryn	5	10	20	30	40	50
Carbophenothion	5	10	20	30	40	50
Napropamide	5	10	20	30	40	50

Table 10
Standards/Manufacturer

General	Storage	Mfgr	Description	Conc.	Part #

	Loc				
				(ug/ml)	
Category 8270/625	F	Supelco	8270 Cal Mix 1	1000	#5-06508
	R	Supelco	8270 Cal Mix 4	2000	#86-1148
	R	Supelco	n-nitrosodiphneylamine	5000	#46702-U
	R	Supelco	CLP OLM04 SV Mix	2000	#47514-U
	R	Supelco	8270 Surrogate Std. Mix	4000	#86-1155
	F	Chemserv	Cyclohexanone	2000	F2326AS
	R	Supelco	EPA 8270 Benzidines Mix	2000	#4-8467
	R	Supelco	2,4,5-Trichlorotoluene	2000	#4-8150
	R	Supelco	4,4'-DDT	5000	#40124
	R	Supelco	DFTPP	2000	#4-8082
	R	Restek	L/C phenol Mix A	2000	#31208
	R	Supelco	Benzoic acid	2000	#47508-U
ISTD	R	Supelco	Equity IS Mix	2000	#46955-U
SIM	R	Supelco	5surr (custom) methanol	1000	20966387-10
2nd source	R	NSI	Check A	100	#Q-1155A
	R	NSI	Check B	100	#Q-1155B
Appendix 9	F	Supelco	Appendix 9 SV cal mix	1000	#506567
	R	Supelco	Appendix 9 SV cal mix 2	2000	#86-1141
	R	Supelco	Hexachlorophene	5000	#4-0323
	R	Supelco	p-Phenylenediamine	2000	#48298
	R	Supelco	Aramite	2000	#47519U
	F	SpexCertiprep	Kepone	2000	#8270-IXD
Golder	R	Restek	Golder combined	2000	559391
OP Pest	R	UltraSci	OP pest mix IN MECL2	2000	US-119
	A	UltraSci	2-Picoline	5000	EPA-1156
	F	Accustd	2-aminopyridine	1000	S-16428-10x
Zeneca	A	Accustd	EPTC	991	P-238S-10X
	A	Accustd	Butylate	1001	P-088S-10X
	A	Accustd	Vernolate	1001	P-111S-10X
	A	Accustd	Pebulate (Tillam)	1000	P-105S-10X
	A	Accustd	Molinate	1000	P-176S-10X
	A	Accustd	Cycloate	991	P-248S-10X
	A	Accustd	Fonofos (Dyfonate)	1000	P-087S-

					10X
	A	Accustd	Ametryn	988	P-003S-10X
	A	Accustd	Napropamide	1002	P-179S-10X
	A	Accustd	Carbophenothion	1000	P-095S-10X
	R	Crescent	R-25788 (Dichlormid)	1000	#8603M.10
Misc.	**R**	Restek	Toluic Acid	1000	#555147
	R	Restek	4-aminopyridine	10000	#CS-6136-1

Storage Codes
A = Ambient Storage
R = Refrigerator Storage
F = Freezer Storage

Table 11.0
Standard Preparation

8270 Curve	Initial Conc.	Amount Used	Final Volume	Final Conc.	Prepare in Mecl2
OLM Intermediate 1	ug/ml	mls	mls	ug/ml	SolmINT1_
8270 Cal Mix 1 (Supelco #5-06508)	1000	2.0	10	200	
8270 Cal Mix 4 (Supelco #86-1148)	2000	1.0	10	200	
n-nitrosodiphneylamine (Supelco #46702-U)	5000	0.40	10	200	
CLP OLM04 SV Mix (Supelco #47514-U)	2000	1.0	10	200	
8270 Surrogate Std. Mix (Supelco #86-1155)	4000	0.50	10	200	
OLM Intermediate 2					SolmINT2_
Cyclohexanone	2000	1.0	10	200	
EPA 8270 Benzidines Mix (Supelco #4-8467)	2000	1.0	10	200	
2,4,5-Trichlorotoluene (Supelco #4-8150)	2000	1.0	10	200	

NOTE: Levels 4/10/20 are all fortified with Acid solutions to bring up the total concentration.

4/10ppm BNA std	Initial Conc. ug/ml	Amount Used mls	Final Volume mls	Final Conc. ug/ml	S8270lv4
OLM Intermediate #1	200	0.040	2	4	
OLM Intermediate #2	200	0.040	2	4	
Benzoic Acid	2000	0.006	2	6	10ug/ml total
L/C phenol Mix A (Restek #31208)	2000	0.006	2	6	10ug/ml for select acids
10/25ppm BNA std					S8270lv10
OLM Intermediate #1	200	0.100	2	10	
OLM Intermediate #2	200	0.100	2	10	
Benzoic Acid	2000	0.015	2	15	25ug/ml total
L/C phenol Mix A (Restek #31208)	2000	0.015	2	15	25ug/ml for select acids
20/30ppm BNA std					S8270lv20
OLM Intermediate #1	200	0.200	2	20	
OLM Intermediate #2	200	0.200	2	20	
Benzoic Acid	2000	0.010	2	10	30ug/ml total
L/C phenol Mix A (Restek #31208)	2000	0.010	2	10	30ug/ml for select acids
40ppm BNA std					S8270lv40
OLM Intermediate #1	200	0.400	2	40	
OLM Intermediate #2	200	0.400	2	40	
DFTPP	2000	0.050	2	50	
4,4'-DDT	5000	0.010	2	25	

60ppm BNA std					S8270lv60
OLM Intermediate #1	200	0.600	2	60	
OLM Intermediate #2	200	0.600	2	60	
80ppm BNA std					S8270lv80
OLM Intermediate #1	200	0.800	2	80	
OLM Intermediate #2	200	0.800	2	80	
400ppm ISTD Mix					SISTDWRK20_
Equity IS Mix (Supelco #46955-U)	2000	2.000	10	400	SISTDSTK_
25ppm DFTPP std					
DFTPP (Supelco #4-8082)	2000	0.025	2	25	Sdftpwrk25

Appendix 9 Curve	Initial Conc.	Amount Used	Final Volume	Final Conc.	Prepare in Mecl2
AP9 Intermediate 1	ug/ml	mls	mls	ug/ml	**SAP9INT_**
Appendix 9 SV cal mix (Supelco #506567)	1000	1	5	200	SAP9STK_
Appendix 9 SV cal mix 2 (Supelco #86-1141)	2000	0.5	5	200	SAP9-2STK_
p-Phenylenediamine (Supelco #48298)	2000	0.5	5	200	SPHENYLSTK_
Aramite (Supelco #47519U)	2000	0.5	5	200	SARAMTSTK_00001
Organophosphorous Mix (US US-119)	2000	0.5	5	200	S_OP_STK
2-aminopyridine(Accustd-S16428-10x)	1000	1	5	200	S2AMPYRSTK_
Hexachlorophene (Supelco #4-0323)	5000ug/ml			5000ug/ml	SHXPHNSTK_

2/75 ppm AP9 std	Initial Conc.	Amount Used	Final Volume	Final Conc.	**SAP9lv2**
Appendix 9 Intermediate	200	0.020	2	2	SAP9INT_
Hexachlorophene	5000	0.030	2	75	SHXPHNSTK_

Need level 2/75 only if running for Dioxane

5/125ppm AP9 std	Initial Conc.	Amount Used	Final Volume	Final Conc.	**SAP9lv5**
10/250ppm ap9 std	200	0.05	2	5.0	SAP9INT_
Hexachlorophene	5000	0.05	2	125	SHXPHNSTK_

10/250 ppm AP9 std	Initial Conc.	Amount Used	Final Volume	Final Conc.	**SAP9lv10**
Appendix 9 Intermediate	200	0.1	2	10	SAP9INT_
Hexachlorophene	5000	0.1	2	250	SHXPHNSTK_

25/400 ppm AP9 std	Initial Conc.	Amount Used	Final Volume	Final Conc.	**SAP9lv25**
Appendix 9 Intermediate	200	0.25	2	25	SAP9INT_
Hexachlorophene	5000	0.16	2	400	SHXPHNSTK_

40/500 ppm AP9 std	Initial Conc.	Amount Used	Final Volume	Final Conc.	**SAP9lv40**
Appendix 9 Intermediate	200	0.4	2	40	SAP9INT_
Hexachlorophene	5000	0.2	2	500	SHXPHNSTK_

60/600 ppm AP9 std	Initial Conc.	Amount Used	Final Volume	Final Conc.	**SAP9lv60**
Appendix 9 Intermediate	200	0.6	2	60	SAP9INT_
Hexachlorophene	5000	0.24	2	600	SHXPHNSTK_

80/800 ppm AP9 std	Initial Conc.	Amount Used	Final Volume	Final Conc.	**SAP9lv80**
Appendix 9 Intermediate	200	0.8	2	80	SAP9INT_
Hexachlorophene	5000	0.32	2	800	SHXPHNSTK_

Zeneca Curve	Initial Conc. ug/ml	Amount Used	Final Volume	Final Conc.	Prepare in Mecl2
Zeneca Intermediate					SZENECINT_
EPTC (P-238S-10x Accustd)	991	1	10	99.1	SEPTC_STK_
Butylate (P-088S-10X Accustd)	1001	1	10	100.1	SBUTYL_STK_
Vernolate (P-111S-10X Accustd)	1001	1	10	100.1	SVERNO_STK_
Pebulate (Tillam) (P-105S-10X Accustd))	1000	1	10	100.0	STILL_STK_
Molinate (P-176S-10X)	1000	1	10	100.0	SMOLIN_STK_
Cycloate (P-248S-10X)	991	1	10	99.1	SCYCLO_STK_
Fonofos (Dyfonate) P-087S-10X	1000	1	10	100.0	SDYFON_STK_
Ametryn (P-003S-10X)	988	1	10	98.8	SAMETR_STK_
Napropamide (p-179S-10X)	1002	1	10	100.2	SNAPRO_STK_
Carbophenothion (P-095S-10X)	1000	1	10	100.0	SCARBO_STK_

5.0ppm Level 1 Standard	Initial Conc.	Amount Used	Final Volume	Final Conc.	**SZENEClv1**
Zeneca Intermediate	100	0.1	2	5.00	SZENECINT_
R-25788 (Dichlormid) (Crescent 8603)	1000	0.01	2	5.00	SDICLMDSTD

10ppm Level 2 Standard	Initial Conc.	Amount Used	Final Volume	Final Conc.	**SZENEClv2**
Zeneca Intermediate	100	0.2	2	10.00	SZENECINT_
R-25788 (Dichlormid) (Crescent 8603)	1000	0.02	2	10.00	SDICLMDSTD

20ppm Level 3 Standard	Initial Conc.	Amount Used	Final Volume	Final Conc.	**SZENEClv3**
Zeneca Intermediate	100	0.4	2	20.00	SZENECINT_
R-25788 (Dichlormid) (Crescent 8603)	1000	0.04	2	20.00	SDICLMDSTD

30ppm Level 4 Standard	Initial Conc.	Amount Used	Final Volume	Final Conc.	**SZENEClv4**
Zeneca Intermediate	100	0.60	2	30.00	SZENECINT_
R-25788 (Dichlormid) (Crescent 8603)	1000	0.06	2	30.00	SDICLMDSTD

40ppm Level 5 Standard	Initial Conc.	Amount Used	Final Volume	Final Conc.	**SZENEClv5**
Zeneca Intermediate	100	0.8	2	40.00	SZENECINT_
R-25788 (Dichlormid) (Crescent 8603)	1000	0.08	2	40.00	SDICLMDSTD

50ppm Level 6 Standard	Initial Conc.	Amount Used	Final Volume	Final Conc.	SZENEClv6
Zeneca Intermediate	100	1	2	50.00	SZENECINT_
R-25788 (Dichlormid) (Crescent 8603)	1000	0.1	2	50.00	SDICLMDSTD

SIM-8270 Curve Prepare in Mecl2

SIM Intermediate	Initial Conc. ug/ml	Amount Used ml	Final Volume ml	Final Conc. ug/ml	SsimINT_
OLM Intermediate #1	200	1.25	5	50	SolmINT1_
OLM Intermediate #2	200	1.25	5	50	SolmINT2_
Sim Surrogate (Supleco #20619885)	1000	0.25	5	50	SSIMSU5STK_

	Initial Conc.	Amount Used	Final Volume	Final Conc.	
0.1ppm SIM std					Ssimlv0.1_
SIM Intermediate	50	0.004	2	0.10	
0.25ppm SIM std					Ssimlv0.25_
SIM Intermediate	50	0.01	2	0.25	
0.50ppm SIM std					Ssimlv0.5_
SIM Intermediate	50	0.02	2	0.50	
1.0ppm SIM std					Ssimlv1.0_
SIM Intermediate	50	0.04	2	1.00	
2.5ppm SIM std					Ssimlv2.5_
SIM Intermediate	50	0.1	2	2.50	
5.0ppm SIM std					Ssimlv5.0_
SIM Intermediate	50	0.2	2	5.00	
10ppm SIM std					Ssimlv10_
SIM Intermediate	50	0.4	2	10.00	

Low Concentration ISTD Mix					SISTDWRK.5_
400ppm ISTD Mix	400ug/ml	125ul	5mls	10ug/ml	

FIGURE 1.0
MASS SPECTRAL INTERPRETATION CHECKLIST

The following compounds are commonly mis-identified due to mass spectral interpretation errors. Please review the mass spectrum and retention times of the following compounds for the described problems when making identification decisions.

COMPOUND	m/z	RT(*)	Potential Identification Problem
1,4-dichlorobenzene-d4	152	6.40	misidentified as 1,2-dichlorobenzene-d4
1,2-dichlorobenzene-d4	152	6.67	misidentified as 1,4-dichlorobenzene-d4
aniline(TIC)	93	5.99	misidentified as bis(2-chloroethyl)ether
bis(2-chloroethyl)ether	93	6.05	misidentified as aniline
1,3-dichlorobenzene	146	6.35	misidentified as 1,4 or 1,2-isomer
1,4-dichlorobenzene	146	6.42	misidentified as 1,3 or 1,2-isomer
1,2-dichlorobenzene	146	6.69	misidentified as 1,3 or 1,4-isomer
4-chlorophenol (TIC)	128	8.22	misidentified as naphthalene
naphthalene	128	8.22	misidentified, actually 4-chlorophenol)
benzyl alcohol	108	6.62	not identified, coelutes with surrogate 1,2-dichlorobenzene-d4 or misidentified with 2-methylphenol or 4-methylphenol
2-methylphenol	108	6.79	misidentified as benzyl alcohol or 4-methylphenol
4-methylphenol	108	6.98	misidentified as benzyl alcohol or 2-methylphenol
2,4,6-trichlorophenol	196	9.42	misidentified as 2,4,5-isomer
2,4,5-trichlorophenol	196	9.46	misidentified as 2,4,6-isomer
2-nitroaniline	138	9.76	misidentified as 3-nitroaniline or 4-nitroaniline
3-nitroaniline	138	10.17	misidentified as 2-nitroaniline or 4-nitroaniline
4-nitroaniline	138	10.74	misidentified as 2-nitroaniline or 3-nitroaniline
phenanthrene	178	11.54	misidentified as anthracene
anthracene	178	11.58	misidentified as phenanthrene
flouranthene	202	12.54	misidentified as pyrene
pyrene	202	12.74	misidentified as fluoranthene
benzo(a)anthracene	228	13.72	misidentified as chrysene
chrysene	228	13.76	misidentified as benzo(a)anthracene
benzo(b)fluoranthene	252	14.84	misidentified as benzo(k)fluoranthene or benzo(a)pyrene
benzo(k)flouranthene	252	14.86	misidentified as benzo(b)fluoranthene or benzo(a)pyrene
benzo(a)pyrene)	252	15.24	misidentified as benzo(b)fluoranthene or benzo(k)flouranthene
indeno(1,2,3-cd)pyrene	276	16.94	misidentified as dibenz(a,h)anthracene or benzo(ghi)perlyene
benzo(ghi)perylene	276	17.43	misidentified as indeno(1,2,3-cd)pyrene
dimethylphthalate	149	9.95	misidentified as another phthalate
diethylphthalate	149	10.61	misidentified as another phthalate
di-n-butylphthalate	149	11.99	misidentified as another phthalate
butylbenzylphthalate	149	13.23	misidentified as another phthalate
bis-2-ethylhexylphthalate	149	13.68	misidentified as another phthalate
di-n-octylphthalate	149	14.24	misidentified as another phthalate

* - The retention times listed will vary according to column length but the elution order will not.

Appendix A
Semivolatile Sim Analysis

1. Prior to the start of any analysis, the instrument must meet the ion abundance criteria for 50ng or less of DFTPP injected in the scan mode. Additionally, the Electron Multiplier voltage in the DFTPP method must be identical to the voltage in the SIM method. Standards and/or samples can be analyzed for twelve hours from the injection of a compliant DFTPP Tune. The last injection must occur within a twelve hour period, but the run can end outside of the twelve hours.

2. The SIM method should contain a primary ion and at least one (but preferably two) confirmatory ions for each compound. There are some compounds for which only a primary ion is practical, such as Benzidine. The ions for the compounds will be placed in groups based on retention times of the compounds. The total cycle time for the ions in a group should not exceed one second. The dwell time for any ion in a group is typically 30-100msec.

3. The initial calibration curve will consist of at least 5 points. The typical calibration curve consists of the following levels: 0.1ng/ul, 0.25ng/ul, 0.5ng/ul, 1.0ng/ul, 2.5ng/ul, 5.0ng/ul, and 10ng/ul. The continuing calibration level is 0.5ng/ul. There are known problems with pentachlorophenol responding poorly at low concentrations, and that compound may be reported using a four point initial calibration. However, if that occurs, it must be noted in the case narrative. The internal standards used are the same as those for the full SCAN analysis however the concentration added is at 0.5ng/ul.

4. The SIM reporting list *with example retention times and suggested groupings) is presented below:

Compound	Ions	RT	Quant IS
Group 1			
Phenol	94,65,66	1	
Aniline	93,66	1	
2-Chlorophenol	128,64,130	1	
1,4-dichlorobenzene-d4(I1)	152,115	3.02	n/a
Group 2			
2-Methylphenol	108,107		
4-Methylphenol	108,107		
Hexachloroethane	117,201,199	1	
2,4-dichlorophenol	162,164,98		
Group 3			
Naphthalene-d8(I2)	136,68	4.25	n/a
Naphthalene	128,129,127	4.27	2
Hexachlorobutadiene	225,223,227	4.40	
2-Methylnaphthalene	142,141	4.97	2
Group 4			
Acenaphthylene	152,151,153	5.89	3
Acenapthene-d10(I3)	164,162,160	6.04	n/a
Acenapthene	153,154,1542	6.07	3

Group 5			
Fluorene-d10(S)	176,88,177	6.59	3
Fluorene	166,165,167	6.62	3
Hexachlorobenzene	284,142,249	7.17	
Group 6			
Pentachlorophenol	266,264,268	7.40	4
Group 7			
Phenathrene-d10(I4)	188,94,189	7.58	n/a
Phenathrene	178,179,176	7.61	4
Anthracene	178,176,179	7.66	4
Group 8			
Fluoranthene	202,101,100	8.85	4
Benzidine	184		
Pyrene-d10(S)	212,106,213	9.057	4
Pyrene	202,101,100	9.06	5
3,3'-Dimethylbenzidine	212,196		
Group 9			
3,3'-Dichlorobenzidine	252,254,126	10.33	
Bis(2-ethylhexyl)phthalate	149,167,279	10.41	5
Chrysene-d12(I5)	240,120,236	10.35	n/a
Benzo(a)anthracene	228,229,226	10.33	5
Chrysene	228,229,226	10.37	5
Group 10			
Benzo(b)fluoranthene	252,253,125	11.38	6
Benzo(k)fluoranthene	252,253,125	11.41	6
Benzo(a)pyrene	252,253,125	11.70	6
Perylene-d12(I6)	264,260,265	11.76	n/a
Group 11			
Indeno(1,2,3-cd)pyrene	276,138,277	13.00	6
Dibenz(ah)anthracene	278,139,276	13.03	6
Benzo(ghi)perlyene	276,138,277	13.36	6

5. An Initial calibration is considered acceptable, and the average response factor may be used for quantitation if the overall average of the %RSD is less than 25%. The continuing calibration verification is considered acceptable if the % difference from the curve is <= 20%. Pentachlorophenol and bis(2-ethylhexyl)phthalate will be allowed up to 40% difference in the continuing calibration verification.

6. To allow for the analysis of SIM and SCAN on the same extract, three additional surrogates are added to the samples prior to extraction. They are Fluorene-d10 and Pyrene-d10 for the base/neutral fraction and 2,4-dichlorophenol-d3 for the acid portion. They are added at a concentration of 1.0ng/ul in the final extract, and until sufficient data has been gathered to generate laboratory specific limits, an acceptance range of 30-150% recovery has been established for both waters and soils.

7. A SIM LCS will be extracted with every extraction batch (in addition to the SCAN LCS). The LCS will be spiked at 0.5ug/L (16.7ug/Kg for soils). Laboratory generated limits are used for all compounds. If there is insufficient data available to generate limits, an acceptance range of 30-150% will be used for both waters and soils. Re-extraction of the batch will not be required if bis(2-ethylhexyl)phthalate recovers above acceptance limits because as the spike amount approaches potential background levels, the apparent recoveries of this compound will be biased high.

8. If the client requests a SIM MS/MSD, it will be performed using the same spiking solution as is used for the LCS. The recovery limits will be the same as for the LCS, and the % RPD will be set at 50%. If the MS/MSD does not meet those limits, it will be noted in the case narrative.

9. The reporting limit will be set at the low point in the curve, which will be 0.1ug/L (6.7u/Kg for soils) with the exception of pentachlorophenol, which will be 1.0ug/L (67ug/Kg for soils), and bis(2-ethylhexyl)phthalate, which will be 0.5ug/L(34ug/kg for soils). These limits can be decreased by a factor of 2 by concentrating the extracts to half the typical final volume.

10. Method blanks should not contain any target compound contamination greater than the reporting limit (with the exception of bis(2-ethylhexyl)phthalate, which may be 5 times the RL. However, since this is a trace analytical method there may be instances where this is not achieved. In that case, the client will be contacted to determine if re-extraction is required.

Appendix C-3

Supporting Information for TCL Pesticides

Organochlorine Pesticides (GC)	8081A

Analyte Description	CAS Number
4,4'-DDD	72-54-8
4,4'-DDE	72-55-9
4,4'-DDT	50-29-3
Aldrin	309-00-2
alpha-BHC	319-84-6
beta-BHC	319-85-7
DCB Decachlorobiphenyl	2051-24-3
delta-BHC	319-86-8
Dieldrin	60-57-1
Endosulfan I	959-98-8
Endosulfan II	33213-65-9
Endosulfan sulfate	1031-07-8
Endrin	72-20-8
Endrin aldehyde	7421-93-4
Endrin ketone	53494-70-5
gamma-BHC (Lindane)	58-89-9
Heptachlor	76-44-8
Heptachlor epoxide	1024-57-3
Methoxychlor	72-43-5
Tetrachloro-m-xylene	877-09-8
Toxaphene	8001-35-2
alpha-Chlordane	5103-71-9
gamma-Chlordane	5103-74-2

Title: SOP for Pesticides
[Method 8081A]

Approvals (Signature/Date):

Technical Manager	Date	Health & Safety Manager / Coordinator	Date
Quality Assurance Manager	Date	Laboratory Director	Date

Facility Distribution No. _____ **Distributed To:_____**

1.0 APPROVALS

The approval of the Laboratory Director, the Facility QA Manager, Group Leader and Health and Safety Officer signify that the document has been prepared in accordance with the format requirements that have been established in this document. In addition, if the document is a Method SOP the signature signifies that it meets the specified requirements.

2.0 SCOPE AND APPLICATION

2.1 This method defines the specific steps for analyzing and determining the concentration of various organochlorine pesticides in multimedia, multi-concentration samples.

2.2 It is the policy of TestAmerica and of the chromatography group to ensure that we administer contracts and orders for goods and services in a manner that is fully compliant with governmental laws and regulations, as well as the TestAmerica Policy Statement on Business, Ethics and Conductivity.

2.3 Refer to Table 6 of this SOP for the list of parameters.

2.4 The document control number for this SOP is CT-GCS-22, revision 9.CT.

3.0 TERMS AND DEFINITIONS

3.1 There are many definitions used with in the laboratory, which may be generic to all laboratory analyses, or more specific for certain methods. For the most recent terms and definitions used with in the laboratory, reference the SOP for Terms and Definitions.

4.0 SUMMARY OF METHOD

4.1 This method outlines the gas chromatographic procedure for the detection of organochlorine pesticides. Samples are extracted using the proper extraction technique. The extracts are analyzed by gas chromatography with an electron capture detector.

4.2 This SOP is based on the following methods:

- EPA Method 8081A (Organochlorine Pesticides by Gas Chromatography)
- EPA Method 8000B (Gas Chromatography)
- EPA Method 8000C (Gas Chromatography)

4.3 Deviations to Method

4.3.1 A modification is that our QC check solution does not routinely contain multi-component pesticides due to peak interferences between the multi-component chromatograms and single-component peaks.

4.3.2 Toxaphene or Technical chlordane QC checks are prepared if required by for a specific project.

5.0 INTERFERENCES

5.1 Phthalate esters can interfere with pesticide determination; avoid any contact with plastics to best minimize this problem.

5.2 Sulfur is also an interference; this can be removed by performing sulfur clean-up on the extract. When using mercury, refer to the Pesticide Extract Sulfur Removal SOP.

6.0 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.

6.1 SPECIFIC SAFETY CONCERNS OR REQUIREMENTS

The gas chromatograph contains 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.

There are areas of high voltage in the gas chromatograph. Depending on the type of work involved, either turn the power to the instrument off, or disconnect it from its source of power.

6.2 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
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.
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.
1 – Exposure limit refers to the OSHA regulatory exposure limit.			

7.0 SAMPLE CONTAINERS, COLLECTION AND PRESERVATION

7.1 Sample Containers

. Water samples are collected in 2x1 liter amber glass containers with Teflon-coated liner and Telfon coated caps.

. Soil samples are collected in 250 or 500 mL glass containers with Teflon coated caps.

. Sample bottles are never reused.

7.2 Sample Collection

- Samples are secured against breakage in the shipping containers and kept at 4°C for transport to the laboratory. Samples should arrive at the laboratory the next day following collection.

7.3 Sample Preservation

- Samples are preserved by cooling to 4°C.

7.4 Holding Times

- Water samples must be extracted within seven days from collection.
- Soil samples must be extracted within 14 days from collection.
- Aqueous samples requiring NYSDEC ASP must be extracted within five days from VTSR.
- Soil samples requiring NYSDEC ASP should be extracted within 14days from collection.
- All extracts must be analyzed within 40 days from date of extraction (40 days from collection for NYSDEC ASP).

8.0 APPARATUS AND MATERIALS

8.1 Sample extracts are analyzed on a gas chromatograph (GC) equipped with an electron-capture detector (ECD), autosampler, data collection system and all other required accessories. The following columns are acceptable for analysis:

Reference Sect 12.1 for Instrument specifications

- Restek Rtx-CLPesticides 30 meter
  0.53mm ID 0.42um film thickness
  or equivalent
- Restek Rtx-CLPesticidesII 30meter
  0.53mm ID 0.42um film thickness
  or equivalent

- Various sizes of syringes, volumetric pipets, volumetric flasks, pipet bulbs
- 0.8 ml and 1.8 ml autosampler vials and caps.
- Borosilicate glass transfer pipets/transfer bulbs.
- Safety glasses, non-powdered polyvinyl gloves, fume hood.
- Properly cooled refrigerators each for sample and standard storage.
- Standard and Instrument Maintenance logbooks.
- Sample Injection logbooks.

9.0 REAGENTS AND STANDARD PREPARATION

9.1 **Solvents**: Hexane, Acetone, Toluene and Isooctane (2,2,4-trimethylpentane) should be pesticide grade or equivalent.

9.2 Commercially prepared stock standards can be used if they are certified and pretested by the manufacturer. See Table 10 for a listing of stock standards, manufacturer & part #'s typically used.

9.3 All stock and working standards are stored in amber screw top bottles at 4°C and replaced after 1 year or earlier if routine QC tests indicate a problem.

9.4 Calibration Stock Standard

9.4.1 Individual Stock Mix A and B

Calibration Stock Standard AB is prepared by diluting the standard mix, purchased from a commercial vendor, into isooctane. See Table 1.0 for individual compounds and concentrations. See Table 2.0-2.2 for Preparation of Mixes. When there is insufficient resolution between compounds, Mix A and B may be prepared separately to create two sets of standards of non-coeluting compounds to be run separately.

9.5 **Single-component Calibration Standards** at a minimum of 5 concentration levels are prepared through dilution of the calibration stock standard, with a lower 6th level (designated as level 0.5) used as needed for lower reporting limits. One concentration level should be near but above the method detection limit. The remaining concentrations should correspond to the linear range of the instrument. See Tables 1.0 for final component concentrations.

9.5.1 For Army Corp. Projects the lowest calibration level should be near but above the MDL and never less than three times the MDL.

9.6 **Pesticide Multi-component Standards** - A single midpoint calibration standard is prepared for Toxaphene and Technical Chlordane as required. Each of these standards are diluted from individual stocks. Each is run after a single component curve. For Multi-component Stock and Standard concentrations see Table 1.0. For Multi-componenet standard preparations see Table 2.3-2.4. If either Toxaphene or Technical chlordane is detected as a positive hit, the sample shall be run against a minimum 5 point calibration curve.

9.7 **Surrogate Standard** is prepared to monitor the performance of the extraction and analytical system. Samples, standards and blanks are spiked with pesticide surrogates. Two surrogates, 2,4,5,6-tetrachloro-m-xylene (TCX) and Decachlorobiphenyl (DCB), are spiked with the proper amount at a level of 0.2 ug/L for waters and soils are spiked at a level of 6.7 ug/Kg. (See Table 3.0).

9.8 **Instrument Breakdown Standard** is prepared as a solution of p,p'DDT and Endrin at a concentration near the midlevel concentration of the calibration standards. (See Table 5.0).

10.0 CALIBRATION

10.1 RT Windows are established by making three injections of the mid concentration standard throughout the course of a 72-hour period and calculating 3 x ± the standard deviation. Retention time windows shall be calculated for each compound on each GC column whenever a new column is installed.

10.1.1 RT windows are established as described above, however, the laboratory has established a minimum RT window of 0.03minutes.

10.2 Calibration Standards

Initial calibration standards are analyzed by injecting 1 ul of each of the levels of the single component calibration standards Mix A & Mix B, and one level of all other multi-response pesticides/PCB's

10.2.1 The initial calibration criteria is 20% RSD for calibration factors form the intial calibration standards for each target analyts. If the %RSD cannot be met using average calibration factor, an alternate curve type can be used. A Linear regression fit requires a minimum of 5 points and a Quadratic fit requires a minimum of 6 points. The criteria is 0.990 or higher for the coefficient. In determining the best fit of a curve, using either linear regression or

Quadratic, the analyst shall choose the best fit possible based on both the coefficient and the slope of the line (b). The coefficient should be greater than 0.990 and as close to 1 as possible. The slope of the line (b) should be as low a value as possible. The analyst may choose between linear regression, a weighted linear regression or a Quadratic fit. The line may be forced through zero, if the (b) value by other means would result in possible negative results.

10.3 The working calibration range of this method is defined by the initial calibration curve. All extracts with target compounds exceeding the curve should be diluted to within the upper half of the calibration range.

10.3.1 The daily calibration verification is acceptable if all of the following are true.

10.3.1.1 The breakdown of Endrin or p'p'DDT is <= 15% based on the presence of Endrin Aldehyde and Endrin Ketone for Endrin, p,p'DDD and p,p'DDE for p,p'DDT. If the breakdown does exceed 15% then corrective action must be taken prior to continuing the calibration verification.

$$\%\ \text{breakdown for p'p'DDT} = \frac{\text{DDD peak area + DDE peak area}}{\text{Total DDT peak area (DDT+DDD+DDE)}} \times 100$$

% breakdown for Endrin =

$$\frac{\text{Endrin Aldehyde peak area + Endrin Ketone peak area}}{\text{Total Endrin peak area (Endrin+Endrin Aldehyde+Endrin Ketone)}} \times 100$$

10.3.1.2 The calibration verification standard must have all compounds (+/-) 15% difference of their expected value. Because of the low concentration of pesticide standards injected on a GC/EC, column adsorption may be a problem when the GC has not been operated for a day. Therefore, the GC column should be primed by injecting a pesticide standard mixture approximately 20 times more concentrated than the midlevel standard.

If the calibration verification standard fails, then it can be rerun. A new calibration curve will be run, if warranted, due to repeated failure. When the calibration verification standard fails, all samples that were injected after the last standard that met the QC criteria must be evaluated to prevent any mis-quants and possible false negatives. Depending on the compound failures the extracts may need reinjection. More frequent analysis of standards will minimize the number of reruns for QC failures.

If the standard analyzed after a group of samples exhibits a response for an analyte that is above the acceptance limit, i.e. >15%, and the analyte was not detected in the specific sample analyzed during the sequence, then the sample extracts do not need to be reanalyzed. If the compound was detected in the sample and the sample bracket is 10 or

less, the chromatography is reviewed by the department supervisor for the possibility of sample matrix. No further action may be taken at this point.

10.3.1.3 An instrument blank standard is run after the calibration verifications standards and prior to any samples to ensure the instrument and its autosampler are clean.

10.3.2 All initial calibrations must be verified with a standard obtained from a second source manufacturer or lot. The same manufacturer may be used, if the lot can be demonstrated from the manufacturer as prepared independently from other lots. Traceability shall be to a national standard when commercially available. The % difference of this standard should pass within +/- 25% from the initial calibration curve. If the second source standard fails to meet criteria, check the preparation of both the initial curve and the second source standard for errors. It may be necessary to re-prepare a solution to check.

10.4 When analyzing samples, any extract that contains a target compound that exceeds the high level calibration must be diluted to within the calibration range. Multi-component targets must also be diluted so that the largest peak in the multi-component does not exceed the response of the largest peak in the high-level single-component calibration standard.

10.5 Calibration Verification - (Each 12 hour shift)

10.5.1 A single mid-level standard (standard mix or multi-compound), is analyzed at a minimum of every 20 samples and at the end of the analysis sequence. If the samples are suspected to cause breakdown and column degradation the calibration check will be analyzed every 10 samples. The calibration factor for each compound to be quantitated, must not exceed a 15 percent difference when compared to the average calibration factor from the calibration curve. When this criteria is exceeded, inspect the GC system to determine the cause and perform whatever maintenance is required before recalibrating and proceeding with sample analysis. All samples that were injected prior to the standard exceeding the criteria must be reinjected if the initial analysis indicated the presence of the specific target analytes that exceeded the criteria.

10.5.2 For Army Corp. Projects a mid-level calibration verification must be analyzed every 10 samples and at the end of the analysis sequence.

10.6 Calculations for water samples:

10.6.1 The calculation for quantitation of single-component target analytes is as follows:

Single Component Calculation

$$ug/L = \frac{\text{Area of sample peak} \times \text{Final Volume of extract(uL)} \times \text{Dilution Factor}}{}$$

Avg Calibration Factor of standard x Sample Volume extracted (mLs) x ul inj.

CF = Peak Area of the Standard
Mass Injected (ng)

Water sample by Linear regression:

b + (Area) m1 = on column amount (ng)

(cn column amount-ng) (final volume [uL])(dilution factor) = ug/L
(sample volume [mLs])(ul inj.)

Water sample by Quadratic:

b + m1(Area) + (m2)*(Area)^2 = on column amount (ng)

(cn column amount-ng) (final volume [uL])(dilution factor) = ug/L
(sample volume [mLs])(ul inj.)

10.6.2 An average calibration factor is calculated for each compound and surrogate from the initial calibration. For multi-component analytes a calibraton factor is calulated for the areas of each peak (using 3 -5). The final concentration is then calculated using the average of the final result for each of the 3 - 5 peaks.

The peaks chosen for quantitation should be major peaks representative of the multicomponent standard. The areas of each peak should be evaluated in relation to each other and any disproportionate peaks should not be used.

Multicomponent Calculation

ug/L = (X) Final Volume of extract (uL) x Dilution Factor
(Y) Sample Volume extracted (mLs) x ul inj.

X = The area of one of the 3-5 peaks from sample.
Y = The average CF for the corresponding peak from standard.

A Cf must be calculated for 3-5 peaks. The final concentration reported is calculated using the average of the final result for each of the 3-5 peaks.

10.7 Calculations for soil and oil samples:

10.7.1 The calculation for quantitation of single-component target analytes is as follows:

Single Component Calculation

ug/Kg = Area of sample peak x Final Volume of Extract (uL) x Dilution Factor
Avg. Calibration Factor of Std. x Sample Volume Extracted(g) x Decimal % Solids x ul inj.

CF = Peak Area of the Standard
Mass Injected (ng)

10.7.2 An average calibration factor is calculated for each compound and surrogate from the initial calibration. For multi-component analytes a calibraton factor is calulated for the areas of each peak (using 3 -5). The final concentration is then calculated using the average of the final result for each of the 3 - 5 peaks.

The peaks chosen for quantitation should be major peaks representative of the multicomponent standard. The areas of each peak should be evaluated in relation to each other and any disproportionate peaks should not be used.

Multicomponent Calculation

ug/Kg = (X) Final Volume of Extract (uL) x Dilution Factor
(Y) Sample Volume Extracted(g) x Decimal % Solids x ul inj.

X = The area of one of the 3-5 peaks from sample.
Y = The average CF for the corresponding peak from standard.

A Cf must be calculated for 3-5 peaks. The final concentration reported is calculated using the average of the final result for each of the 3-5 peaks.

Note: Oils will not have decimal percent solids.

10.7.3 When more than one multicomponent is detected in a sample, non-overlapping peaks are chosen for quantitation. If it is not possible to choose non-overlapping peaks, peaks with the least amount of overlap are chosen.

10.8 Corrective Action for Initial Calibration

10.8.1 If the technical acceptance criteria for the initial calibration are not met, inspect the system for problems. It may be necessary to change the column, bake out the detector, clean the injection port, or take other corrective actions to achieve the acceptance criteria.

10.8.2 Corrective Action for Calibration Verification

10.8.3 If the technical acceptance criteria for the calibration verification are not met, inspect the system for problems and take corrective actions to achieve the criteria.

10.8.3 Major corrective actions such as replacing the column or detector will required a new

initial calibration.

10.8.4 Minor corrective actions may not require a new initial calibration provided a calibration check meets all acceptance criteria.

11.0 QUALITY CONTROL

11.1 Demonstration of Analyst Capability-Spike four sets of reagent using the Pesticide QC solution. Process the samples through the whole analytical procedure.

11.1.1 Calculate the average recovery (x) and the standard deviation (s) for each analyte from the four results. Compare the s and x with the criteria generated from the laboratory control charts for each compound for each matrix. The limits are derived from laboratory-generated data, and are updated as needed. If all analytes meet the acceptance criteria, analysis of samples can begin. If any analyte fails, the cause for the failure must be determined and the test must be repeated for that analyte.

11.1.2 The demonstration of analyst capability will be verified on an annual basis.

11.2 Method detection limits (MDL's) for this method will be verified on an annual basis as detailed in the latest version of the corporate SOP on MDL's. The MDL check sample is an extracted sample containing each target analyte at a concentration close to the MDL. Each analyte must be detected in order for the instrument to be considered capable of reporting estimated result to the calculated MDL. If an analyte cannot be detected at the given concentration, instrument maintenance should be performed and the MDL check sample re-analyzed. If the compound is still not detected, a new MDL should be prepared and analyzed at a higher concentration. The new MDL should be used in reporting results.

11.3 External PT samples are randomly submitted by the QC officer and are processed as any other client's samples would be.

11.4 Refer to Table 6.0 for Practical Quantitation Limits (PQL's) for all compounds.

11.5 Matrix spike (MS), matrix spike duplicates (MSDs) and matrix spike blanks (MSB's), if applicable, are analyzed within every set of 20 samples or less. Recoveries must be within the laboratory generated control limits. If these criteria are not met, but the blank spike data meet all of the recovery criteria, then the MS/MSD are documented as having matrix interferences. If the blank spike fails, check for instrument and/or column related problems and reanalyze the spikes. If the problem is corrected, the samples are reanalyzed.

11.6 A QC check sample is performed at a frequency of one per 20 samples extracted of similar

matrix. See Table 7.0 for spike compounds. Control charts are used to establish laboratory generated control limits.

11.7 Blanks

11.7.1 Method Blank

Method blanks are spiked with surrogates, extracted, and analyzed following the same procedure that is used with the associated samples. A water method blank is one liter of reagent water and a soil method blank is 30 grams of sodium sulfate for sonication and 15 grams of sodium sulfate for automated soxtherm extraction.

Frequency - method blanks must be analyzed with each case, 20 samples of a similar matrix, or whenever samples are extracted by the same procedure, whichever is more frequent.

Acceptance criteria - method blanks must contain less than half the PQL for all the target compounds listed in Table 1.0 & 1.1.

All samples associated with an unacceptable method blank must be reextracted and reanalyzed.

11.8 Surrogates

11.8.1 The surrogates TCX and DCB are added to each standard, sample, blank and QC prior to extraction.

11.8.2 The QC limits for surrogate recovery are listed in Table 3.1 and pertain to all samples, blanks and spikes.

11.9 Analytical QC Samples

Daily Calibration Check Sample	One per 12 hour shift minimum
Breakdown Standard	One per 12 hour shift minimum
Instrument Blank	One per 12 hour shift minimum

*It may be necessary to analyze a solvent blank after high concentration samples.

Preparation QC Samples

MS/MSD	One pair every 20 samples minimum
QC Check	Every extraction batch
Surrogates	Every sample and standard
Method Blank	One per batch

11.10 Analytical Documentation Procedures

11.10.1 Instrument Batches

An instrument batch is created for each analytical sequence to organize all the associated data. Batch designations are of the format:

XXnnn

where: XX = instrument identifier
nn = number of batch

(i.e. C5001)

Instrument batches are number sequentially so a unique batch identifier identifies each analytical sequence. The batch consists of a file folder with all the associated QC information for the analytical sequence. The raw data is then scanned for all initial and continuing calibrations.

11.10.2 Data Archiving

All data files are archived on a daily basis using a 12.0gb data storage cartridge. The associated method files are also archived daily to provide an accurate historical record. Care shall be exercised when purging data off the hard drives to ensure that all data being purged has been archived.

11.10.3 Instrument Run Logs

It is the company's policy that all measurement data be recorded in logbooks or on preprinted log sheets in permanent ink. Run logs are created from the Target data system by generating a file which contains a sequential list of all files analyzed. The record shall reflect the measurement performed and all appropriate details for conclusions related to the measurement. The record shall be signed and dated by the individual performing the measurement on the day the measurement is performed. Corrections shall be made by drawing a single line through the error, and initialing and dating the correction. A secondary authorization of the logbook is required and shall be performed by the department's manager or designee.

Each instrument has its own set of bound run logs (see Figure 1.0) which are sequentially numbered and paginated. Run logs are filed in the laboratory once they have been filled, for future reference. Each analytical sequence shall be started on a new page of the log and continued on the next page, if necessary. The header information designating the standard

codes used shall be completed for each sequence. All standards used are recorded in this field for future traceability. The data file, sample number, dilution factor, analyst's signature, and date are recorded.

11.10.4 Corrective Action Reports

A corrective action report (CAR) is initiated when a problem is encountered during analysis, data reduction or deliverables preparation, data validation, or when any deviations from this SOP occur. The CAR is initiated by the analyst or dept. manager first identifying the problem through the NCM module in the LIMS system. It is then electronically forwarded to all appropriate departments, QA officer, and Lab Manager. Reference SOP for correction action reports.

11.10.5 Chain of Custody Record

When samples are removed from storage for preparation or analysis they must be signed out utilizing the chain of custody record (COC). The samples shall then be signed back in on the COC upon their return to storage or designated "used" if the sample volume is consumed during the preparation or analysis.

11.10.6 Sample Tracking Record

Notification of sample arrival is done by the Sample Control department by issuing a preliminary notification sheet. Samples are tracked for extraction and analysis by using the laboratory's LIMS system.

11.11 Quality Control Check Points

11.11.1 Analysis quality control approval report

Specific quality control checkpoints have been established for the analysis of sample extracts. The specific check points in the analysis logbook, are initialed and dated by the analyst to ensure the consistency and accuracy of the data produced.

11.11.2 Specific quality control checkpoints have been established for the preparation of data deliverables, which are monitored through a Lims Organics Data Review Checklist – Doc # QAF04300.CT. The specific check points must be reviewed by the analyst 1st level reviewing and the secondary reviewer 2nd level revieweing the data to ensure the consistency and accuracy of the data produced. Refer to Figure 3.0 for the document and specific control points covered.

12.0 SAMPLE PREPARATION AND INSTRUMENTAL PROCEDURES

12.1 HP6890 GC's with dual micro ECD's and CTC (Leap Technologies) Autosampler is currently being used to run Pesticides/PCBs under this SOP. Instruments, columns and conditions following are guidance:

a) Column type: 30 meter Rtx-CLPesticides (See Section 7.1)
Column flow: ~ 5 mL/min Hydrogen or Helium
Detector Make-up Flow: 60° mL/min N_2
Injector Temperature: 225 C
Detector Temperature: 330°C
Temperature Programming:
Initial Temperature: 110°C Hold 1.0 min
Initial Ramp: 20°/min to 245, 0min Hold
Second Ramp: 6/min to 310, no hold

b) Column type: 30meter Rtx-CLPesticides II (See Section 7.1)
Oven and temperatures same as above

Instrument and column conditions may have equivalent programming as long as all method QC requirements are met.

12.2 **Aqueous Sample Preparation for Pesticides:**

12.2.1 Brief Summary:

A 1 liter sample aliquot is spiked with the surrogate and extracted with methylene chloride at a pH between 5 and 9. Using either the separatory funnel extraction method or the continuous liquid-liquid extraction method is acceptable. The methylene chloride extract is dried and solvent exchanged to hexane and adjusted to a final volume of 10 mL. The sample extraction must be completed within 7 days of sample collection. Samples requiring NYSDEC ASP must be completed within seven days of VTSR.

A 1 uL aliquot of the sample extract is injected into the gas chromatograph (GC). The GC separates the compounds. Compound identification is performed by the comparison of GC retention times to those of known analytical standards. Quantitative analysis is performed by the comparison of compound peak height or peak areas to those of an analytical standard at a known concentration.

12.2.2 For TCLP samples, 800 mLs of reagent water is added to 200 mLs of the TCLP leached sample and the procedure would follow Section 11.2.1. See Table 9 for TCLP PQL's and target compounds.

12.2.3 Sample Extraction

Refer to Standard Operating Procedure for Method 3510C for the water extraction procedure.

12.3 Soil Sample Preparation for Pesticides

12.3.1 Brief Summary

A 30 g sample aliquot is spiked with surrogate and extracted with a 1:1 mixture of hexane/ acetone using the sonication extraction method. The hexane /acetone extract is dried and concentrated to a final volume of 10 mL. The sample extraction must be completed within 14 days of sample collection. A 1uL aliquot of the sample extract is injected into the gas chromatograph (GC). The GC separates the compounds. Compound identification is performed by the comparison of GC retention times to those of known analytical standards. Quantitative analysis is performed by the comparison of compound peak height or peak areas to those of an analytical standard at a known concentration.

12.3.2 A 15 g sample aliquot is spiked with surrogate and extracted with a 1:1 mixture of hexane/ acetone using the soxtherm apparatus. The hexane /acetone extract is dried and concentrated to a final volume of 5 mL. The sample extraction must be completed within 14 days of sample collection. A 1uL aliquot of the sample extract is injected into the gas chromatograph (GC). The GC separates the compounds. Compound identification is performed by the comparison of GC retention times to those of known analytical standards. Quantitative analysis is performed by the comparison of compound peak height or peak areas to those of an analytical standard at a known concentration.

12.3.3 Sample Extraction

Refer to the Standard Operating Procedure for method 3550B and 3541 soil extraction procedures.

12.4 Oil Sample Preparation for Pesticides

12.4.1 Brief Summary

A 1 gm sample aliquot is spiked with surrogate and brought to a final volume of 10 mLs in hexane or other appropriate solvent.

12.4.2 If the sample is a TCLP oil, a 1 mL sample aliquot is spiked with surrogate and brought to a final volume of 10 mLs in hexane or other appropriate solvent.

12.5 Sample Analysis Procedures

12.5.1 Sample Extract Analysis

Sample extracts are removed from storage in the G.C. instrument room and are signed out on the extract chain of custody form. All sample extracts are signed back in after they are returned to storage.

Make sure all instrumental operating conditions are correctly set.

In a vial with a 200 ul insert, load a 200 ul aliquot of sample extract. A 1.0 ul injection of the sample extract onto the GC column is made with an autosampler and then the GC temperature program sequence is started.

This method is intended to achieve the quantitation limits whenever possible. If sample chromatograms have interfering peaks, high baseline, or off-scale peaks, then those samples must be reanalyzed following dilution, cleanup, or reextraction. No limit is placed on the number of reextractions of samples that may be required because of contaminated method blanks.
The sample must be analyzed at the most concentrated level that is consistent with achieving satisfactory chromatography (defined in Section 12.7).

No target analyte concentrations may exceed the upper limit of the initial calibration.

12.6 Qualitative Analysis

12.6.1 Target Compounds

The identification of single component pesticides is based primarily on retention time data. The RT of a peak can be verified only from an on-scale chromatogram.

If a compound falls within the R.T. windows of the compound in the calibration curve, and is greater than the MDL, that sample would require confirmation by analysis on a second column.

12.6.2 If the same compound falls within the R.T. window of the compound on the column used for confirmation, the compound is determined to be present and therefore reported as a target compound. This second column should be of a dissimilar phase from the first column, see section 8.1.

12.7 Quantitative Analysis

12.7.1 Target Compounds

Target compounds are quantitated by the external standard technique using peak area and

the calibration factor determined during the initial calibration sequence.

A second column confirmation analysis is performed on all samples for analytes with concentrations greater than the MDL.

When compound concentrations are below the PQL, but the compound meets identification criteria, report the concentration with a "J" qualifier.

When a compound exceeds the linear working range of the initial calibration, the sample must be diluted to bring the analyte concentration within the calibration range.

The following are guidelines on performing dilutions and exceptions to this requirement:

. If the response is still above the high calibration point after the dilution of 1:100,000, the laboratory shall contact the client.

. Use the results of the original analysis to determine the approximate dilution factor required to get the largest analyte peak within the initial calibration range.

12.7.2 The lab routinely reports the higher concentration of the results between the two columns used for analysis. If there is a greater than 40% difference between the two columns, the lower result is reported.

12.8 Instrument Maintenance

12.8.1 Preventative Maintenance

All instrumentation is covered by a service contract with an external instrumentation service vendor, or by personnel trained in preventative maintenance. All instrument preventative maintenance is performed according the manufacturers recommended procedures, by trained personnel. All preventative maintenance shall be thoroughly documented in the maintenance log (see Figure 4.0), as to a description of the maintenance performed, the date performed, and the personnel performing the maintenance.

12.8.2 Corrective Maintenance Determinants and Procedures

Corrective maintenance is deemed necessary when the analytical system, after reanalysis, cannot meet calibration, resolution, chromatography, breakdown, or other protocol specific QC criteria. Corrective maintenance may include, but is not limited to, decontamination of the system, injection port cleaning, column cutting or replacement, syringe cleaning or replacement, or detector baking out or replacement. All corrective maintenance is performed according the manufacturers recommended procedures, by trained personnel. All corrective maintenance shall be thoroughly documented in the maintenance log, as to a

description of the maintenance performed, the date performed, and the personnel performing the maintenance.

12.8.3 Maintenance Authorization

The department's manager, or designee authorizes all preventative and corrective maintenance. When maintenance is deemed necessary, a service call is placed for all equipment covered under a service contract, by the department's manager, or designee.

12.9 Data System

12.9.1 Data Acquisition and System Operation

Data is acquired from sample analyses using Chemstation software. Analytical batches are set up with all the associated sample ID, dilution, and data file information. Raw data files are manually copied to the Target data system for integration and quantitation. Turbochrom has instrument control.

12.9.2 Instrument Errors

System errors are logged to the system console at time of occurrence. The system manager shall be responsible for checking and providing corrective actions for all system errors.

12.9.3 Manual Integration and Editing Flags

Manual integrations are required when the automated software doesn't correctly integrate extracted ion current profiles (EICP). A user shall be logged into the Target system as their own name. This name will signify who performed the manual integration. To perform a manual integration, the target compound of interest is selected and the EICPs are graphically presented. The peak can then be correctly integrated. A reason code shall be selected for the type of integration performed by selecting Review codes from the menu options.

UN = Unidentified peak based on spectra or concentration
ID = Identified a peak based on spectra
INT = Integrated a peak due to incorrect integration

A new quantitation report is produced. The manually integrated data file is the saved by exiting and saving from file edit. Manual integrations are flagged by the data system with the "M" qualifier beside any manually integrated area. The manually integrated data file is the saved by exiting from Target review. Manual integrations are flagged by the data system with the "M" qualifier beside any manually integrated area on the hardcopy quant report. The analyst name will appear on the electronic manual

integration report which is uploading to the LIMS system when data files are uploaded. A hardcopy print out of the EICP of the quant ion displaying the manual integration shall be produced for the before and after integrations and is included in the raw data to the clients when the report is generated during final packaging.

13.0 CALCULATIONS

See Sections 10.5.1, 10.5.2, 9.6.1 and 10.6.2 of this document.

13.1 Calculation of Calibration Factor

$$CF = \frac{\text{Peak Area of the Standard}}{\text{Mass Injected (ng)}}$$

13.1.1 Calculation of Percent Difference

The following formula is used to calculate % difference in the calculated versus expected values of standards.

$$\% \text{ Difference} = \frac{\text{Calculated conc. - expected conc.}}{\text{Expected conc.}} \times 100$$

13.1.2 Calculation of Surrogate, Spike and QC Check Recoveries

The following calculation is used for spiked sample recoveries.

$$\% \text{ Recoveries} = \frac{\text{Amount Recovered - Amount in Sample}}{\text{Amount Added}} \times 100\%$$

13.1.3 Percent Relative Standard Deviation (%RSD)

$$\%RSD = \frac{\text{Standard Deviation}}{\text{Average CF}} \times 100$$

13.1.4 Percent Moisture

$$\% \text{ Moisture} = \frac{\text{g of Wet Sample - g of Dry Sample}}{\text{g of Wet Sample}} \times 100$$

13.1.5 Adjusted Estimated Practical Quantitation Limit for Samples

Adjusted Estimated PQL = $\frac{(PQL) \times Df}{D}$

where:

D = $\frac{100 - \%\ Moisture}{100}$

DF = Dilution Factor

14.0 ACCEPTANCE OF DATA

14.1 Daily Calibration Check Standard (Required every 12 hour shift minimum)

Verification of the calibration curve with a single midlevel standard mix or multi-component calibration standard is obtained if the calculated concentration of all the compounds to be quantitated are (+/-) 15% of the expected value.

14.2 Breakdown Check (Required every 12 hour shift minimum)

Instrument Breakdown of DDT and Endrin is considered under control if the % Breakdown of each analyte is <= 15%.

14.3 Instrument Blank

The instrument blank is used to verify that the analytical system is free of contaminants. The instrument blank shall be free of any target compounds above half the quantitation limits and shall not contain any unusual interference. The instrument blank contains the same surrogates that are in all samples and standards.

14.4 Method Blanks

Method blanks are extracted with every batch of up to 20 samples to ensure that there is no contamination from the extraction process. The method blanks shall be free of any target compounds half of the quantitation limits and shall not contain any unusual interferences.

14.5 Matrix Spikes and Matrix Spike Duplicates

Matrix spikes and matrix spike duplicates are extracted with every batch of up to 20 samples to verify extraction efficiencies. Acceptance criteria are listed in Table 8.0.

14.5.1 MSB's are extracted with every batch of 20 samples as applicable to client requested protocol. Acceptance criteria are listed in Table 8.0

14.5.2 QC reference samples are extracted with every extraction batch of up to 20 samples to verify extraction efficiencies. Acceptance criteria are listed in Table 7.0.

15.0 REPORTING OF RESULTS

15.1 All results are reported to two significant figures. Water samples are reported in ug/L, soil samples are reported in ug/Kg dry weight and waste samples are reported in ug/Kg. Check reporting deliverables required from the traveller. All job packages require a case narrative and quality control approval report. The case narrative should outline in detail any problems with client samples during analysis. The following indicates the different levels of reporting.

Level I

- Case Narrative
- Sample Results

Level II

- Case Narrative
- Sample results
- Surrogate Recovery forms
- LCS & MS/MSD recovery forms

Level III/NJ
-Everything listed below, *Except* standard scans and area reports

CLP/NYSDEC

- Case Narrative
- Form 1 (Organic Analysis Data Sheet)
- Surrogate Recovery forms
- LCS & MS/MSD recovery forms
- MSB recovery form as applicable)
- Form 4C (Method Blank Summary)
- Initial Calibration Forms
- Analytical Sequence Form
- Breakdown Check Form
- Continuing Calibration Forms
- Form 10 (Pesticide/PCB Identification)
- Sample and Standard Scans and Area Reports
- Standard Concentration Summary

- GC/MS Confirmation (if applicable)

16.0 SUPPLEMENTAL DOCUMENTS

16.1 SOP for Pesticide Extract Sulfur Removal.

16.2 SOP for Pesticides in Water Extraction by Method 3510C.

16.3 SOP for Pesticides in Soil Extraction by Method 3550B.

16.4 Tables attached include the following:

Table 1.0 and 1.1 - Single-Component Calibration Concentrations
Table 2.0 - Multi-Component Concentration
Table 3.0 - Surrogate Mix
Table 3.1 - Surrogate Recovery Limits
Table 4.0 - Pesticide QC Check and Matrix Spike
Table 5.0 - Breakdown Standard Concentration
Table 6.0 - Practical Quantitation Limits (PQL)
Table 7.0 - QC Check Recovery Criteria
Table 8.0 - MS/MSD/MSB Recovery Criteria
Table 9.0 - TCLP Compounds and PQL's
Table 10 – Stock stds – Manufactuer/Part #

17.0 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). Employees must abide by the policies in Section 13 of the Corporate Safety Manual for "Waste Management and Pollution Prevention."

17.1 Pollution prevention is the reduction and/or elimination of wastes or the toxicity of wastes at the point of generation. The laboratory strives to keep all wastes at a minimum through a variety of techniques. These include the following.

17.1.1 Inventory control: Rotate stocks (first in, first out) and only purchase reasonable amounts of chemicals. Do not purchase excessive quantities, which will be discarded as wastes at some future point.

17.1.2 Material Handling and Storage: Keep containers properly labeled. Keep solvents covered. Store and handle all chemicals, reagents, standards, etc. to prevent contamination and

degradation.

17.1.3 Personnel: All staff must be trained in the proper handling and disposal of waste.

17.1.4 Waste Reduction: Reduce the volume of waste generated wherever possible.

17.1.5 Chemical/material substitution: Use less toxic chemicals whenever possible. Avoid the use of chromic acid (Chromerge) solutions.

18.0 WASTE MANAGEMENT & POLLUTION PREVENTION

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 Tracking anc Collection of Hazardous Waste SOP. The following waste streams are produced when this method is carried out.

18.1 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**.**

18.2 Personnel who handle or generate waste must be trained within six months of employment in proper waste handling and requirements.

18.3 Autosampler vials containing pesticides only shall be disposed of in the 5 gallon bucket labeled for Hexane waste.

19.0 REFERENCES

19.1 "Method 8000B - Determinative Chromatographic Separations", EPA SW846, 3rd Edition.

19.2 "Method 8081A - Organochlorine Pesticides by Gas Chromatography, SW846, 3rd Edition.

19.3 "Methods of Organic Chemical Analysis of Municipal and Industrial Wastewater", Federal Register Vol. 49, No. 209, October 26, 1984.

19.4 "USEPA CLP OLM03.2 Statement of Work" - pg.D-53/Pest, Section 10.1.8.2, Florisil Cleanup.

20.0 SUBSTANTIVE REVISIONS

20.1 Changed name of laboratory from AEN to STL; Feb. 8, 1999.

20.2 Revised section 8.3 changing standards hold time, revised section 9.6.2changing the calculation fo multiresponse pesticides. Revised section 9.7.3 to include reference to the Added Appendix A, revised section 10.4 changing the method blank criteria, revised section 13.5.2 to reference Table 7.0 for acceptance criteria; 03/05/99.

20.3 Added Terms and Definitions section, Pollution Prevention, and Waste Management; 2/15/2000. Added Initial calibration %RSD criteria and linear regression criteria. Corrected Mix 5 concentrations in Table 1.1 for alpha and gamma Chlordane.

20.4 Added to the Safety Section.

20.5 Added to the Safety section and Waste Management sections. Added EH&S Officer to Approval section. Removed Appendix A (multi-peak identification). Section 11.4.1 Added soxtherm blank information. Section 12.1 added autosampler. Fixed Isodrin and Chlorobenzilate reporting limits in Table 6.0. Changed Tables 3.1 and 7.0 to refer to LIM system for updated control limits. Section 12.3.2 added Soxtherm brief summary. Changed Toxaphene reporting limits in Table6.0-1/14/2004.

20.5 May 7, 2004- Added 40% Rule for reporting of results to Section 12.7.

20.6 January 14,2005-Added 4.3.2 Section concerning QC check solutions for Toxaphene and Technical Chlordane.

20.7 January 14, 2005 –Added 9.5.1 Section. Army Corp. requirements for low point calibration standard.

20.8 January 14, 2005-Added 9.6.1 Section. Army Corp. requirements for 3 levels of calibration for Multicomponent Pesticides.

20.9 January 14, 2005-Minutes added to 0.05 for retention time criteria in section 10.1.1.

20.10 January 14, 2005- Section 10.2.1 Added to calibration criteria further details.

20.11 January 14, 2005- Section 10.6.1 Linear regression calculation wording fixed.

20.12 January 14, 2005- Sections 11.6, 14.1, 14.2, and 14.3 Minor changes to wording.

20.13 January 14, 2005-Section 13.1.1 Absolute sign removed from calculation.

20.14 February 15, 2005- Sections 10.6.2 and 10.7.2 Guidance for Selecting Multi-component peaks.

20.15 February 15, 2005-Section 12.6.2 added.

20.16 February 15, 2005-Section 12.7.1 Clarified second column confirmation procedures.

20.17 February 15, 2005-Added to Section 11, Initial Demonstration and MDL criteria.

20.18 February 23, 2005 –Section 13.1.5, Changed naming to Adjusted Estimated PQL.

20.19 March 29, 2005 - Section 12.6.1, Changed PQL to MDL.

20.20 March 29, 2005 – Section 10.5.1 modified to analyze a CCV per 10 samples of poor matrix (not per 20). Section 10.5.2 Added for ACOE requirement of CCV per 10 samples.

20.21 April 19, 2005 – Sect 12.7.1 the Following statement was removed, “Data for more than two analyses shall not be submitted”.

20.22 April 19, 2005 – Sect 19, added 8000C SW846 reference.

20.23 April 19, 2005 – Sect 11, clarified requirements for analysis of PT samples and also the use of control charts to establish in-house Control charts.

20.24 May 15, 2007 – Section 8.1 – Updated columns to those currently used. Sect. 9.3 Changed from 6months to 1 year. Sect. 9.4.1 – changed to one mix prepared with possibility of needing two in some circumstances. Section 9.5 – added lowest 6^{th} level of calibration curve as needed. Sect. 9.8 – Removed. Done in extraction dept. Sect. 10.2.1 – Coefficient is 0.990 or higher. Sect 10.3.1.2 – Reworded end ccal failures. Sect 11 – Renumbered entire section. Sect 11.10.1 – files now scanned, not filed. Sect. 11.9.2 removed filing system section. 11.10.3 – changed from Turbochrom to Target for runlogs. Sect 11.10.4 – changed CAR procedure to electronic. Sect 11.10.6 – Removed reference to Labnet. Sect 11.11.2 – Changed from QCAR to Lims checklist for $1^{st}/2^{nd}$ level review. Sect 12.1 – updated to 6890’s, columns, temperatures and ramps. Sect 12.9.1 changed to Chemstation and manually copied. Sect 12.9.3 – updated manual integration signing. Table 1.0 replaced with updated concentrations and levels.

20.24 9/12/2007 – Removed Sect 5.3 & 5.4 on cleanups. Sect 6.2 – footnote 1 removed. Sect 7.1 – added caps. Sect 8.1 – referenced 12.1. Added Tables 2.0-2.4 for preparation of stds. Sect 10.2.1 – added curve criteria for Linear regr. & quadratic. Sect 12.3.1 & 12.3.2 –

removed methylene chloride for soils. Sect 12.6.2 – added ref to 8.1. Added sect 10.3.2 for second source analysis. Sect 10.1.1 – lowered min. RT window. Sect 11.10.4 – updated process.

20.25 Revised 11/25/08 – Removed ACOE references. Section 9.6 – Added 5 levels to multi-component req't. Sections Updated for Safety, pollution control and waste management. Table 4.0 and Table 6.0 updated. Added Table 10 – Stock stds/Mfgr.

Table 1.0
Compound Concentrations in Curve levels ug/ml

Pesticides	Level 0.5	Level 1	Level 2	Level 3	Level 4	Level 5
alpha-BHC	0.0025	0.005	0.01	0.025	0.05	0.10
beta-BHC	0.0025	0.005	0.01	0.025	0.05	0.10
delta-BHC	0.0025	0.005	0.01	0.025	0.05	0.10
gamma-BHC	0.0025	0.005	0.01	0.025	0.05	0.10
Heptachlor	0.0025	0.005	0.01	0.025	0.05	0.10
Aldrin	0.0025	0.005	0.01	0.025	0.05	0.10
Heptachlor Epoxide	0.0025	0.005	0.01	0.025	0.05	0.10
Endosulfan I	0.0025	0.005	0.01	0.025	0.05	0.10
Dieldrin	0.005	0.01	0.02	0.05	0.10	0.20
4,4'-DDE	0.005	0.01	0.02	0.05	0.10	0.20
Endrin	0.005	0.01	0.02	0.05	0.10	0.20
Endosulfan II	0.005	0.01	0.02	0.05	0.10	0.20
4,4'-DDD	0.005	0.01	0.02	0.05	0.10	0.20
Endosulfan Sulfate	0.005	0.01	0.02	0.05	0.10	0.20
4,4'-DDT	0.005	0.01	0.02	0.05	0.10	0.20
Methoxychlor	0.025	0.05	0.10	0.25	0.50	1.00
Endrin Aldehyde	0.005	0.01	0.02	0.05	0.10	0.20
Endrin Ketone	0.005	0.01	0.02	0.05	0.10	0.20
alpha-Chlordane	0.0025	0.005	0.01	0.025	0.05	0.10
gamma-Chlordane	0.0025	0.005	0.01	0.025	0.05	0.10
Toxaphene		0.20	0.50	1.00	2.00	4.00
Technical Chlordane		0.05	0.10	0.20	0.40	0.80
Tetrachloro-m-xylene (surrogate)	0.0025	0.005	0.01	0.025	0.05	0.10
Decachlorobiphenyl (surrogate)	0.005	0.01	0.02	0.05	0.10	0.20
* Isodrin	0.0025	0.005	0.01	0.025	0.05	0.10
* Chlorobenzilate	0.025	0.05	0.10	0.25	0.50	1.00
Mirex	0.0025	0.005	0.01	0.025	0.05	0.10
Alachlor	0.025	0.05	0.10	0.25	0.50	1.00

* Appendix 9 compounds

Table 2.0
Preparation of Pest Mix A/B

Pest Mix A/B

Prepared in
Iso-octane

Pest Mix A/B - Interm (level 5)	Initial Conc ug/ml	Amount Used ml	Final Volume mls	Final Conc. ug/ml	GINDABINT_
Mix A & B (8/16/80) (#32292)	8	2.5	200	0.10	
TCX	200	0.1	200	0.10	
DCB	200	0.2	200	0.20	

Pest Mix AB - Level 0.5	Initial Conc.	Amount Used	Final Volume	Final Conc.	made as needed
Pest A/B -Interm. LVL5	0.01	0.1	0.2	0.0025	
TCX				0.0025	
DCB				0.0050	

Pest Mix AB - Level 1	Initial Conc.	Amount Used	Final Volume	Final Conc.	GINDABWRK1_
Pest A/B -Interm. LVL5	0.10	2.5	50	0.005	
TCX				0.005	
DCB				0.010	

Pest Mix AB - Level 2	Initial Conc.	Amount Used	Final Volume	Final Conc.	GINDABWRK2_
Pest A/B -Interm. LVL5	0.10	5	50	0.010	
TCX				0.010	
DCB				0.020	

Pest Mix AB - Level 3	Initial Conc.	Amount Used	Final Volume	Final Conc.	GINDABWRK3_
Pest A/B -Interm. LVL5	0.10	50	200	0.025	
TCX				0.025	
DCB				0.050	

Pest Mix AB - Level 4	Initial Conc.	Amount Used	Final Volume	Final Conc.	GINDABWRK4_
Pest A/B -Interm. LVL5	0.10	25	50	0.050	
TCX				0.005	
DCB				0.010	

Table 2.1
Preparation of Miscelleanous compounds

AP9 Compounds

Prepare in Iso-octane

AP9 - Interm (level 5)	Initial Conc ug/ml	Amount Used **ul**	Final Volume mls	Final Conc. ug/ml	GINDABINT_
Isodrin	100	50	50	0.10	
Chlorobenzilate	1000	50	50	1.00	
Mirex	1000	5	50	0.10	

AP9 - Level 0.5	Initial Conc.	Amount Used	Final Volume	Final Conc.	
AP9 - Interm (level 5)	0.10	250	10	0.0025	Isodrin
	1.00			0.0250	Chlorobenzilate
	0.10			0.0025	Mirex

AP9 - Level 1	Initial Conc.	Amount Used	Final Volume	Final Conc.	GINDABWRK1_
AP9 - Interm (level 5)	0.10	500	10	0.0050	Isodrin
	1.00			0.0500	Chlorobenzilate
	0.10			0.0050	Mirex

AP9 - Level 2	Initial Conc.	Amount Used	Final Volume	Final Conc.	GINDABWRK2_
AP9 - Interm (level 5)	0.10	1000	10	0.0100	Isodrin
	1.00			0.1000	Chlorobenzilate
	0.10			0.0100	Mirex

AP9 - Level 3	Initial Conc.	Amount Used	Final Volume	Final Conc.	GINDABWRK3_
AP9 - Interm (level 5)	0.10	2500	10	0.0250	Isodrin
	1.00			0.2500	Chlorobenzilate
	0.10			0.0250	Mirex

AP9 - Level 4	Initial Conc.	Amount Used	Final Volume	Final Conc.	GINDABWRK4_
AP9 - Interm (level 5)	0.10	5000	10	0.0500	Isodrin
	1.00			0.5000	Chlorobenzilate
	0.10			0.0500	Mirex

Table 2.2
Preparation of Alachlor

Alachlor Curve

Prepared in Iso-Octane

Alachlor - (Intermediate)	Initial Conc ug/ml	Amount Used ml	Final Volume mls	Final Conc. ug/ml	GAlacINT_
Alachlor (Restek-32204)	1000	0.1	10	10	G_ALACHLOR_

Alachlor - (level 5)	Initial Conc ug/ml	Amount Used ml	Final Volume mls	Final Conc. ug/ml	**GAlacWRK5_**
Alachlor-Intermediate	10	1	10	1.0	GAlacINT_

Alachlor - Level 4	Initial Conc.	Amount Used	Final Volume	Final Conc.	**GAlacWRK4_**
Alachlor-Intermediate	10	0.5	10	0.500	GAlacINT_

Alachlor - Level 3	Initial Conc.	Amount Used	Final Volume	Final Conc.	**GAlacWRK3_**
Alachlor-Intermediate	10	0.25	10	0.250	GAlacINT_

Alachlor - Level 2	Initial Conc.	Amount Used	Final Volume	Final Conc.	**GAlacWRK2_**
Alachlor-Intermediate	10	0.1	10	0.100	GAlacINT_

Alachlor - Level 1	Initial Conc.	Amount Used	Final Volume	Final Conc.	**GAlacWRK1_**
Alachlor-Intermediate	10	0.05	10	0.050	GAlacINT_

Alachlor - Level 0.5	Initial Conc.	Amount Used	Final Volume	Final Conc.	made as needed
Alachlor-Intermediate	0.05	0.1	0.2	0.0250	GAlacINT_

Table 2.3
Preparation of Technical Chlordane

Technical Chlordane Prepare all standards in Iso-octane

Technical chlordane (Intermediate)	Initial Conc ug/ml	Amount Used ml	Final Volume mls	Final Conc. ug/ml	**G_TCLR_INT**
Technical chlordane (32021-Restek)	1000	0.5	10	50	G_TCLR_STK

Technical Chlordane - Level 1	Initial Conc.	Amount Used	Final Volume	Final Conc.	**G_TCLRLVL1**
TCLR -Intermediate	50	0.05	50	0.05	G_TCLR_INT
TCX/DCB Intermediate (5/10)	5	0.05	50	0.005	GSURINT_
Technical Chlordane - Level 2					**G_TCLRLVL2**
TCLR -Intermediate	50	0.1	50	0.10	G_TCLR_INT
TCX/DCB Intermediate (5/10)	5	0.1	50	0.01	GSURINT_
Technical Chlordane - Level 3					**G_TCLRLVL3**
TCLR -Intermediate	50	0.2	50	0.20	G_TCLR_INT
TCX/DCB Intermediate (5/10)	5	0.25	50	0.025	GSURINT_
Technical Chlordane - Level 4					**G_TCLRLVL4**
TCLR -Intermediate	50	0.4	50	0.40	G_TCLR_INT
TCX/DCB Intermediate (5/10)	5	0.5	50	0.05	GSURINT_
Technical Chlordane - Level 5					**G_TCLRLVL5**
TCLR -Intermediate	50	0.8	50	0.80	G_TCLR_INT
TCX/DCB Intermediate (5/10)	5	1	50	0.10	GSURINT_

Table 2.4
Preparation of Toxaphene

Toxaphene Prepare all standards in Iso-octane

Toxaphene (Intermediate)	Initial Conc ug/ml	Amount Used ml	Final Volume mls	Final Conc. ug/ml	**G_TOX_INT**
Toxaphene (32005-Restek)	1000	0.5	10	50	G_TOX_STK

Toxaphene - Level 1	Initial Conc.	Amount Used	Final Volume	Final Conc.	**G_TOX_LVL1**
Toxaphene -Intermediate	50	0.2	50	0.20	G_TOX_INT
TCX/DCB Intermediate (5/10)	5	0.05	50	0.005	GSURINT_
Toxaphene - Level 2					**G_TOX_LVL2**
Toxaphene -Intermediate	50	0.5	50	0.50	G_TOX_INT
TCX/DCB Intermediate (5/10)	5	0.1	50	0.01	GSURINT_
Toxaphene - Level 3					**G_TOX_LVL3**
Toxaphene -Intermediate	50	1	50	1.00	G_TOX_INT
TCX/DCB Intermediate (5/10)	5	0.25	50	0.025	GSURINT_
Toxaphene - Level 4					**G_TOX_LVL4**
Toxaphene -Intermediate	50	2	50	2.00	G_TOX_INT
TCX/DCB Intermediate (5/10)	5	0.5	50	0.05	GSURINT_
Toxaphene - Level 5					**G_TOX_LVL5**
Toxaphene -Intermediate	50	2	25	4.00	G_TOX_INT
TCX/DCB Intermediate (5/10)	5	0.5	25	0.10	GSURINT_

TABLE 3.0
SURROGATE MIX, UG/ML

Surrogate	Stock	Mix for Extractions (added to each sample and spike)
TCX	2.0	0.20
DCB	2.0	0.20

TABLE 3.1
SURROGATE RECOVERY LIMITS

Surrogate recovery limits are entered into the laboratory information system.

TABLE 4.0
PESTICIDE QC CHECK (LCS) AND FULL MATRIX SPIKE, UG/ML

Aldrin	2.0
Dieldrin	2.0
p,p'DDT	2.0
p,p'DDE	2.0
p,p'DDD	2.0
Endosulfan I	2.0
Endosulfan II	2.0
Endosulfan Sulfate	2.0
Endrin	2.0
Endrin Aldehyde	2.0
Heptachlor	2.0
Heptachlor Epoxide	2.0
alpha-BHC	2.0
beta-BHC	2.0
gamma-BHC	2.0
delta-BHC	2.0
Methoxychlor	2.0
Endrin Ketone	2.0
4,4'DDT	2.0
Alachlor*	10
Mirex*	2.0

See Table 7.0 for control limits
* These are not routinely spiked analytes, but can be done upon request.

TABLE 5.0
BREAKDOWN STANDARD CONCENTRATION, UG/ML

Compound	Stock (Individual Compound)	Standard (Working)
Endrin	1.0	0.1
p,p'DDT	1.0	0.1

TABLE 6.0
PRACTICAL QUANTITATION LIMITS (PQL)

Analyte	Quantitation Limits, Water ug/L	Quantitation Limits, Soil ug/Kg
alpha-BHC	0.05	1.7
beta-BHC	0.05	1.7
delta-BHC	0.05	1.7
gamma-BHC	0.05	1.7
Heptachlor	0.05	1.7
Aldrin	0.05	1.7
Heptachlor Epoxide	0.05	1.7
Endosulfan I	0.05	1.7
Dieldrin	0.10	3.3
p,p'DDE	0.10	3.3
Endrin	0.10	3.3
Endosulfan II	0.10	3.3
p,p'DDD	0.10	3.3
Endosulfan Sulfate	0.10	3.3
p,p'DDT	0.10	3.3
Methoxychlor	0.50	17
Toxaphene	2.0	67
Technical Chlordane	0.50	17
Endrin Aldehyde	0.10	3.3

TABLE 6.0 - CONTINUED
PRACTICAL QUANTITATION LIMITS (PQL)

Analyte	Quantitation Limits, Water ug/L	Quantitation Limits, Soil ug/Kg
alpha chlordane	0.05	1.7
gamma chlordane	0.05	1.7
* Isodrin	0.05	1.7
* Chlorobenzilate	0.5	17
Alachlor	0.50	17
Mirex	0.05	1.7
* Endrin Ketone	0.10	3.3

* App.IX Compounds

**Endrin Ketone is not on the Appendix IX list of reported compounds.

TABLE 7.0
QC CHECK CRITERIA

LCS control limits are entered into the Laboratory Information System.

TABLE 8.0
MS/MSD/MSB RECOVERY CRITERIA

MS/MSD/MSB control limits are entered into the Laboratory Information System.

TABLE 9.0
TCLP PESTICIDES

Analyte	Quantitation Limits, ug/L
Technical Chlordane	2.5
Toxaphene	12.5
Endrin	0.50
Heptachlor	0.25
Heptachlor Epoxide	0.25
Lindane	0.25
Methoxychlor	2.5

Table 10
Stock Standards – Manufacturer & Part #'s

Mfgr	Analyte/Mix	ug/mL	Part #
Restek	Pest Std Mix A	8-80	32003
Restek	Pest Std Mix B	8-16	32004
Restek	Pest Mix AB#2	8-80	32292
Restek	Chlordane	1000	32021
Restek	Toxaphene	1000	32005
Restek	Chlorobenzilate	1000	32211
Accustd	Isodrin	100	P-471S
NSI	Mirex	1000	219-02-01
Restek	Endrin	1000	32219
Restek	4,4'-DDT	1000	32203
Supelco	Pest Mix A(SecondSource)	0.5/1	47977
Supelco	Pest Mix B(SecondSource)	5/10	48196
Restek	Alachlor	1000	32204

Appendix C-4

Supporting Information for TCL PCBs

Polychlorinated Biphenyls (PCBs) by GC	8082

Analyte Description	CAS Number
PCB-1016	12674-11-2
PCB-1221	11104-28-2
PCB-1232	11141-16-5
PCB-1242	53469-21-9
PCB-1248	12672-29-6
PCB-1254	11097-69-1
PCB-1260	11096-82-5

Title: SOP for GC Method 8082 PCBs
[Method SW846 8082]

Approvals (Signature/Date):

Technical Manager	10/14/09 Date	Health & Safety Manager/Coordinator	XX/XX /09 Date
Quality Assurance Manager	10/22 /09 Date	Laboratory Director	11/01/09 Date

This SOP was previously identified as SOP No. CT-GCS-23_8.

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 APPROVALS

The approval of the Laboratory Director, the Facility QA Manager, Group Leader and Health and Safety Officer signify that the document has been prepared in accordance with the format requirements that have been established in this document. In addition, if the document is a Method SOP the signature signifies that it meets the specified requirements.

2.0 SCOPE AND APPLICATION

2.1 This method defines the specific steps for analyzing and determining the concentration of various polychlorinated biphenyls (PCBs) in multimedia, multi-concentration samples.

2.2 It is the policy of TestAmerica and of the chromatography group to ensure that we administer contracts and orders for goods and services in a manner that is fully compliant with governmental laws and regulations, as well as the TestAmerica Policy Statement on Business, Ethics and Conductivity.

2.3 Refer to Table 5 of this SOP for the list of parameters.

2.3 The document control number for this SOP is CT-GCS-23, Rev 9.

3.0 TERMS AND DEFINITIONS

3.1 There are many definitions used with in the laboratory, which may be generic to all laboratory analyses, or more specific for certain methods. For the most recent terms and definitions used with in the laboratory, reference the SOP for Terms and Definitions.

4.0 SUMMARY OF METHOD

4.1 This method outlines the gas chromatographic procedure for the detection of polychlorinated biphenyls. Samples are extracted using the proper extraction technique. The extracts are analyzed by gas chromatography with an electron capture detector.

4.2 This SOP is based on the following methods:

- EPA Method 8082 (PCB's as Aroclors by Gas Chromatography)
- EPA Method 8000B (Determinative Chromatographic Separation)
- EPA Method 8000C (Determinative Chromatographic Separation)

4.3 Deviations to Method

4.3.1 The laboratory at this time, does not analyze samples for individual PCB congeners.

5.0 INTERFERENCES

5.1 Phthalate esters can interfere with PCB determinations; avoid any contact with plastics to best minimize this problem.

5.2 Sulfur is also an interference; this can be removed by performing sulfur clean-up on the extract by using TBA cleanup. See Sulfur cleanup SOP for further information.

5.3 If an extract is to be analyzed for PCB's only, then an optional sulfuric acid cleanup may be performed. This cleanup procedure will remove contamination, which may interfere with the analysis of the eluting PCB's, however; the procedure may also cause degradation of other pesticide and surrogate compounds. If contamination, due to sample matrix, is suspected in the sample, or if the original extract analysis exhibits interferences, then the extract may be cleaned up with concentrated sulfuric acid and reanalyzed. A portion of the original method blank is also acid cleaned up and analyzed.

5.3.1 Acid cleanup is performed by adding one part sulfuric acid to two parts of the hexane extract in a clean vial. Tighten the vial and agitate the sample for thirty seconds. After cleanup, pipet the extract layer (top layer) to another vial. Repeat the cleanup procedure until the acid layer is light brown to orange in color. Complete the acid cleanup procedure by performing a sulfur cleanup. This cleanup must also be performed on the corresponding method blank and QC if all samples required cleanup, or a portion of the method blank and QC if only some of the samples required cleanup. Refer to Sulfur Removal SOP. Document in the case narrative each sample on which sulfur cleanup was performed.

6.0 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.

6.1 SPECIFIC SAFETY CONCERNS OR REQUIREMENTS

The gas chromatograph contains 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.

There are areas of high voltage in the gas chromatograph. Depending on the type of work involved, either turn the power to the instrument off, or disconnect it from its source of power.

6.2 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
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.
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.
1 – Exposure limit refers to the OSHA regulatory exposure limit.			

7.0 SAMPLE CONTAINERS, COLLECTION AND PRESERVATION

7.1 Sample Containers

- Water samples are collected in 2x1 liter amber glass containers with Teflon-coated liner and Teflon coated caps..
- Soil samples are collected in 250 or 500 mL glass containers with Teflon coated caps.
- Sample bottles are never reused.

7.2 Sample Collection

- Samples are secured against breakage in the shipping containers and kept at 4°C for transport to the laboratory. Samples should arrive at the laboratory the next day following collection.

7.3 Sample Preservation

- Samples are preserved by cooling to 4°C.

7.4 Holding Times

- Water samples must be extracted within seven days from collection.

. Soil samples must be extracted within 14 days from collection.

. Aqueous samples requiring NYSDEC ASP must be extracted within five days from VTSR.

. Soil samples requiring NYSDEC ASP should be extracted within 14 days from collection.

. All extracts must be analyzed within 40 days from date of extraction (40 days from collection for NYSDEC ASP).

8.0 APPARATUS AND MATERIALS

8.1 Sample extracts are analyzed on a gas chromatograph (GC) equipped with an electron-capture detector (ECD), autosampler, data collection system and all other required accessories. The following columns are acceptable for analysis:

See section 12.1 for instrumentation specifications

. Restek Rtx-CLPesticides 15 meter
0.53mm ID 0.50um film thickness
or equivalent

. Restek Rtx-CLPesticidesII 15meter
0.53mm ID 0.50um film thickness
or equivalent

. Turbochrom Data Acquisition Software System or Chemstation software

. Target Software

. Various sizes of syringes, volumetric pipets, volumetric flasks, pipet bulbs

. 0.8 ml and 1.8 ml autosampler vials and caps.

. Borosilicate glass transfer pipets/transfer bulbs.

. Safety glasses, non-powdered polyvinyl gloves, fume hood.

. Properly cooled refrigerators each for sample and standard storage.

. Standard and Instrument Maintenance logbooks.

. Sample Injection logbooks.

9.0 REAGENTS AND STANDARD PREPARATION

9.1 Solvents: Hexane, Acetone, Toluene and Isooctane (2,2,4-trimethylpentane) should be pesticide grade or equivalent.

9.2 Commercially prepared stock standards can be used if they are certified and pre-tested by the manufacturer.

9.3 Standard Expiration Dates:

- Once opened, all stock standards are stored in amber screw top vials(2ml) at 4°C and replaced after 1 year or earlier if routine QC tests indicate a problem.
- All working standards are stored in amber screw top bottles at 4°C and replaced after 6 months or earlier if routine QC tests indicate a problem.

9.4 Calibration Stock Standard

9.4.1 **Multi-component Standards** - A minimum five point calibration curve for combination standard mix of Aroclor-1016 and Aroclor-1260 is performed and a single midpoint calibration standard of all other Multi-components. All of these standards are diluted from individual stocks. For Multi-component Stock and Standard concentrations see Table 1.0. For preparation of standards see Table 2.0.

9.5 **Surrogate Standard** is prepared to monitor the performance of the extraction and analytical system. Samples, standards and blanks are spiked with pesticide surrogates. Two surrogates, 2,4,5,6-tetrachloro-m-xylene (TCX) and Decachlorobiphenyl (DCB), are spiked with the proper amount at a level of 0.2 ug/L for waters and soils are spiked at a level of 6.7 ug/Kg. (See Table 3.0).

9.6 **Quality Control Check and Matrix Spiking Standard Solutions**: A mixture of Aroclor 1016/1260 is used as the LCS spike solution. A mixture of Aroclor 1016/1260 is also used as the MS/MSD spike solution. 1.0 ml is spiked into each aliquot of the sample for the matrix spike and matrix spike duplicate, and to 1.0 L of reagent water for the QC check sample. (See Table 4.0).

10.0 CALIBRATION

10.1 RT Windows are established by making three injections of the mid concentration standard throughout the course of a 72-hour period and calculating 3 x ± the standard deviation. Retention time windows shall be calculated for each compound on each GC column whenever a new type of column is installed.

10.1.1 RT windows are established as described above; however, the laboratory has established a minimum RT window of 0.05.

10.2 Calibration Standards

Initial calibration standards are analyzed by injecting 1 ul of six levels of AR1660 standard, and one level of all other multi-response PCB's. (Aroclor 1016 and 1260 include all congeners present in the different regulated Aroclors).

AR1660 is analyzed at six levels, within a minimum requirement of 5 being used. The lowest level analyzed is below routine reporting limits and may be excluded. A point in the middle of the curve may not be excluded unless it meet requirements as outlined in the corporate SOP on Selection of Calibration points.

10.2.1 The initial calibration criteria is 20% RSD. If the %RSD cannot be met using the average calibration factor, an alternate curve type can be used. The criteria are 0.990 or higher for the coefficient.

10.2.2 In determining the best fit of a curve, using either linear regression or Quadratic, the analyst shall choose the best fit possible based on both the coefficient and the slope of the line (b). The coefficient should be greater than 0.990 and as close to 1 as possible. The slope of the line (b) should be as low a value as possible. The analyst may choose between linear regression, a weighted linear regression or a Quadratic fit. The line may not be forced through zero.

10.3 The working calibration range of this method is defined by the initial calibration curve. All extracts with target compounds exceeding the curve must be diluted to within the upper half of the calibration range.

10.3.1 The daily calibration verification is acceptable if all of the following are true.

10.3.1.1 The calibration verification standard must have all compounds (+/-) 15% difference of their expected value. Because of the low concentration of pesticide standards injected on a GC/ECD, column adsorption may be a problem when the GC has not been operated for a day. Therefore, the GC column should be primed by injecting a standard mixture approximately 20 times more concentrated than the mid-level standard.

If the calibration verification standard fails, then it can be rerun. A new calibration curve will be run, if warranted, due to repeated failure. When the calibration verification standard fails, all samples that were injected after the last standard that met the QC criteria must be evaluated to prevent any mis-quants and possible false negatives. Depending on the compound failures the extracts may need re-injection. More frequent analysis of standards will minimize the number of reruns for QC failures.

10.3.1.2 An instrument blank standard is run after the calibration verifications standards and prior to any samples to ensure the instrument and its autosampler are clean.

10.3.2 All initial calibrations must be verified with a standard obtained from a second source manufacturer or lot. The same manufacturer may be used, if the lot can be demonstrated from the manufacturer as prepared independently from other lots. Traceability shall be to a national standard when commercially available. The % difference of this standard should pass within +/- 25% from the initial calibration curve. If the second source standard fails to meet criteria, check the preparation of both the initial curve and the second source standard for errors. It may be necessary to re-prepare a solution to check.

10.4 When analyzing samples, any extract that contains a target compound that exceeds the high level calibration must be diluted to within the calibration range.

10.5 Calibration Verification - (Each 12 hour shift)

10.5.1 A single multi-compound standard is analyzed at a minimum of every 20 samples and at the end of the analysis sequence (It is recommended every 10). The % difference of the average of the 5 peaks for an aroclor, must not exceed a 15 percent. When this criteria is exceeded, inspect the GC system to determine the cause and perform whatever maintenance is required before recalibrating and proceeding with sample analysis.

10.6 Calculations for water samples:

10.6.1 An average calibration factor is calculated for each peak (using 3-5 peaks) and surrogate from the initial calibration.

Multi-component Calculation

$$ug/L = \frac{(X)\ \text{Final Volume of extract (uL)} \times \text{Dilution Factor}}{(Y)\ \text{Sample Volume extracted (mLs)} \times \text{ul inj.}}$$

X = The area of one of the 3-5 peaks from sample.
Y = The average CF for the corresponding peak from standard.

A Cf must be calculated for 3-5 peaks. The final concentration reported is calculated using the average of the final result for each of the 3-5 peaks.

10.7 Calculations for soil and oil samples:

10.7.1 An average calibration factor is calculated for each peak (using 3-5 peaks) and surrogate from the initial calibration.

Multi-component Calculation

$$ug/Kg = \frac{(X)\ \text{Final Volume of Extract (uL)} \times \text{Dilution Factor}}{(Y)\ \text{Sample Volume Extracted(g)} \times \text{Decimal \% Solids} \times \text{ul inj.}}$$

X = The area of one of the 3-5 peaks from sample.
Y = The average CF for the corresponding peak from standard.

A Cf must be calculated for 3-5 peaks. The final concentration reported is calculated using the average of the final result for each of the 3-5 peaks.

Note: Oils will not have a decimal percent solid.

10.7.2 When more than one multi-component is detected in a sample, non-overlapping peaks are chosen for quantitation. If it is not possible to choose non-overlapping peaks, peaks with the least amount of overlap are chosen. Refer to Appendix A for the most common peaks chosen for quantitation. Three to five of the peaks are used to calculate the calibration curve, and any results from corresponding samples.

10.8 Corrective Action for Initial Calibration

10.8.1 If the technical acceptance criteria for the initial calibration are not met, inspect the system for problems. It may be necessary to change the column, bake out the detector, clean the injection port, or take other corrective actions to achieve the acceptance criteria.

10.8.2 Corrective Action for Calibration Verification

10.8.3 If the technical acceptance criteria for the calibration verification are not met, inspect the system for problems and take corrective actions to achieve the criteria.

10.8.3 Major corrective actions such as replacing the column or detector will require a new initial calibration. A return to control indicating the next passing curve shall be documented in the maintenance log.

10.8.4 Minor corrective actions may not require a new initial calibration provided a calibration check meets all acceptance criteria.

11.0 QUALITY CONTROL

11.1 Refer to Table 5.0 for Practical Quantitation Limits (PQL's) for all compounds.

11.2 Matrix spike (MS), matrix spike duplicates (MSDs) and matrix spike blanks (MSB's), if applicable, are analyzed within every set of 20 samples or less. Recoveries must be within the limits listed in Table 4.1 of this SOP. If these criteria are not met, but the blank spike data meet all of the recovery criteria, then the MS/MSD are documented as having matrix interferences. If the blank spike fails, check for instrument and/or column related problems and reanalyze the spikes. If the problem is corrected, the samples are reanalyzed.

11.3 A QC check sample is performed at a frequency of one per 20 samples extracted of similar matrix. See Table 4.0 for spike compounds and control limits.

11.4 Blanks

11.4.1 Method Blank

Method blanks are spiked with surrogates, extracted, and analyzed following the same procedure that is used with the associated samples. A water method blank is one liter of reagent water and a soil method blank is 30 grams of sodium sulfate for sonication extraction and 15 grams of sodium sulfate for automated soxtherm extraction.

Frequency - method blanks must be analyzed with each case, 20 samples of a similar matrix, or whenever samples are extracted by the same procedure, whichever is more frequent.

Acceptance criteria - method blanks must contain less than half the PQL for all the target compounds listed in Table 1.0 & 1.1.

All samples associated with an unacceptable method blank must be re-extracted and reanalyzed.

11.5 Surrogates

11.5.1 The surrogates TCX and DCB are added to each standard, sample, blank and QC prior to extraction. Results for the surrogates are calculated from the Aroclor 1016/1260 calibration standards.

11.5.2 The QC limits for surrogate recovery are listed in Table 3.1 and pertain to all samples, blanks and spikes.

11.6 Daily Calibration Verification: One per 12 hour shift
Instrument Blank: One per 12 hour shift
MS/MSD: Every 20 samples
QC Check: Every extraction batch
Surrogate: All samples and standards
Method Blank: One per batch

11.7 Analytical Documentation Procedures

11.7.1 Instrument Batches

An instrument batch is created for each analytical sequence to organize all the associated data. Batch designations are of the format:

XXXnnn####

where: XXX = instrument identifier
nnn = number of batch
####=Method

(i.e. CD9001-8082)

Instrument batches are number sequentially so a unique batch identifier identifies each analytical sequence. The batch consists of all the associated QC information for the analytical sequence.

11.7.2 Data Archiving

All data files are archived on a daily basis using a 12.0gb data storage cartridge. The associated method files are also archived daily to provide an accurate historical record. Care shall be exercised when purging data off the hard drives to ensure that all data being purged has been archived.

11.7.3 Instrument Run Logs

It is TestAmerica's policy that all measurement data be recorded in logbooks or on preprinted log sheets in permanent ink. Transcriptions shall be avoided whenever possible. The record shall reflect the measurement performed and all appropriate details for conclusions related to the measurement. The record shall be signed and dated by the individual performing the measurement

on the day the measurement is performed. Corrections shall be made by drawing a single line through the error, and initialing and dating the correction. A secondary authorization of the logbook is required and shall be performed by the department's manager or designee.

Each instrument has its own set of bound run logs which are sequentially numbered and paginated. Run logs are filed in the laboratory once they have been filled, for future reference. Each analytical sequence shall be started on a new page of the log and continued on the next page, if necessary. The header information designating the standard codes used shall be completed for each sequence. All standards used are recorded in this field for future traceability. The data file, job number, sample number, quantitation factor, dilution factor, analyst's signature, and date are recorded.

11.8.5 Corrective Action Reports

A corrective action report (CAR) is initiated when a problem is encountered during analysis, data reduction or deliverables preparation, data validation, or when any deviations from this SOP occur. The CAR is initiated by the analyst or dept. manager first identifying the problem through the NCM module in the LIMS system. It is then electronically forwarded to all appropriate departments, QA officer, and Lab Manager. Reference SOP for correction action reports.

11.8.6 Chain of Custody Record

When samples are removed from storage for preparation or analysis they must be signed out utilizing the chain of custody record (COC). The samples shall then be signed back in on the COC upon their return to storage or designated "used" if the sample volume is consumed during the preparation or analysis.

11.8.7 Sample Tracking Record

The Sample Control department does notification of sample arrival by issuing a preliminary notification sheet. Samples are tracked for extraction and analysis by using the laboratory's LIMS system.

11.9 Quality Control Check Points

11.9.1 Analysis quality control approval report

Specific quality control checkpoints have been established for the analysis of sample extracts. The specific check points in the analysis logbook, are initialed and dated by the analyst to ensure the consistency and accuracy of the data produced.

11.9.2 Specific quality control checkpoints have been established for the preparation of data deliverables, which are monitored through a Lims Organics Data Review Checklist. The specific check points must be reviewed by the analyst 1st level reviewing and the secondary reviewer 2nd level reviewing the data to ensure the consistency and accuracy of the data produced. Refer to Figure 1.0 for the document and specific control points covered.

12.0 SAMPLE PREPARATION AND INSTRUMENTAL PROCEDURES

12.1 HP5890 Series II GC's with dual ECD's or HP6890's with Micro ECD's and 7673A Twin Tower Autosampler or CTC (Leap Technologies) Autosampler is currently being used to run Pesticides/PCBs under this SOP. Instruments, columns and guidances conditions are as follows:

a) Column flow: ~8 mL/min He
Detector Make-up Flow: 65 mL/min N_2
Injector Temperature: Tracks oven temperature +3° C
Detector Temperature: 330°C

Temperature Programming:
Initial Temperature: 150°C Hold 0.8 min
Initial Ramp: 11°/min to 208, 0.6min Hold
Final Ramp and Temp.: 35°/min to 315, Hold 0.7 min

Instrument and column conditions may have equivalent programming as long as all method QC requirements are met.

12.2 Aqueous Sample Preparation for PCBs:

12.2.1 Brief Summary:
A 1 liter sample aliquot is spiked with the surrogate and extracted with methylene chloride at a pH between 5 and 9. Using either the separatory funnel extraction method or the continuous liquid-liquid extraction method is acceptable. The methylene chloride extract is dried and solvent exchanged to hexane and adjusted to a final volume of 10 mL.

A 1 uL aliquot of the sample extract is injected into the gas chromatograph (GC). The GC separates the compounds. Compound identification is performed by the comparison of GC retention times to those of known analytical standards. Quantitative analysis is performed by the comparison of compound peak height or peak areas to those of an analytical standard at a known concentration.

12.2.2 For TCLP samples, 800 mLs of reagent water is added to 200 mLs of the TCLP leached sample and the procedure would follow Section 12.2.1.

12.2.3 Sample Extraction

Refer to TestAmerica's Standard Operating Procedure for water extraction.

12.3 Soil Sample Preparation for PCB's

12.3.1 Brief Summary

A 30 g sample aliquot is spiked with surrogate and extracted with a 1:1 mixture of hexane/ acetone using the sonication extraction method. The hexane /acetone extract is dried and concentrated to a final volume of 10 mL. A 1 uL aliquot of the sample extract is injected into the gas chromatograph (GC). The GC separates the compounds. Compound identification is performed by the comparison

of GC retention times to those of known analytical standards. Quantitative analysis is performed by the comparison of compound peak height or peak areas to those of an analytical standard at a known concentration.

12.3.2 A 15 g sample aliquot is spiked with surrogate and extracted with a 1:1 mixture of hexane/ acetone using the soxtherm apparatus. The hexane /acetone extract is dried and concentrated to a final volume of 5 mL. The sample extraction must be completed within 14 days of sample collection. A 1uL aliquot of the sample extract is injected into the gas chromatograph (GC). The GC separates the compounds. Compound identification is performed by the comparison of GC retention times to those of known analytical standards. Quantitative analysis is performed by the comparison of compound peak height or peak areas to those of an analytical standard at a known concentration.

12.3.3 Sample Extraction

Refer to TestAmerica's Standard Operating Procedure for soil extraction by sonication and soxtherm.

12.4 Sample Analysis Procedures

12.4.1 Sample Extract Analysis

Sample extracts are removed from storage in the G.C. instrument room and are signed out on the extract chain of custody form. All sample extracts are signed back in after they are returned to storage.

Make sure all instrumental operating conditions are correctly set.

In a vial with a 200 ul insert, load approximately 200 ul aliquot of sample extract. A 1.0 ul injection of the sample extract onto the GC column is made with an autosampler and then the GC temperature program sequence is started.

This method is intended to achieve the quantitation limits whenever possible. If sample chromatograms have interfering peaks, high baseline, or off-scale peaks, then those samples must be reanalyzed following dilution, cleanup, or reextraction. No limit is placed on the number of re-extractions of samples that may be required because of contaminated method blanks.

The sample must be analyzed at the most concentrated level that is consistent with achieving satisfactory chromatography (defined in Section 12.7).

No target analyte concentrations may exceed the upper limit of the initial calibration.

12.5 Qualitative Analysis

12.5.1 Target Compounds

The identification is based on retention time data as well as Aroclor pattern.

If the pattern appears to be that of an Aroclor, and is greater than the PQL, that sample may be

confirmed by analysis on a second column of dissimilar phase, see section 8.1.

12.6 Quantitative Analysis

12.6.1 Target Compounds

Target compounds are quantitated by the external standard technique using peak area and the calibration factor determined during the initial calibration sequence.

When compound concentrations are below the PQL, but the compound meets identification criteria, report the concentration with a "J" qualifier.

When a compound exceeds the linear working range of the initial calibration, the sample must be diluted to bring the analyte concentration within the calibration range.

The following are guidelines on performing dilutions and exceptions to this requirement:

. If the response is still above the high calibration point after the dilution of 1:100,000, the laboratory shall contact the client.

. Use the results of the original analysis to determine the approximate dilution factor required to get the largest analyte peak within the initial calibration range.

12.6.2 The lab routinely reports the higher concentration of the results between the two columns used for analysis. If there is a greater than 40% difference between the two columns, the lower result is reported.

12.7 Instrument Maintenance

12.7.1 Preventative Maintenance

All instrumentation is covered by a service contract with an external instrumentation service vendor, or by TestAmerica personnel trained in preventative maintenance. All instrument preventative maintenance is performed according the manufacturers recommended procedures, by trained personnel. All preventative maintenance shall be thoroughly documented in the maintenance log (see Figure 4.0), as to a description of the maintenance performed, the date performed, and the personnel performing the maintenance.

12.7.2 Corrective Maintenance Determinants and Procedures

Corrective maintenance is deemed necessary when the analytical system, after reanalysis, cannot meet calibration, resolution, chromatography, breakdown, or other protocol specific QC criteria. Corrective maintenance may include, but is not limited to, decontamination of the system, injection port cleaning, column cutting or replacement, syringe cleaning or replacement, or detector baking out or replacement. All corrective maintenance is performed according the manufacturers recommended procedures, by trained personnel. All corrective maintenance shall be thoroughly documented in the maintenance log, as to a description of the maintenance performed, the date performed, and the personnel performing the maintenance. A return to control documenting the

next valid curve shall be noted in the maintenance log in the same section.

12.7.3 Maintenance Authorization

The department's manager or designee authorizes all preventative and corrective maintenance. When maintenance is deemed necessary, a service call is placed for all equipment covered under a service contract, by the department's manager, or designee.

12.8 Data System

12.8.1 Data Acquisition and System Operation

Data is acquired from sample analyses using the Perkin Elmer TurboChrom or Chemstation computer system. Analytical batches are set up with all the associated sample ID, dilution, and data file information. Automated post-acquisition is qued with the appropriate method file and sent to the Target data system for integration and quantitation. Turbochrom and/or Chemstation has instrument control.

12.8.2 Instrument Errors

System errors are logged to the system console at time of occurrence. The system manager shall be responsible for checking and providing corrective actions for all system errors.

12.8.3 Manual Integrations and Editing Flags

Manual integrations are required when the automated software doesn't correctly integrate extracted ion current profiles (EICP). A user shall be logged into the Target system as their own name. This name will signify who performed the manual integration. To perform a manual integration, the target compound of interest is selected and the EICPs are graphically presented. The peak can then be correctly integrated. A reason code shall be selected for the type of integration performed by selecting Review codes from the menu options.

UN = Unidentified peak based on spectra or concentration
ID = Identified a peak based on spectra
INT = Integrated a peak due to incorrect integration

A new quantitation report is produced. The manually integrated data file is the saved by exiting and saving from file edit. Manual integrations are flagged by the data system with the "M" qualifier beside any manually integrated area. The manually integrated data file is the saved by exiting from Target review. Manual integrations are flagged by the data system with the "M" qualifier beside any manually integrated area on the hardcopy quant report. The analyst name will appear on the electronic manual integration report which is uploading to the LIMS system when data files are uploaded. A hardcopy print out of the EICP of the quant ion displaying the manual integration shall be produced for the before and after integrations and is included in the raw data to the clients when the report is generated during final packaging.

13.0 CALCULATIONS

See Sections 10.5.1, 10.5.2, 10.6.1 and 10.6.2 of this document.

13.1 Calculation of Calibration Factor

$$CF = \frac{\text{Peak Area of the Standard}}{\text{Mass Injected (ng)}}$$

13.1.1 Calculation of Percent Difference

The following formula is used to calculate % difference in the calculated versus expected values of standards.

$$\% \text{ Difference} = \frac{|\text{Calculated conc. - expected conc.}|}{\text{Expected conc.}} \times 100$$

13.1.2 Calculation of Surrogate, Spike and QC Check Recoveries

The following calculation is used for spiked sample recoveries.

$$\% \text{ Recoveries} = \frac{\text{Amount Recovered - Amount in Sample}}{\text{Amount Added}} \times 100\%$$

13.1.3 Percent Relative Standard Deviation (%RSD)

$$\%RSD = \frac{\text{Standard Deviation}}{\text{Average CF}} \times 100$$

13.1.4 Percent Moisture

$$\% \text{ Moisture} = \frac{\text{g of Wet Sample - g of Dry Sample}}{\text{g of Wet Sample}} \times 100$$

13.1.5 Adjusted Practical Quantitation Limit for Samples

$$\text{Adjusted PQL} = \frac{(PQL) \times Df}{D}$$

where:

$$D = \frac{100 - \% \text{ Moisture}}{100}$$

DF = Dilution Factor

14.0 ACCEPTANCE OF DATA

14.1 Daily Calibration Check Standard (Required every 12 hour shift)

Verification of the calibration curve with a multi-component calibration standard is obtained if the calculated concentration of all the compounds to be quantitated are (+/-) 15% of the expected

value.

14.2 Instrument Blank

The instrument blank is used to verify that the analytical system is free of contaminants. The instrument blank shall be free of any target compounds above half the quantitation limits and shall not contain any unusual interferences.

14.3 Method Blanks

Method blanks are extracted with every batch of up to 20 samples to ensure that there is no contamination from the extraction process. The method blanks shall be free of any target compounds above half the quantitation limits and shall not contain any unusual interferences.

14.4 Matrix Spikes and Matrix Spike Duplicates

Matrix spikes and matrix spike duplicates are extracted with every batch of up to 20 samples to verify extraction efficiencies. Acceptance criteria is listed in Table 4.0.

14.4.1 MSB's are extracted with every batch of 20 samples as applicable to client requested protocol. Acceptance criteria are listed in Table 4.0

14.4.2 QC reference samples are extracted with every extraction batch of up to 20 samples to verify extraction efficiencies.

15.0 REPORTING OF RESULTS

15.1 All results are reported to two significant figures. Water samples are reported in ug/L, soil samples are reported in ug/Kg dry weight and waste samples are reported in ug/Kg.

Check reporting deliverables required from LIMS. All job packages require a case narrative and quality control approval report. The case narrative should outline in detail any problems with client samples during analysis. The following indicates the different levels of reporting.

Level I

- Case Narrative
- Sample results

Level II

- Case Narrative
- Sample Results
- Surrogate Recovery form
- LCS and MS/MSD recovery forms

Level III

-Includes everything listed below, *Except* standard scans.

NJ/CLP/NYSDEC /Level 4

- Case Narrative
- Form 1 (Organic Analysis Data Sheet)
- Surrogate Recovery form
- LCS and MS/MSD recovery forms
- MSB recovery form as applicable
- Form 4C (Method Blank Summary)
- Initial Calibration Forms
- Analytical Sequence Form
- Breakdown Check Form
- Continuing Calibration Forms
- Form 10 (Pesticide/PCB Identification)
- Sample and Standard Scans and Area Reports
- Standard Concentration Summary
- GC/MS Confirmation (if applicable)

16.0 SUPPLEMENTAL DOCUMENTS

16.1 SOP for Pesticide Extract Sulfur Removal by Method 3660B.

16.2 SOP for Pesticides in Water Extraction by Method 3510C.

16.3 SOP for Pesticides in Soil Extraction by Method 3550B.

16.4 Tables attached include the following:

Table 1.0 - Surrogate Calibration Concentrations
Table 2.0 and 2.1 - Multi-Component Concentration
Table 3.0 - Surrogate Mix
Table 3.1 - Surrogate Recovery Limits
Table 4.0 - Aroclor QC Information
Table 4.1 - Aroclor Matrix Spike Information
Table 5.0 - Practical Quantitation Limits (PQL)

Figure 1.0 - Lims Organics Data Review Checklist

17.0 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). Employees must abide by the policies in Section 13 of the Corporate Safety Manual for "Waste Management and Pollution Prevention."

17.1 Pollution prevention is the reduction and/or elimination of wastes or the toxicity of wastes at the point of generation. The laboratory strives to keep all wastes at a minimum through a variety of

techniques. These include the following.

17.1.1 Inventory control: Rotate stocks (first in, first out) and only purchase reasonable amounts of chemicals. Do not purchase excessive quantities, which will be discarded as wastes at some future point.

17.1.2 Material Handling and Storage: Keep containers properly labeled. Keep solvents covered. Store and handle all chemicals, reagents, standards, etc. to prevent contamination and degradation.

17.1.3 Personnel: All staff must be trained in the proper handling and disposal of waste.

17.1.4 Waste Reduction: Reduce the volume of waste generated wherever possible.

17.1.5 Chemical/material substitution: Use less toxic chemicals whenever possible. Avoid the use of chromic acid (Chromerge) solutions.

18.0 WASTE MANAGEMENT AND POLLUTION PREVENTION

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 Tracking and Collection of Hazardous Waste SOP. The following waste streams are produced when this method is carried out.

18.1 All autosampler vials containing PCB's should be disposed of in the 5 gallon bucket labeled for Hexane waste.

19.0 REFERENCES

19.1 "Method 8000C – "Gas Chromatography", EPA SW846, 3rd Revision.

19.2 "Method 8000B – "Gas Chromatography", EPA SW846, 3rd Edition
Project Dependent.

19.3 "Method 8082 - PCBs as Aroclors by Gas Chromatography, EPA SW846, 3rd Edition

19.4 "Methods of Organic Chemical Analysis of Municipal and Industrial Wastewater", Federal Register Vol. 49, No. 209, October 26, 1984.

20.0 SUBSTANTIVE REVISIONS

20.1 Revised section 8.3 changing the standard storage time frame, revised section 9.7.2 adding a reference to the added Appendix A - Chromatograms of Aroclors, revised section 10.4.1 changed method blank criteria; 03/05/99.

20.2 Added Terms and Definitions section, Pollution Prevention, and Waste Management; 2/15/2000. Added Initial calibration %RSD criteria and linear regression criteria.

20.3 Changed name on cover from Monroe to Connecticut, no other changes required at this time; 8/16/00.

20.4 Added to Safety section.

20.5 Added to the Safety section and Waste Management sections. Added EH&S Officer to Approval section. Section 8.1 – added equivalent column. Section 11.4.1 Added soxtherm blank information. Section 9.6 – changed Aroclor-1242 to Aroclor-1016. Section 12.1 – added autosampler. Changed Tables 3.1, 4.0, and 4.1 to refer to LIM system for updated control limits. Section 18.1 revised wording of autosampler vial disposal. Section 12.3.2 added Soxtherm brief summary-1/19/2004.

20.6 May 7, 2004- Added 40% Rule for reporting of results to Section 12.7.

20.7 April 19, 2005- Section 9.6, Changed MS/MSD solution to be Aroclor 1016/1260 and updated Table 4.1. Update Revision # and Date Effective. Added to Section 19, 8000C Reference.

20.8 May 15, 2007 – Section 5.2 – Added use of copper cleanup for sulfur. Sect. 8.1 – updated to use current columns. Sect 9.3 – changed from 6months to 1 year. Sect.10.2.1 – changed from .995 to 0.990 correlation coefficient. Sect 10.8.3 – Added return to control documenting. Sect 11.7.1 – files now scanned, not filed. Sect. 11.7.2 removed filing system section. Sect 11.8.5 – changed CAR procedure to electronic. Sect 11.8.7 – Removed reference to Labnet. Sect 11.9.2 – Changed from QCAR to Lims checklist for $1^{st}/2^{nd}$ level review. Sect 12.1 – updated to 6890's, columns, temperatures and ramps. Sect 12.6.1 – Removed that 2 analyses shall not be submitted. Sect 12.7.2 – Added to document a return to control. Sect 12.8.1 – Added Chemstation. Sect 12.8.3 – Changed manual integration signing. Sect 15.1 – changed reference from Labnet to Lims. Table 5.0 – added Aroclor-1262 and Aroclor-1268 as options.

20.9 Sept. 10, 2007- Sect 10.2.2 – added on choosing best fit for linear regression. Sect 5.3.2 and 5.3.3 removed on silica gel and florisil cleanup. Sect 6.2 – footnote 1 removed. Sect 7.1 – added caps. Sect 8.1 – added reference to sect 12.1. Sect 9.4.1 – added preparation to tables 1.0 and 2.0. Sect 12.3.1 – removed mecl2/acetone reference. Sect 10.3.2 added for 2^{nd} sources. Sect. 11.8.5 updated process.

20.10 10/22/09 - Section 5.2-Removed copper and mercury cleanup for sulfur and added TBA. Section 10.2-Alternate curve types cannot be forced thru zero. Added sixth calibration point option. Section 11.5.1-Removed annotation concerning calculation of surrogates from IND mixes if Pesticides are required. Section11.7.1-Analytical batch numbering-updated and gave new example. Table 1.0, Table 2.0, and Table 5.0 changed information on Aroclor 1221, lowering concentration of standard and lowering Quantitation limits. Sect 9.3 updated standard expiration dates. Updated safety, pollution control and management sections.

Table 1.0 (preparation)

AR1660 Mixes

Aroclor 1016/1260 Intermediate	Initial Conc ug/ml	Amount Used mls	Final Volume mls	Final Conc. ug/ml	G_1660_INT_
Ar1016/1260 (G_1660_STK_)	1000	0.5	10	50	

TCX/DCB Intermediate	Initial Conc ug/ml	Amount Used mls	Final Volume mls	Final Conc. ug/ml	GSURINT_
TCX (G_TCX_STK_)	200	0.625	25	5	
DCB (G_DCB_STK_)	200	1.25	25	10	

Ar1660 Mix0.5	Initial Conc.	Amount Used	Final Volume	Final Conc.	Made as needed
G1660WRK1_	0.05	0.1	0.2	0.025	
Surrogate (concentration)				0.0025	DCB 2x higher

Ar1660 Mix1	Initial Conc.	Amount Used	Final Volume	Final Conc.	G1660WRK1_
Ar1660Int (G_1660_INT_)	50	0.1	100	0.050	
SurrInt (GSURINT_)	5.0	0.1	100	0.005	DCB 2x higher

Ar1660 Mix2	Initial Conc.	Amount Used	Final Volume	Final Conc.	G1660WRK2_
Ar1660Int (G_1660_INT_)	50	0.2	100	0.1000	
SurrInt (GSURINT_)	5.0	0.2	100	0.0100	DCB 2x higher

Ar1660 Mix3	Initial Conc.	Amount Used	Final Volume	Final Conc.	G1660WRK3_
Ar1660Int (G_1660_INT_)	50	0.4	100	0.2000	
SurrInt (GSURINT_)	5.0	0.5	100	0.0250	DCB 2x higher

Ar1660 Mix4	Initial Conc.	Amount Used	Final Volume	Final Conc.	G1660WRK4_
Ar1660Int (G_1660_INT_)	50	0.8	100	0.4000	
SurrInt (GSURINT_)	5.0	1	100	0.0500	DCB 2x higher

Ar1660 Mix5	Initial Conc.	Amount Used	Final Volume	Final Conc.	G1660WRK5_
Ar1660Int (G_1660_INT_)	50	1.6	100	0.8000	
SurrInt (GSURINT_)	5.0	2	100	0.1000	DCB 2x higher

Ar1660- Mix3 ICV	Initial Conc.	Amount Used	Final Volume	Final Conc.	G1660ICV_
Ar1016 (G_1016_SS_)	1000	0.01	50	0.20	
Ar1260 (G_1260_SS_)	1000	0.01	50	0.20	
SurrInt (GSURINT_)	5.0	0.25	50	0.025	DCB 2x higher

PCB Mixes

Aroclor 1221 Intermediate	Initial Conc ug/ml	Amount Used mls	Final Volume mls	Final Conc. ug/ml	G_1221_INT_
Ar1221 (G_1221_STK_)	1000	0.5	10	50	Restek 32007

Ar1221 Mix 2	Initial Conc.	Amount Used	Final Volume	Final Conc.	G1221WRK2_
Ar1221 Int (G_1221_INT_)	50	0.1	50	0.10	
SurrInt (GSURINT_)	5	0.1	50	0.010	DCB 2x higher

Aroclor 1232 Intermediate	Initial Conc ug/ml	Amount Used mls	Final Volume mls	Final Conc. ug/ml	G_1232_INT_
Ar1232 (G_12321_STK_)	1000	0.5	10	50	Restek 32008

Ar1232 Mix 2	Initial Conc.	Amount Used	Final Volume	Final Conc.	G1232WRK2_
Ar1232 Int (G_1232_INT_)	50	0.1	50	0.10	
SurrInt (GSURINT_)	5	0.1	50	0.010	DCB 2x higher

Aroclor 1242 Intermediate	Initial Conc ug/ml	Amount Used mls	Final Volume mls	Final Conc. ug/ml	G_1242_INT_
Ar1242 (G_1242_STK_)	1000	0.5	10	50	Restek 32009

Ar1242 Mix 2	Initial Conc.	Amount Used	Final Volume	Final Conc.	G1242WRK2_
Ar1242 Int (G_1242_INT_)	50	0.1	50	0.10	
SurrInt (GSURINT_)	5	0.1	50	0.010	DCB 2x higher

Aroclor 1248 Intermediate	Initial Conc ug/ml	Amount Used mls	Final Volume mls	Final Conc. ug/ml	G_1248_INT_
Ar1248 (G_1248_STK_)	1000	0.5	10	50	Restek 32010

Ar1248 Mix 2	Initial Conc.	Amount Used	Final Volume	Final Conc.	G1248WRK2_
Ar1248 Int (G_1248_INT_)	50	0.1	50	0.10	

SurrInt (GSURINT_)	5	0.1	50	0.010	DCB 2x higher

Aroclor 1254 Intermediate	Initial Conc ug/ml	Amount Used mls	Final Volume mls	Final Conc. ug/ml	G_1254_INT_
Ar1254 (G_1254_STK_)	1000	0.5	10	50	Restek 32011

Ar1254 Mix 2	Initial Conc.	Amount Used	Final Volume	Final Conc.	G1254WRK2_
Ar1254 Int (G_1254_INT_)	50	0.1	50	0.10	
SurrInt (GSURINT_)	5	0.1	50	0.010	DCB 2x higher

Aroclor 1262 Intermediate	Initial Conc ug/ml	Amount Used mls	Final Volume mls	Final Conc. ug/ml	G_1262_INT_
Ar1262 (G_1262_STK_)	1000	0.5	10	50	Restek 32409

Ar1262 Mix 2	Initial Conc.	Amount Used	Final Volume	Final Conc.	G1262WRK2_
Ar1262 Int (G_1262_INT_)	50	0.1	50	0.10	
SurrInt (GSURINT_)	5	0.1	50	0.010	DCB 2x higher

Aroclor 1268 Intermediate	Initial Conc ug/ml	Amount Used mls	Final Volume mls	Final Conc. ug/ml	G_1268_INT_
Ar1268 (G_1268_STK_)	1000	0.5	10	50	Restek 32410

Ar1268 Mix 2	Initial Conc.	Amount Used	Final Volume	Final Conc.	G1268WRK2_
Ar1268 Int (G_1268_INT_)	50	0.1	50	0.10	
SurrInt (GSURINT_)	5	0.1	50	0.010	DCB 2x higher

TCX/DCB Intermediate	Initial Conc ug/ml	Amount Used mls	Final Volume mls	Final Conc. ug/ml	GSURINT_
TCX (G_TCX_STK_)	200	0.625	25	5	
DCB (G_DCB_STK_)	200	1.25	25	10	

TABLE 2.0
MULTI-COMPONENT
CALIBRATION CONCENTRATIONS, UG/ML

Analyte	Mix 0.5	Mix 1	Mix 2	Mix 3	Mix 4	Mix 5
Aroclor-1016/1260	0.025	0.05	0.10	0.20	0.40	0.80
Aroclor-1221			0.10			
Aroclor-1232			0.10			
Aroclor-1242			0.10			
Aroclor-1248			0.10			
Aroclor-1254			0.10			
Aroclor-1262			0.10			
Aroclor-1268			0.10			
TCX	0.0025	0.005	0.01	0.025	0.05	0.10
DCB	0.005	0.01	0.02	0.050	0.10	0.20

TABLE 3.0
SURROGATE MIX, UG/ML

Surrogate	Stock	Mix for Extractions (added to each sample and spike)
TCX	2.0	0.20
DCB	2.0	0.20

TABLE 3.1
SURROGATE RECOVERY LIMITS

Surrogate recovery limits are entered into the Laboratory Information Management system.

TABLE 4.0
AROCLOR QC CHECK SOLUTION (UG/ML)

Compound	Stock	STD Mix for Extractions (Added to each Aroclor QC Check Sample)	Water Control Limits	Soil Control Limits
Aroclor-1016	1000	5	See LIMS for updated limits	See LIMS for updated limits
Aroclor-1260	1000	5	See LIMS for updated limits	See LIMS for updated limits

TABLE 4.1
AROCLOR MATRIX SPIKE SOLUTION (UG/ML)

Compound	Stock	STD Mix for Extractions (Added to each Aroclor Matrix Spike Sample)	Water Control Limits	Soil Control Limits
Aroclor – 1016	1000	2.0	See LIMS for Updated limits	See LIMS for Updated limits
Aroclor-1260	1000	2.0	See LIMS for updated limits	See LIMS for updated limits

TABLE 5.0
PRACTICAL QUANTITATION LIMITS (PQL)

Analyte	Quantitation Limits, Water ug/L	Quantitation Limits, Soil ug/Kg
Aroclor-1016	0.50	17
Aroclor-1221	0.50	17
Aroclor-1232	0.50	17
Aroclor-1242	0.50	17
Aroclor-1248	0.50	17
Aroclor-1254	0.50	17
Aroclor-1260	0.50	17
Aroclor-1262 *	0.50	17
Aroclor-1268 *	0.50	17

- Additional compounds require special client request.

FIGURE 1.0
LIMS Organics Data Review Checklist

First Level	Second Level
1 -Prep Batch created (VOA)	
-TCLP's/MeOH Preps	
2 -Check Method	
3 -Dilutions appropriate	
4 -Initial Calibration *	
- Check Manual Integrations	ReCheck
- Check % RSD's and Minimum RRFs	ReCheck
- Check minimum level req. (avg/linear/quad)	ReCheck
- Check for missing levels	ReCheck
- Check all analytes present	ReCheck
*** - ICAL checked – empty folder created in Target batch to signify that the curve has been reviewed.**	
5 -Continuing Calibration	
- Check Manual Integrations	ReCheck
- Check % differences and Minimum RRFs	ReCheck
- Check all analytes present	ReCheck
*** - CCAL checked – empty folder created in Target batch to signify that the ccv has been reviewed.**	
6- Q-Editing (including Manual integrations)	**Check**
7 - TIC's called	**Recheck**
8 - AD Worksheet (VOA) Init/FV	**Recheck**
9 - AD Reagents (VOA) codes/amts	**Recheck**
10 - Sample Results	**Recheck**
-RL's look correct	
-Flagging correct	
-RA/RE/DL suffixes as needed	
11 - GC Dual Column criteria applied	**Recheck**
-P/A sample results	
-P/S surrogates	
12 - Surrogate recoveries present/flagged	**Recheck**
13 - TIC's reported correctly	**Recheck**
14 - QC linking correct	**Recheck**
15 - NCM's created (record batch#)	**Read**
	JOB LOCKED (PM Desktop)

QAF04303.ct (last update 10/14/09)

Appendix C-5

Supporting Information for TAL Metals

Metals (ICP/MS)	6020

Analyte Description	CAS Number
Silver	7440-22-4
Aluminum	7429-90-5
Arsenic	7440-38-2
Barium	7440-39-3
Beryllium	7440-41-7
Calcium	7440-70-2
Cadmium	7440-43-9
Cobalt	7440-48-4
Chromium	7440-47-3
Copper	7440-50-8
Iron	7439-89-6
Potassium	7440-09-7
Magnesium	7439-95-4
Manganese	7439-96-5
Sodium	7440-23-5
Nickel	7440-02-0
Lead	7439-92-1
Antimony	7440-36-0
Selenium	7782-49-2
Thallium	7440-28-0
Vanadium	7440-62-2
Zinc	7440-66-6

Mercury (CVAA)	7471A

Analyte Description	CAS Number
Mercury	7439-97-6

Title: SOP for Metals Digestion- Aqueous
[Methods SW846 3010A(prep)]

Approvals (Signature/Date):

10/14/09		10/15 /09	
Technical Manager	Date	Health & Safety Manager/Coordinator	Date
10/13/09		10/26/09	
Quality Assurance Manager	Date	Laboratory Director	Date

This SOP was previously identified as CT-MES-9_11.

Facility Distribution No. Electronic **Distributed To: Facility Intranet**

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1.0 APPROVALS

The approval of the Laboratory Director, the Facility QA Manager, Group Leader and Health and Safety Officer signify that the document has been prepared in accordance with the format requirements that have been established in this document. In addition, if the document is a Method SOP the signature signifies that it meets the specified requirements.

2.0 SCOPE AND APPLICATION

2.1 This digestion procedure is used for the preparation of aqueous samples for analysis by ICP and ICP-MS, for the metals listed below. The procedure is used to determine total metals for unfiltered samples that are HNO3 preserved to a pH less than 2 and dissolved metals for samples that are filtered prior to acid preparation.

2.2

Aluminum	Lead	Titanium
Antimony	Magnesium	Vanadium
Arsenic	Manganese	Zinc
Barium	Molybdenum	Boron
Beryllium	Nickel	Strontium
Cadmium	Potassium	Silicon
Calcium	Selenium	Zirconium
Chromium	Silver	Lithium
Cobalt	Sodium	Uranium
Copper	Thallium	Thorium
Iron	Tin	Sulfur

2.3 The document control number for the SOP is CT-MES-9, rev 12.

3.0 TERMS AND DEFINITIONS

3.1 There are many definitions used with in the laboratory, which may be generic to all laboratory analyses, or more specific for certain methods. For the most recent terms and definitions used with in the laboratory, reference the SOP for Terms and Definitions.

4.0 SUMMARY OF METHOD

4.1 A mixture of HNO_3 and material to be analyzed is heated in a 100 mL tube until the volume is reduced to 5-10 mL. After cooling, the digestion is refluxed with HCl and brought to 50 ml. If the sample should go to dryness at any time during the digestion, it must be discarded and the sample reprepared.

4.2 This method is based on EPA method 3010A.

4.3 This SOP is equivalent to EPA Method 3010A except for the following differences: The final volumes for 6010B are brought to 50ml, and 6020 are brought to 500ml equivalent.

5.0 INTERFERENCES

5.1 Samples of varying matrices provide different interferences. A list of quality controls are performed to evaluate these and are described in section 10.0.

5.2 Contamination can be a problem during sample preparation and must be minimized. Clean all work surfaces daily with DI water including bench tops, hood and hot plates. Take care not to cross contaminate samples during digestion by making sure that none of the samples boil or splatter.

6.0 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.

6.1 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.

6.2 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.
1 – Always add acid to water to prevent violent reactions.			
2 – Exposure limit refers to the OSHA regulatory exposure limit.			

7.0 SAMPLE CONTAINERS, COLLECTION AND PRESERVATION

7.1 Samples are collected in 500 mL plastic bottles acidified to pH of < 2 with nitric acid.

7.2 Samples that are received unpreserved or have insufficient preservation must be acidified to pH <2 with nitric acid, mixed, and held for 16 hours. The pH will be verified at <2 prior to withdrawing an aliquot for processing.

7.3 Samples are stable when digestion is complete and need no preservation. Sample bottles are not to be reused.

7.4 Holding time is 180 days.

8.0 APPARATUS AND MATERIALS

8.1 Equipment

8.1.1 Ventilation Hood
8.1.2 Environmental Express Hot Block Digestor, model SC100
8.1.3 Environmental Express certified digestion tubes
8.1.4 Eppendorf pipette, 1000uL, 100uL

9.0 REAGENTS AND STANDARD PREPARATION

9.1 Water used in the preparation of standards or reagents must have a specific conductivity of less than 1 µmho/cm (or specific resistivity of greater than 1.0 megohm-cm) at 25°C.

9.2 Concentrated Nitric Acid, Trace Grade

9.3 Concentrated Hydrochloric acid, Trace Grade

9.4 Sample matrix spike stocks are purchased from Inorganic Ventures or Elements. For matrix spike analysis, add appropriate amount of each spike stock directly to the designated QC sample. Expiration date is one year from date of manufacture.

The following solutions comprise the Matrix Spike (MS)

Spike Solution(Tals based code)	Element	Concentration (ug/mL)
CLPP-SPK-1 (MSPK1_)	Ag, Be	50
	Cr	200
	Cu	250
	Co, Mg, Ni, Zn, V	500
	Fe	1000
	Al, Ba	2000
CLPP-SPK-5 (MSPK5_)	Pb	20
	As	40
	Cd, Se, Tl	50
	Sb	100
Cal-2 (MCAL2_)	B, Mo, Sb, Si, Sn, Ti, Zr	1000
Earth Metals (MEM_)	Ca, K, Mg, Na	5000
*Uranium (MU_)	U	100
*Thorium (MTH_)	Th	100
*Lithium (MLi_)	Li	100

* Spiked only when element is specifically requested.

9.5 Laboratory Control Sample Stock purchased from Inorganic Venture or Elements. Expiration date is one year from date of manufacture.

Spike Solution(Tals based code)	Element	Concentration (ug/mL)
LCS Stock 3 (MLCS3_)	Be	100
	Mn	200
	Ag, Ba, Cd, Co, Cr, Cu, Ni, V, Zn	300
	Se	500
	As, Pb, Tl	1000
	K	20000
LCS Stock 4 (MLCS4_)	Na	2500
	Al	3000
	Mg	7500
	Fe	12500
	Ca	15000

Cal-2 (MCAL2_)	B, Mo, Sb, Si, Sn, Ti, Zr	1000
Na Stock (MNA_)	Na	10000
*Uranium (MU_)	U	100
*Thorium (MTH_)	Th	100
*Lithium (MLi_)	Li	100

* Spiked only when element is specifically requested.

9.6 LCS Intermediate Solution – Used for both Methods 6010B and 6020.

	Initial Conc ug/L	Amount Used mls	Final Volume mls	Final Conc. ug/L
Na Stock (MNA_)	10000	2.5	1000	25
LCS Stock 4 (MLCS4_)	2500-15000	2.0	1000	5.0 - 30
LCS Stock 3 (MLCS3_)	100-20000	1.0	1000	0.1 - 20
Cal 2 (MCAL2_)	1000	1.0	1000	1.0

Stock standards are added to a 1000ml volumetric containing Reagent water, 13ml Nitric acid and 13ml HCl.

A 50ml aliquot of the LCS intermediate solution is used as the LCS spike.

9.6.1 Additional Element Spikes – LCS

When Uranium, Thorium and Lithium are requested elements, the following spike amounts shall be added to the 50ml aliquot for the analysis method noted:

LCS solution (Tals reagent code)	Spike added (uls) – For Method 6020 Analysis
Uranium Spike (MU_)	100
Thorium Spike (MTH_)	100
Lithium Spike (Mli_)	100

9.7 Matrix Spike preparation – the following solutions are added into one digestion cup containing sample to produce the MS.

Matrix Spike	Method 6010B Spike Added (uls)	Method 6020 Spike Added (uls)
CLPP-SPK-1 (MSPK1_)	50	50
CLPP-SPK-5 (MSPKs_)	100	50
Earth Metals Spike (MEM_)	100	100
Cal 2 (MCAL2_)	50	50

9.7.1 Additional Element Spikes – MS

When Uranium and Thorium are requested elements, the following spike amounts shall be added to the digestion cup in addition to the other spiking solutions:

MS solution (Tals reagent code)	Spike added (uls) – For Method 6020 Analysis
Uranium Spike (MU_)	100
Thorium Spike (MTH_)	100
Lithium Spike (MLi_)	100

10.0 CALIBRATION

10.1 Eppendorf Pipettes

Eppendorf pipettes must be calibrated quarterly at similar volumes used to dispense standards and reagents. Refer to SOP for Pipette Calibration for proper procedures and requirements.

10.2 Hot Block Digestion Unit (SC100)

The temperature of the digester must be monitored during each batch. This is done by filling a digestion tube ¾ full with reagent water and placing it in a digestion slot. A calibrated thermometer is then placed in the tube and monitored throughout the digestion. The temperature is recorded at the beginning and end of the cycle in TALS in the batch comments section.

10.3 Digestion Tube Calibration

Tare the weight of a digestion tube from a newly opened lot of tubes. Fill the tube with reagent water that is room temperature to the 100mL mark and weigh. Record that weight on the certificate supplied with the tubes. Repeat this procedure for the 50 mL mark. Weights should be within 2 percent of the respective volume amounts. Certificates are compiled in a separate folder. All volumes to be used (initial and final volumes), shall be verified by either the analyst or the manufacturer.

11.0 QUALITY CONTROL

11.1 Preparation blanks are processed one per 20 samples through all steps of the digestion and are performed to check for contamination

11.2 A laboratory control sample is prepared one per 20 samples to check that no analyte is lost or gained during the batch preparation.

11.3 A duplicate sample is prepared one per 20 samples. The RPD is calculated from the original sample.

11.4 A sample spike is also performed one per 20 samples with each batch.

12.0 SAMPLE PREPARATION

12.1 Hot Block Digestion

- Accurately transfer 100 ml of a well mixed sample or an aliquot diluted to 100 ml to a 125 mL digestion tube. For duplicate and matrix spikes shake the sample between each subsequent pourings.
  - TCLP samples – initial volume used is 20ml.
  - SPLP samples – initial volume used is 100ml.
- QC samples
  - MS - Add stock spikes to the QC sample as described in section 9.7.
  - LCS - treat the LCS solution like a sample using an aliquot of the LCS intermediate solution prepared in section 9.6.
- Add 2 mL of concentrated HNO3 to the tube and place it in the hot block digester.
- Heat the sample until the volume is reduced to 5-10 mL.
- Make certain that the sample does not boil and that no portion of the bottom of the tube is allowed to go dry. Discard and reprepare any sample that goes dry.
- Add additional acid (conc. HNO3), until the digestion appears complete (generally indicated when the digestate is light in color or does not change in appearance with continued refluxing).
- Cool the tube and add 1 mL of concentrated HCl.
- Return the tube to the hot block and heat for 15 minutes.
- Remove the tube from the heat source and while still warm, rinse down the sides of the tube with reagent water.
- Once cool, add reagent water to the 50 mL mark, close cap and mix by inverting several times. The sample is now ready for analysis.

Digestates that are going to be used for Method 6010B and Method 6020 analysis, will be handled prior to analysis in the following manner.

6010B - Digestates at 50mls, are analyzed as is(unless samples require filtration). The Final volume entered into the prep batch is 50mls.

6020 – Digestates at 50mls, are diluted 1:10 prior to analysis. The Final volume entered into the prep batch is 500mls to account for this dilution factor. All samples and QC are treated the same.

13.0 CALCULATIONS

13.1 None.

14.0 ACCEPTANCE OF DATA

14.1 Acceptance of the data is described in section 10.0

15.0 REPORTING OF RESULTS

15.1 Samples are entered into the TALS system. All appropriate sections must be completed. The data prep page must be kept with digestates.

15.2 A batch system is used. Only 20 samples are allowed in a batch. Each batch should have a duplicate sample, a spiked sample, spiked duplicate, a prep-blank and a laboratory control sample. All of these

samples should be indicated in the system logbook page.

15.3 Record matrix, initial volume and final volume.

15.4 Record lot number of all solutions and reagents added.

15.5 Record in comment column if sample is diluted, concentrated, limited, preserved, reprepped or other changes.

16.0 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). Employees must abide by the policies in Section 13 of the Corporate Safety Manual for "Waste Management and Pollution Prevention."

16.1 Pollution prevention is the reduction and/or elimination of wastes or the toxicity of wastes at the point of generation. The laboratory strives to keep all wastes at a minimum through a variety of techniques. These include the following.

16.1.1 Inventory control: Rotate stocks (first in, first out) and only purchase reasonable amounts of chemicals. Do not purchase excessive quantities, which will be discarded as wastes at some future point.

16.1.2 Material Handling and Storage: Keep containers properly labeled. Keep solvents covered. Store and handle all chemicals, reagents, standards, etc. to prevent contamination and degradation.

16.1.3 Personnel: All staff must be trained in the proper handling and disposal of waste.

16.1.4 Waste Reduction; Reduce the volume of waste generated wherever possible.

16.1.5 Chemical/material substitution: Use less toxic chemicals whenever possible. Avoid the use of chromic acid (Chromerge) solutions.

17.0 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 Tracking and Collection of Hazardous Waste SOP. The following waste streams are produced when this method is carried out.

17.1 All waste shall be managed in accordance with all state and federal requirements. See the TESTAMERICA-CT Hazardous Waste Management Plan.

17.2 All personnel who handle or generate waste must be trained within six months of employment in proper waste handling and requirements.

17.3 All acid waste is disposed of into a 55 gallon polyethylene drum marked acid waste.

18.0 SUPPLEMENTAL DOCUMENTS

18.1 **None**

19.0 REFERENCES

19.1 Method 3010A, Acid Digestion of Aqueous Samples and Extracts for Total Metals for Analysis by FLAA or ICP Spectroscopy, July 1992.

19.2 Employee Chemical Safety Handbook

19.3 EPA Method 200.7, Determination of Metals and Trace Elements in Water and Wastes by Inductively Coupled Plasma Atomic emission spectrometry. Revision 4.4. 1994.

19.4 EPA Method 200.8, Determination of Trace Elements in Water and Wastes by Inductively Coupled Plasma Mass Spectrometry. Rev 5.4, 1994.

20.0 SUBSTANTIVE REVISIONS

20.1 Changed name to STL from IEA. Updated method reference

20.2 Added section 3 – Terms and definitions, 16 & 17 for waste management and pollution prevention, renumber other sections: 02/12/00.

20.3 Added the Hot block digestion procedure in section 12; added reference to labnet in section added calibration of Eppendorf and Hotblock digestor in section10.0: 03/31/03.

20.4 Updated section 6 to include the corporate health and safety SOP: 01/28/2004.

20.5 Added spike and LCS tables to section 9.0,: 04/01/2004

20.6 Section 2.1: removed digestion of dissolved metals; Section 4.3: added addressed antimony and silver method deviation 03/18/2005

20.7 Modified Section 12.1, HNO3 final volume 5%, HCL 1%; 09/19/2007.

20.8 Added new TestAmerica SOP header and control number, changed name, added section 10.3 – digestion tube calibration 01/16/08.

20.9 Added 200.7 and 200.8 methods to SOP. 2.2 – Added 4 elements. Added to safety, pollution control and waste management sections 5-5-09.

20.10 Added section 7.2 to address samples received with insufficient preservation. Updated sect 9.1 conductivity requirements. Sect 10.1 updated Eppendorf requirements. Updated section 9.4 – 9.7.1

for spiking solutions and QC preparation. Updated 12.1 to include final volume differences, additional acid during digestion. Added sect 4.3 method deviations. 10/12/09.

Title: SOP for Metals Digestion -Soils and Wipes
[Method SW846 3050B]

Approvals (Signature/Date):

Technical Manager	10/14/09 Date	Health & Safety Manager/Coordinator	10/15 /09 Date
Quality Assurance Manager	10/13/09 Date	Laboratory Director	10/26/09 Date

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2.0 SCOPE AND APPLICATION

2.1 This document is used to specifically describe the digestion of soils, sediments and sludge samples for analysis by ICP-MS and ICP-OES. Samples prepared by these methods may be analyzed for the listed metals. Users should be aware that this digestion is not a total digestion for most samples. If absolute total digestion is required, then alternate methods should be utilized.

2.2 Elements

Aluminum	Magnesium	Arsenic
Antimony	Manganese	Selenium
Barium	Molybdenum	Thallium
Beryllium	Nickel	Boron
Cadmium	Potassium	Strontium
Calcium	Silver	Zirconium
Chromium	Sodium	Thorium
Cobalt	Tin	Silicon
Copper	Titanium	Sulfur
Iron	Vanadium	Lithium
Lead	Zinc	Uranium

2.3 The document control number for this SOP is CT-MES-10, rev 12.

3.0 TERMS AND DEFINITIONS

3.1 There are many definitions used with in the laboratory, which may be generic to all laboratory analyses, or more specific for certain methods. For the most recent terms and definitions used with in the laboratory, reference the SOP for Terms and Definitions.

4.0 SUMMARY OF METHOD

4.1 A representative 1 – 2.5 gm (wet weight) of soil sample is digested in nitric acid and hydrogen peroxide. For a wipe, the whole wipe sample is digested in nitric acid and hydrogen peroxide. The digestate is then refluxed with hydrochloric acid and brought to 50 ml final volume with deionized water.

4.2 This method is based on EPA method 3050B.

4.3 This SOP is equivalent to EPA Method 3050B except for the following differences: HCL is added to digestates for samples that are to be analyzed by ICP-MS. Current technology and the dilution performed upon samples allows this reagent to be used. 2mls of HCL is added to digestates that are to be analyzed by ICP.

5.0 INTERFERENCES

5.1 Samples of varying matrices provide different interferences. A list of quality controls are performed to evaluate these and are described in section 11.0.

5.2 Contamination can be a problem during sample preparation and must be minimized. Clean all work surfaces daily with DI water including bench-tops, hood and hot blocks. Take care not to cross contaminate samples during digestion by making sure that none of the samples boil or splatter.

6.0 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.

6.1 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.

6.2 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.
1 – Always add acid to water to prevent violent reactions.			
2 – Exposure limit refers to the OSHA regulatory exposure limit.			

7.0 SAMPLE CONTAINERS, COLLECTION AND PRESERVATION

7.1 Samples are collected in glass soil jars and refrigerated upon receipt.

7.2 Samples are stable when digestion is complete and need no preservation.

7.3 Holding time is 180 days.

8.0 APPARATUS AND MATERIALS

8.1 50 mL graduated digestion tubes

8.2 Environmental Express Hot Block Digestion Unit

8.3 Calibrated Thermometer

8.4 Eppendorf pipets (Brinkmann 2000 series, variable, 100uL and 1000uL).

8.5 Sledge hammer

8.6 Balance calibration weights

9.0 REAGENTS AND STANDARD PREPARATION

9.1 Water used in the preparation of standards or reagents must have a specific conductivity of less than 1 µmho/cm (or specific resistivity of greater than 1.0 megohm-cm) at 25°C.

9.2 Concentrated Nitric Acid, Trace Grade

9.3 Concentrated Hydrochloric Acid, Trace Grade

9.4 30% Hydrogen Peroxide

9.5 Sample matrix spike stocks are purchased from Inorganic Ventures or Elements. For matrix spike analysis, add appropriate amount of each spike stock directly to the designated QC sample. Expiration date is one year from date of manufacture.

The following solutions comprise the Matrix Spike (MS)

Spike Solution(Tals based code)	Element	Concentration (ug/mL)
CLPP-SPK-1 (MSPK1_)	Ag, Be	50
	Cr	200
	Cu	250
	Co, Mg, Ni, Zn, V	500
	Fe	1000
	Al, Ba	2000
CLPP-SPK-5 (MSPK5_)	Pb	20
	As	40
	Cd, Se, Tl	50
	Sb	100
Cal-2 (MCAL2_)	B, Mo, Sb, Si, Sn, Ti, Zr	1000
Earth Metals (MEM_)	Ca, K, Mg, Na	5000
*Uranium (MU_)	U	100
*Thorium (MTH_)	Th	100
*Lithium (MLi_)	Li	100

* Spiked only when element is specifically requested.

9.6 Laboratory Control Sample Stock purchased from Inorganic Venture or Elements. Expiration date is one year from date of manufacture.

Spike Solution(Tals based code)	Element	Concentration (ug/mL)
LCS Stock 3 (MLCS3_)	Be	100
	Mn	200
	Ag, Ba, Cd, Co, Cr, Cu, Ni, V, Zn	300
	Se	500
	As, Pb, Tl	1000
	K	20000
LCS Stock 4 (MLCS4_)	Na	2500
	Al	3000
	Mg	7500
	Fe	12500
	Ca	15000
Cal-2 (MCAL2_)	B, Mo, Sb, Si, Sn, Ti, Zr	1000

Na Stock (MNA_)	Na	10000
*Uranium (MU_)	U	100
*Thorium (MTH_)	Th	100
*Lithium (MLi_)	Li	100

* Spiked only when element is specifically requested.

9.7 LCS preparation. – the following solutions are spiked into a soil matrix (glass beads), to produce the LCS.

LCS	Method 6010B Spike Added (uls)	Method 6020 Spike Added (uls)
Na Stock (MNA_)	625	625
LCS Stock 4 (MLCS4_)	500	500
LCS Stock 3 (MLCS3_)	250	250
Cal 2 (MCAL2_)	250	250

9.7.1 Additional Element Spikes – LCS

When Uranium and Thorium are requested elements, the following spike amounts shall be added to the digestion cup in addition to the other spiking solutions:

LCS solution (Tals reagent code)	Spike added (uls) – For Method 6020 Analysis
Uranium Spike (MU_)	200
Thorium Spike (MTH_)	200
Lithium Spike (MLi_)	200

9.8 Matrix Spike preparation – the following solutions are added into one digestion cup containing sample to produce the MS.

Matrix Spike	Method 6010B Spike Added (uls)	Method 6020 Spike Added (uls)
CLPP-SPK-1 (MSPK1_)	50	50
CLPP-SPK-5 (MSPKs_)	200	100
Earth Metals Spike (MEM_)	100	100
Cal 2 (MCAL2_)	50	50

9.8.1 Additional Element Spikes – MS

When Uranium and Thorium are requested elements, the following spike amounts shall be added to the digestion cup in addition to the other spiking solutions:

MS solution (Tals reagent code)	Spike added (uls) – For Method 6020 Analysis

Uranium Spike (MU_)	200
Thorium Spike (MTH_)	200
Lithium Spike (MLi_)	200

10.0 CALIBRATION

10.1 The balance shall be calibrated each day used. This is done by first zeroing the balance and then placing a known standard weight of 1.0 grams on the balance and allowing the balance to stabilize. Enter the amount of the weight and press enter. The balance is now calibrated.

10.2 Eppendorf Pipettes (Brinkmann 2000 series, variable, 100uL, 1000uL.)

Eppendorf pipettes must be calibrated quarterly at similar volumes used to dispense standards and reagents. Refer to SOP for Pipette Calibration for proper procedures and requirements.

10.3 Hot Block Digestion Unit

The temperature of the digestor must be monitored during each batch. This is done by filling a digestion tube ¾ full with reagent water and placing it in a digestion slot. A calibrated thermometer is then placed in the tube and monitored throughout the digestion. The temperature is recorded at the beginning and end of the cycle in TALS.

10.4 Digestion Tube Calibration:

Tare the weight of a digestion tube from a newly opened lot of tubes. Fill the tube with reagent water that is room temperature to the 50mL mark and weigh. Record the weight on the certificate supplied with the tubes. Weights should be within 2 percent of the respective volume amounts. Certificates are compiled in a separate folder. All volumes to be used (initial and final volumes), shall be verified by either the analyst or the manufacturer.

11.0 QUALITY CONTROL

11.1 Preparation blanks are carried throughout the entire sample and analytical process to check for contamination

11.2 A lab control sample is prepared one per 20 samples to check that no analyte is lost or gained during the batch preparation.

11.3 A duplicate sample is prepared one per 20 samples and the RPD is calculated from the original sample.

11.4 A sample spike is prepared one per 20 samples and the %Rec is calculated from the original sample.

11.5 QC related Activities

Prep. Blank	Every Batch not to exceed 20 samples
Lab Control Sample	Every Batch not to exceed 20 samples
Sample Spike	Every Batch not to exceed 20 samples
Duplicate	Every Batch not to exceed 20 samples

12.0 SAMPLE PREPARATION

12.1 For soil samples, mix the sample thoroughly to achieve homogeneity, reference the SOP for homogenization and compositing.

- For soils, place 1 – 2.5 g of wet sample weighed to the nearest 0.01g in the bottom of a 50 ml disposable digestion tube, making sure that little or no sample is stuck to the sides of the tube. For wipes, place the whole wipe in the bottom of a 50ml disposable digestion tube, making sure that little or no sample is stuck to the sides of the tube.
- QC samples
  - MS - Add stock spikes to the QC sample as described in section 9.8.
  - LCS - Add stock spikes to the QC sample as described in section 9.7.
- Add approximately 5 ml of DI water followed by 5 ml of concentrated nitric acid. Swirl to mix the slurry.
- Heat the sample in a block digester at 95° C ± 5 °C and reflux for 10 minutes without boiling.
- Allow the sample to cool, add 5 ml of concentrated nitric acid.
- Heat for an additional 30 minutes. Do not allow the volume to be reduced to less than 5 ml.
- Cool the sample and add 2 ml of DI water and 3 ml of 30% hydrogen peroxide.
- Return the tube to the heat source for warming to start the peroxide reaction. Care must be taken to ensure that losses do not occur due to excessive vigorous effervescence. Heat until effervescence subsides, and cool the tube. Continue to add peroxide in 1 ml aliquots with warming until the effervescence is minimal or until the general sample appearance is unchanged. Do not add more than a total of 10 ml of peroxide. When adding additional amounts of peroxide, always add the same amount to the preparation blank and laboratory control sample.
- Return the hydrogen peroxide digestate to the heat source for two hours or until the digestate reaches approximately 5 mL, making sure that the sample does not boil or splatter.
- Cool the sample, add 2 ml of concentrated HCl, return the tube to the digester.
- Heat for an additional 15 minutes.
- After the sample has cooled add DI water to the 50 ml mark. Cap tightly and shake well. The sample is now ready for analysis.

Digestates that are going to be used for Method 6010B and Method 6020 analysis, will be diluted prior to analysis in the following manner.

6010B - Digestates at 50mls, are diluted 1:5 for soil samples. The Final volume entered into the prep batch is 250mls.

6020 – Digestates at 50mls, are diluted 1:20 for soil Samples. The Final volume entered into the prep batch is 1000mls to account for this dilution factor. All samples and QC are treated the same.

Note: For solid samples(such as concrete) requiring particle size reduction in order to properly perform the

method, a sledge hammer shall be used. Before this equipment is used, the client's consent shall be obtained and informed of the potential for contamination from the equipment used. The necessity of reducing sample size shall be noted in an NCM associated with the particular sample.

13.0 CALCULATIONS

13.1 None.

14.0 ACCEPTANCE OF DATA

14.1 N/A

15.0 REPORTING RESULTS

15.1 Samples are entered into TALS with all prep information.

15.2 A batch system is used. Only 20 samples are allowed in a batch. Each batch should have a duplicate sample, a spiked sample, a blank sample and a laboratory control sample. All of these samples should be indicated in TALS.

15.3 Record weight (to nearest 0.01 gram), matrix, initial volume, color and clarity and final volume for each individual sample.

15.4 Record lot number of all solutions and reagents added.

15.5 Record in comment column if sample is diluted, concentrated, limited, or other changes.

16.0 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). Employees must abide by the policies in Section 13 of the Corporate Safety Manual for "Waste Management and Pollution Prevention."

16.1 Pollution prevention is the reduction and/or elimination of wastes or the toxicity of wastes at the point of generation. The laboratory strives to keep all wastes at a minimum through a variety of techniques. These include the following.

16.1.1 Inventory control: Rotate stocks (first in, first out) and only purchase reasonable amounts of chemicals. Do not purchase excessive quantities, which will be discarded as wastes at some future point.

16.1.2 Material Handling and Storage: Keep containers properly labeled. Keep solvents covered. Store and handle all chemicals, reagents, standards, etc. to prevent contamination and degradation.

16.1.3 Personnel: All staff must be trained in the proper handling and disposal of waste.

16.1.4 Waste Reduction: Reduce the volume of waste generated wherever possible.

16.1.5 Chemical/material substitution: Use less toxic chemicals whenever possible. Avoid the use of chromic acid (Chromerge) solutions.

17.0 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 Tracking and Collection of Hazardous Waste SOP. The following waste streams are produced when this method is carried out.

17.1 All waste shall be managed in accordance with all state and federal requirements. See the Testamerica-CT Hazardous Waste Management Plan.

17.2 All personnel who handle or generate waste must be trained within six months of employment in proper waste handling and requirements.

17.3 All acid waste is disposed of into a 55 gallon polyethylene drum marked acid waste.

18.0 SUPPLEMENTAL DOCUMENTS

18.1 **None**

19.0 REFERENCES

19.1 Method 3050B, SW-846, 3rd ed., Test Methods for Evaluating Solid Waste, EPA Office of Solid Waste and Emergency Response, Dec. 1996.

19.3 Employee Chemical Safety Handbook

20.0 SUBSTANTIVE REVISIONS

20.1 Original issue - 4/21/93

20.2 Modified sections 3.1 and 11.5 to include As, Cd, Pb, Se, and Tl to ICAP analysis for trace ICP. Changed name to STL from IEA. Added section 3 – Terms and definitions, 16 & 17 for waste management and pollution prevention, renumber other sections; 02/12/00.

20.3 Modified sections 8.1 and section 12 to include the use of hot block March 20, 2001.

20.4 Updated for labnet in section 15; added Eppendorf and hotblock calibration in section 10; March 15, 2003.

20.5 Updated section 6 to include the corporate health and safety SOP January 28, 2004.

20.6 Added spiking concentration table to section 9.7, April 1, 2004.

20.7 Updated section 12.10: to include reference to SOP on sample homogenization; correct sample weight. Update section 12.11 and 12.21 to include nitric acid step. Update section 12.13 and 12.23

to include hydrogen peroxide step. Modify section 12.14 to change HCL digestion time from 10 minutes to 15 minutes. Modify section 12.24 to change final volume from 200 mL to 100 mL. Modify section 12.24 to change final volume from 100 mL to 50 mL. Removed section 12.2. Hotblock is no longer used. Added the use of AuCl3 as a stabilizer for Hg analysis by ICP-MS. Added new TestAmerica SOP header and control number, changed name, added section 10.4 – digestion tube calibration; 01/16/08.

20.8 Combined wipes into this SOP(sect's 4.2, 12.10). Sect 2.2. – added 4 elements. Added to Safety, pollution control and waste management sections 5/5/09.

20.9 Updated sect 9.1 reagent water requirements. Updated sect 10.2 for Eppendorf requirements. Sect 10.4 updated digestions tube verification requirements. Updated sect 9.4 – 9.8.1 for spiking solutions and QC preparation. Updated 12.1 to include final volume differences. Updated sect 12.1 to change from 4mls to 5mls nitric and 4mls to 5mls DI water. Sect 4.3 added for method deviations. Updated sect 9.7 to include glass beads. Sect 12.1 updated to remove mortar and pestle and replace with sledge hammer and its requirements prior to use. 10/12/09.

Title: SOP for ICP Metals Analysis
[Method SW846 6010B and EPA 200.7]

Approvals (Signature/Date):

	10/14/09		10/15 /09
Technical Manager	Date	Health & Safety Manager/Coordinator	Date
	10/13 /09		10/26/09
Quality Assurance Manager	Date	Laboratory Director	Date

This SOP was previously identified as CT-MES-20_8.

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 APPROVALS

The approval of the Laboratory Director, the Facility QA Manager, Group Leader and Health and Safety Officer signify that the document has been prepared in accordance with the format requirements that have been established in this document. In addition, if the document is a Method SOP the signature signifies that it meets the specified requirements.

2.0 SCOPE AND APPLICATION

2.1 Inductively coupled plasma-atomic emission spectrometry determines trace elements, including metals, in solution. The method is applicable to aqueous samples, TCLP and SPLP extracts, industrial wastes, soils, sludges and sediments. All samples are digested prior to analysis. Samples for method 6010 are digested using method 3010 for liquids (CT-MES-9) and 3050 for solids (CT-MES-10). Samples for method 200.7 are digested using the 200.7/200.8 digestion procedure (CT-MES-52).

2.2 Use of this method is restricted to spectroscopists who are knowledgeable in the correction of spectral, chemical, and physical interferences common to optical emission techniques.

2.3 The analytes determined using this technique are:

Aluminum	Cobalt	Potassium	Zinc
Antimony	Copper	Selenium	Boron
Arsenic	Iron	Silver	Silicon
Barium	Lead	Sodium	Strontium
Beryllium	Magnesium	Tin	
Cadmium	Manganese	Titanium	
Calcium	Molybdenum	Thallium	
Chromium	Nickel	Vanadium	

2.4 The document control number for this SOP is CT-MES-20, Rev. 9.

3.0 TERMS AND DEFINITIONS

3.1 There are many definitions used with in the laboratory, which may be generic to all laboratory analyses, or more specific for certain methods. For the most recent terms and definitions used with in the laboratory, reference the SOP for Terms and Definitions.

4.0 SUMMARY OF METHOD

4.1 This method is used to analyze samples that have been previously digested for ICP analysis. Samples are introduced into a cross flow nebulizer and spray chamber using a peristaltic pump. Element specific emission spectra are produced by a radio frequency inductively coupled plasma. The spectra are dispersed by a grating spectrometer, and the intensities of the emission lines are measured. These intensities are compared to a standard curve and the concentration of each element is determined.

4.2 This method is based on SW846 method 6010B and EPA Method 200.7.

5.0 INTERFERENCES

Spectral interferences include overlaps from other elements, unresolved overlap of molecular band

spectra, background contribution from continuous or recombination phenomena and background from stray light or high concentration elements. This is minimized by using interelement correction factors. Multiplication factors are set up to compensate for positive and negative results for all of the other elements. These factors will vary slightly over time and will change when major maintenance is done on the instrument. To determine the appropriate location for off-line background correction, the user must scan the area on either side adjacent to the wavelength and record the apparent emission intensity from all other method analytes. This spectral information must be documented and kept on file. A computer routine must be used for automatic correction on all determinations. Tests to determine spectral interference must be done using analyte concentrations that will adequately describe the interference. These are set up by analyzing all of the elements in 2.3 individually at concentrations near the upper linear range. Background emission and stray light can be compensated for by subtracting the background emission determined by measurements adjacent to the analyte wavelength peak. To determine the appropriate location for off-line background correction scan the area on either side adjacent to the wavelength and record the apparent emission intensity from all other method analytes.

When interelement corrections are applied, their accuracy should be verified daily by analyzing spectral interference check solutions. All interelement spectral correction factors or multivariate correction matrices must be verified and updated every six months or when an instrumentation change, such as in the torch, nebulizer, injector, or plasma conditions occurs. Standard solution should be inspected to ensure that there is no contamination that may be perceived as a spectral interference.

Physical interferences are associated with sample viscosity or high dissolved solids. Viscosity is minimized by using a peristaltic pump, and high solids are overcome by sample dilution and/or addition of internal standard to all samples and standards.

Chemical interferences are not normally pronounced in ICP and can be minimized by selection of proper operating conditions.

6.0 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.

6.1 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.

6.2 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.
1 – Always add acid to water to prevent violent reactions.			
2 – Exposure limit refers to the OSHA regulatory exposure limit.			

7.0 SAMPLE CONTAINERS, COLLECTION AND PRESERVATION

7.1 Samples are stable when digestion is complete and need no preservation. Sample bottles are not to be reused.

7.2 Holding time is 180 days.

8.0 APPARATUS AND MATERIALS

8.1 Optical emission spectrometer

8.2 Mass Flow Controller

8.3 Peristaltic Pump

8.4 High purity argon, welders grade or better

8.5 Auto sampler

8.6 Personal Computer

8.7 100 and 1,000 uL variable Eppendorf pipettes

8.8 10 ml graduated serological pipettes

8.9 12 mL capacity polypropylene sample tubes (Borosilicate glass must not be used for the analysis of Boron or Silicon in order to avoid sample contamination.

9.0 REAGENTS AND STANDARD PREPARATION

9.1 Reagent water - ASTM Type II, 18 megohm, deionized

9.2 Trace grade nitric acid.

9.3 ICP stock standards: All standards are to be plasma grade and must come with certificates of analysis traceable to NIST standards. All analytes in the calibration verification standards must be from a different source than those found in the calibration standards. The following analyte combinations are to be used:

9.3.1 Calibration Standard 1: 100 ppm Ag, 1000 ppm each Al, As, Ba, Be, Cd, Cr, Co, Cu, Fe, Pb, Mn, Ni, Se, Sr, Tl, V and Zn.
9.3.2 Calibration Standard 2: 1000 ppm each Sb, B, Mo, Si, Sn, Ti and Zr.
9.3.3 Earth Metals Calibration Standard: 5000 ppm each Ca, Mg, K and Na.
9.3.4 1000 ppm Al Calibration Standard
9.3.5 1000 ppm Fe Calibration Standard
9.3.6 Calibration Verification Standard 1: 1000 ppm each Ca, Mg, K and Na, 500 ppm each Al, As, Ba, Be, Cd, Cr, Co, Cu, Fe, Pb, Mn, Ni, Se, Sr, Tl, V and Zn.
9.3.7 1000 ppm Al second source
9.3.8 1000 ppm Fe second source
9.3.9 10000 ppm Na
9.3.10 10000 ppm Ca
9.3.11 10000 ppm Mg
9.3.12 10000 ppm K
9.3.13 100 ppm Ag
9.3.14 Calibration Verification Standard 2: 500 ppm each Sb, B, Mo, Si and Ti.
9.3.15 Calibration Verification Standard 3: 500 ppm each Sn and Zr
9.3.16 CRI Standard: 500 ppm each Ca, Mg, K and Na, 20 ppm each Al and Ba, 10 ppm Fe, 6 ppm each Sb and Zn, 5 ppm each Co and V, 4 ppm Ni, 3.5 ppm Se, 2.5 ppm each Cu and Tl, 1.5 ppm each As and Mn, 1 ppm each Cr, Pb and Ag, 0.5 ppm each Be and Cd.
9.3.17 ICSA Standard: 5000 ppm each Ca, Mg, Al, and 2000 ppm Fe.
9.3.18 ICSAB Standard: 100 ppm each Cd, Ni and Zn, 60 ppm Sb, 50 ppm each Ba, Be, Cr, Co, Cu, Mn and V, 20 ppm Ag, 10 ppm each As and Tl and 5 ppm each Pb and Se.

9.4 Working Standards: All working standards are made in volumetric flasks with a final acid concentration of 5 % nitric. Standards that contain Boron or Silicon must be stored in plastic bottles.

9.4.1 Standard 1: Reagent water contained in a 1L plastic bottle. This solution is also used for sample dilution and all ICB/CCB's.

9.4.2 Standard 2: 100 ul of the standard from section 9.3.1 + 100 ul of the standard from section 9.3.2 + 1000 ul of the standard from section 9.3.3 + 1000 ul of the standard from section 9.3.4 + 1000 ul of the standard from section 9.3.5 to 100.0 ml.

9.4.3 CRI: 1000 ul of the standard from section 9.3.16 to 100.0 ml.

9.4.4 ICSA: 10.0 ml of the standard from section 9.3.17 to 100.0 ml.

9.4.5 ICSAB: 10.0 ml of the standard from section 9.3.17 + 1000 ul of the standard from section 9.3.18 to 100.0 ml.

9.4.6 ICV and CCV: 200 ul of the standard from section 9.3.6 +200 ul of the standard from section 9.3.14 + 200 ul of the standard from section 9.3.15 + 1000 ul of the standard from section 9.3.7 + 1000 ul of the standard from section 9.3.8 + 400 ul of the standard from section 9.3.9 + 375 ul of the standard from section 9.3.10 + 375 ul of the standard from section 9.3.11 + 200 ul of the standard from section 9.3.12 + 100 ul of the standard from section 9.3.13 to 200.0 ml.

9.4.7 Internal standard : All calibration and verification standards, digested quality control standards and actual samples are made to contain 1 ppm yttrium prior to analysis.

9.4.8 Post Digestion Spike Solution : 1.00 ml standard from section 9.3.1 + 1.00 ml standard from section 9.3.2 + 10.0 ml standard from section 9.3.3 to 50.0 ml. 100 ul of this solution is added to 10.0 ml of digestate. Post spike concentrations are as follows : 200 ppb each Al, As, Ba, Be, Cd, Co, Cr, Cu, Fe, Mn, Ni, Pb,Se, Sr, Tl, V, Zn, Sb, Sn, Ti, Mo, B, 10000 ppb each Ca, Mg, K, Na, 20 ppb Ag.

10.0 CALIBRATION

10.1 Calibration must be performed once every 24 hours. The calibration sequence is as followed: calibration of standard 1, then calibration of standard 2. Quality control solutions are then analyzed to verify calibration. See section 11 for quality control solutions.

10.2 The average of two burns is used for all calibration, quality control, and client samples.

11.0 QUALITY CONTROL

11.1 The Initial Calibration Verification solution (ICV) is analyzed immediately following calibration. The results of the ICV are to agree within 10% of the true values for 6010B or 5% for 200.7. If not, terminate the analysis, correct the problem, and recalibrate the instrument.

11.2 Initial Calibration Blank (ICB) is analyzed immediately following the ICV. The results of the ICB are to be less than ½ the RL for 6010B and less than MDL for 200.7. If not, terminate the analysis, correct the problem, and recalibrate the instrument.

11.3 Low Level QC Check Standard (CRI) is analyzed at the beginning of each analytical run. The results of the CRI are to agree within 50% of the true values.

11.4 Interference Check Solutions (ICSA and ICSAB) are analyzed at the beginning of each analytical run to verify the interelement and background correction factors. The non-spiked B elements in the ICSA shall be less than two times the RL, unless they are verified trace impurities. The spike results are to agree within 20% of the true values.

11.5 Continuing Calibration Verification (CCV) is analyzed prior to any samples, after every ten samples, and at the end of an analytical run. The results of the CCV are to agree within 10% of the true values. If not, terminate the analysis, correct the problem, recalibrate the instrument, and reanalyze all samples that are not bracketed by acceptable CCV's.

11.6 Continuing Calibration Blank (CCB) is analyzed immediately following the CCV, after every ten samples, and at the end of an analytical run. The results of the CCB are to be less than ½ the RL for 6010B and less than MDL for 200.7. If not, terminate the analysis, correct the problem, and recalibrate the instrument. Reanalyze all samples not bracketed by acceptable CCB's except those where analyte concentrations are greater than 10 times the CCB level for the affected analytes.

11.7 Method Blank (PB) is analyzed along with the sample batch it was prepared with. The PB must be carried through the entire procedure and contain the same acid concentration as the samples being analyzed. The results of the PB are to be less than the reporting limit. If not, redigest all samples in the associated batch and reanalyze the samples. This does not apply to samples where analyte concentrations are greater than 10 times the PB level for the affected analytes.

11.8 Laboratory Control Sample (LCS) is also carried through the entire procedure as the samples being analyzed. The results of the LCS are to agree within 20% of the true values for 6010B or 15% for 200.7, for aqueous samples and within vendor specified limits for solids. The acceptance criteria for silver in soil is 75-120% or within vendor specified limits. If not, redigest all samples in the associated batch and reanalyze samples.

11.9 Matrix Duplicate is analyzed at a frequency of one per matrix batch that does not exceed 20 actual samples for 6010B or 10 samples for 200.7. A duplicate sample is brought through the entire sample preparation and analytical process in duplicate. A control limit of 20% RPD is used for sample values greater than ten times the MDL.

11.10 Matrix Spike is analyzed at a frequency of one per matrix batch that does not exceed 20 actual samples for 6010B or 10 samples for 200.7. The matrix spike concentrations can be referenced in TestAmerica CT SOP CT-MES_9 section 9.4.

11.11 Post Digestion Spike (PDS) is analyzed for every matrix spike failure to check for matrix interferences. A portion of digested sample is spiked with post spike solution from section 9.4.8. A control limit of 25% of the true values is used for sample values less than four times the spike added.

11.12 Practical Quanitation Limits (PQL). The PQL is calculated as five to ten times the MDL. See Table #2.

11.13 Method Detection Limit (MDL). A minimum of seven replicate standards are digested and analyzed following 40 CFR Part 136, Appendix B. This is done annually

11.14 Each day, and before any samples can be analyzed, a blank and a high standard are analyzed to set the linear calibration range.

11.15 LDR study – Performed every six months the upper limit of the linear dynamic range must be

established for each wavelength utilized by determining the signal responses from a minimum of three different concentration standards across the range. The laboratory analyzes 4 levels of standards, one of which is close to the upper limit of the linear range and of which is at or below the Reporting limit. The upper range limit is an observed signal within <±10% of that extrapolated from the lower standards. Any sample concentrations detected at greater than 90% of the upper LDR limit, require dilution and reanalysis.

11.16 Serial Dilution. One five fold serial dilution must be analyzed for each batch of 20 samples to evaluate the effect of interferences on the non-diluted results. Those elements with concentrations greater than 50 times the MDL will be used to determine if there are any interferences present. A control limit of 10% is used. The client will be informed whenever an element is outside the control limit.

11.17 For analysis using the Thermo 61E instrument, each day before any samples can be analyzed, the polychromater profile is checked. From the main menu select <profile>, select <automatic>, introduce a 5 ppm arsenic solution, select <run>. The profile must fall within 0.2 units of zero, if not, select <calc SS>, adjust the vernier dial to the new calculated position.

11.18 Instrument Detection Limit (IDL). The average of the standard deviations of three runs on three non-consecutive days of a blank solution with seven consecutive measurements per day.
Each measurement must be performed as though it were a separate analytical sample. IDL's are to be determined every six months.

12.0 INSTRUMENTAL PROCEDURES

12.1 WARNING: Ensure that no pacemaker users are in the vicinity of the spectrometer. RF generator radiation may interfere with pacemaker operation.

A) Prior to initiation of the plasma, the operator must be fully trained on the use of the instrument.

B) Before starting the instrument, check the exhaust vents and cooling water to ensure they are operating.

C) Check that the pressure of the argon supply is 50 psi.

D) Check that the drain tube is immersed into the plastic waste container. Ensure there are no crimps in this tubing.

E) Connect the rinse and sample tubing on the peristaltic pump. Check that there is sufficient rinse solution in the rinse supply container. Start the instrument and allow at least 30 minutes to warm up prior to profile and calibration.

12.2 Sample Evaluation

A) Locate the sample digestates to be analyzed and compare them to the applicable prep log in order to determine the analysis protocols. Check login to determine which elements are required and prepare the appropriate run sequence.

B) Visually inspect the sample digestates, noting which samples were in duplicate and which were spiked. Also note initial and final sample volumes, which laboratory control samples were used and also the sample weights and volumes. Check this against the sample prep log for completeness for all these mentioned items.

C) Samples must be free of particulates before being aspirated. Carefully inspect samples as they are poured into the autosampler tubes. Gravity filter samples as needed.

D) All soil digestates with a 50ml final volume must be diluted 5x with 1% nitric acid for matrix matching. This dilution is taken into account in the preparation batch in the form of a increased final volume. This dilution is performed on all QA and samples. Any dilution performed above a 1:5 is noted as such as a DF in the analytical batch.

13.0 CALCULATIONS

13.1 Calculations are performed by the analytical software with direct reading in ppb (ug/L). The conversion to mg/Kg is as follows:

$$\frac{\text{(Reading in ppb)(sample digestate volume in liters)}}{\text{Sample Wt.in grams X dec. \% solids}} = \text{results in mg/Kg}$$

14.0 ACCEPTANCE CRITERIA

Acceptance criteria can be found in Quality Control Section 11 for all QC samples.

15.0 REPORTING OF RESULTS

15.1 Samples are entered into the LIMS system. All appropriate sections must be completed. The prep batch sheets are kept with digestates until analyzed.

15.2 A batch system is used. Only 20 samples are allowed in a batch. Each batch should have a duplicate sample, a spiked sample, a prep-blank and a laboratory control sample for 6010B. Each batch should have 2 duplicate samples, 2 spiked samples, a prep-blank and a laboratory control sample for 200.7. All of these samples should be indicated on the prep sheet.

15.3 Record pH, matrix, initial volume and final volume for each sample.

15.4 Record lot number of all solutions and reagents added.

15.5 Record in comment column if sample is diluted, concentrated, limited, preserved, reprepped or other changes.

16.0 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). Employees must abide by the policies in

Section 13 of the Corporate Safety Manual for "Waste Management and Pollution Prevention."

16.1 Pollution prevention is the reduction and/or elimination of wastes or the toxicity of wastes at the point of generation. The laboratory strives to keep all wastes at a minimum through a variety of techniques. These include the following.

16.1.1 Inventory control: Rotate stocks (first in, first out) and only purchase reasonable amounts of chemicals. Do not purchase excessive quantities, which will be discarded as wastes at some future point.

16.1.2 Material Handling and Storage: Keep containers properly labeled. Keep solvents covered. Store and handle all chemicals, reagents, standards, etc. to prevent contamination and degradation.

16.1.3 Personnel: All staff must be trained in the proper handling and disposal of waste.

16.1.4 Waste Reduction; Reduce the volume of waste generated wherever possible.

16.1.5 Chemical/material substitution: Use less toxic chemicals whenever possible. Avoid the use of chromic acid (Chromerge) solutions.

17.0 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 Tracking and Collection of Hazardous Waste SOP. The following waste streams are produced when this method is carried out.

17.1 All waste shall be managed in accordance with all state and federal requirements. See the TestAmerica Hazardous Waste Management Plan.

17.2 All personnel who handle or generate waste must be trained within six months of employment in proper waste handling and requirements.

17.3 All acid waste is disposed of into a 55 gallon polyethylene drum marked acid waste.

18.0 SUPPLEMENTAL DOCUMENTS

18.1 **None**

19.0 REFERENCES

19.1 USEPA SW846 Third edition Method No. 6010B.

19.2 EPA Method 200.7, Determination of Metals and Trace Elements in Water and Wastes by Inductively Coupled Plasma Atomic emission spectrometry. Revision 4.4. 1994.

20.0 SUBSTANTIVE REVISIONS

20.1 Changed name to STL from IEA. Updated method reference

20.2 Added section 3 – Terms and definitions, 16 & 17 for waste management and pollution prevention, renumber other sections: 02/12/00.

20.3 Updated section 6 to include the corporate health and safety SOP: 10/01/2004.

20.4 Updated to Method 6010B. Updated acceptance criteria for ICB and CCB section 11.2 and 11.6. Updated acceptance criteria for MB section 11.7. Updated acceptance criteria for CRI section 11.3. Updated acceptance criteria for PQL section 11.12. Updated linear range section 11.14, 03/21/05.

20.5 Updated section 11.16 to include instrument profile procedure; 09/27/07.

20.6 Combined 200.7 into this SOP, sect 4.2, 4.3, 11.1, 11.8, 11.9, 11.10, 15.2, 19.2. Removed DOD reference in 11.10. Updated Tables 1 & 2. Added to sections Safety, Pollution control, waste management. 5/6/09.

20.7 Changed sections 11.2 and 11.6 from three times the MDL to three times the IDL as per method 6010. Added section 11.17 to include IDL requirement. 5/22/09.

20.8 Section 2.1 clarified prep methods correlating to analysis methods. Sect 7.1 – removed preservation reqt's. Removed 9.4.3 for ICV and added to CCV section. Added 9.4.8 Post Digestion spike information. Table 2 updated for all elements, RL's and wavelengths. Sect 11.15 added to updated LDR requirements. Sect 12.2 D updated to clarify dilution requirements. Updated 11.2, 11/6 and Table 1 for CCB/ICB requirements. 10/13/09.

Table #1

Quality Control Sample	6010B Control Limit	200.7 Control Limit	Failure Action
ICV	± 10 %	± 5 %	Recalibrate
ICB	< ½ RL	< MDL	Recalibrate
CRI	± 50 %	± 50 %	Recalibrate
ICSA	± 20 %	± 20 %	Recalibrate
ICSAB	± 20 %	± 20 %	Recalibrate
CCV	± 10 %	± 10 %	Rerun Samples
CCB	< ½ RL	< MDL	Rerun Samples
Duplicate	± 20 % RPD	± 20 % RPD	Flag Sample
Sample Spike	± 25 % Recovery	± 25 % Recovery	Flag Sample
Prep Blank	< Reporting Limit	< Reporting Limit	Reprep Samples
Lab Control Sample	± 20 %, 95 % Confid Win	± 15 %, 95 % Confid Win	Reprep Samples

Table #2

Analyte	Wavelength (nm)	PQL Water (ug/L)	PQL Soil (Mg/Kg)
Aluminum	308.2	250	50
Antimony	206.8	15.0	3.3
Arsenic	189.0	15.0	4.2
Barium	493.4	5.0	1.0
Beryllium	313.0	5.0	1.0
Cadmium	226.5	5.0	1.0
Calcium	317.9	500	50
Chromium	267.7	5.0	1.0
Cobalt	228.6	5.0	1.0
Copper	324.7	10.0	2.4
Iron	271.4	125.0	25
Lead	220.3	15.0	3.0
Magnesium	279.0	500	50
Manganese	257.6	8.0	1.5
Nickel	231.6	5.0	1.0
Potassium	766.4	500	50
Selenium	196.0	38.0	7.5
Silver	328.0	5.0	1.0
Sodium	588.9	500	50
Thallium	190.8	15.0	3.0
Vanadium	292.4	5.0	1.0
Zinc	206.2	25.0	5.0
Boron	249.6	15.0	3.0
Molybdenum	202.0	15.0	3.0
Strontium	421.5	5.0	1.0
Titanium	334.9	15.0	3.0
Tin	189.9	15.0	3.0

Title: SOP for Determination of Trace metals by ICP/MS
[Method(s) 6020 / 200.8]

Approvals (Signature/Date):

10/14/09	10/15 /09
Technical Manager Date	Health & Safety Manager/Coordinator Date
10/13/09	10/26/09
Quality Assurance Manager Date	Laboratory Director Date

This SOP was previously identified as CT-MES-51_3.

1.0 APPROVALS

The approval of the Laboratory Director, the Facility QA Manager, Group Leader and Health and Safety Officer signify that the document has been prepared in accordance with the format requirements that have been established in this document. In addition, if the document is a Method SOP the signature signifies that it meets the specified requirements.

2.0 SCOPE AND APPLICATION

2.1 Inductively coupled plasma-mass spectrometry is applicable to the determination of sub part per billion concentrations of a large number of elements. The method is applicable to aqueous samples, TCLP and SPLP extracts, industrial wastes, soils, sludges and sediments. All samples are digested prior to analysis. Samples for method 6020 are digested using the 3010 method (SOP–CT-MES-9) for liquids and 3050 method (SOP-CT-MES-10) for solids. Samples for method 200.8 are digested using the 200.7/200.8 digestion procedure (SOP-MES-52).

2.2 The elements determined using this technique are:

Aluminum	Cobalt	Potassium	Zinc
Antimony	Copper	Selenium	Boron
Arsenic Iron	Silver	Silicon	
Barium Lead	Sodium	Beryllium	
Magnesium	Tin	Zirconium	Sulfur
Cadmium	Manganese	Titanium	Lithium
Calcium	Molybdenum	Thallium	Thorium
Chromium	Nickel	Vanadium	Uranium

2.3 The document control number for this SOP is CT-MES-51, rev 4.

3.0 TERMS AND DEFINITIONS

3.1 There are many definitions used with in the laboratory, which may be generic to all laboratory analyses, or more specific for certain methods. For the most recent terms and definitions used with in the laboratory, reference the SOP for Terms and Definitions.

4.0 SUMMARY OF METHOD

4.1 This technique measures ions produced by a radio frequency inductively coupled plasma. Analytes originating from a digestate are nebulized and the resulting aerosol transported by argon gas into the plasma torch. The ions are extracted from the plasma and separated on the basis of their mass to charge ratio by the mass spectrometer.

4.2 This method uses guidance from methods 6020 and 200.8.

5.0 INTERFERENCES

5.11 Isobaric elemental interferences are caused by isotopes of different elements which form singly or doubly charged ions of the same nominal mass-to-charge ratio. Masses selected for quantification are chosen such that they are free of isobaric elemental interferences. The Agilent Chemstation library lists all known interferences associated with the available masses.
In addition doubly charged species are monitored daily by analyzing for Ce++/Ce+ during the daily tuning of the instrument. Acceptable levels of the cerium species is less than three percent.

5.12 Isobaric polyatomic ion interferences are caused by ions consisting of more than one atom which have the same nominal mass-to-charge ratio as the isotope of interest. These ions are commonly formed in the plasma or interface system. The collision cell facilitates the reduction of polyatomic

species by lowering their energies and thus allowing for energy discrimination. The collision of the interferences with helium gas reduces the kinetic energy of the interfering molecule (larger in size). Energy discrimination is then used to block these lower energy interferences from entering the quadrupole. In addition polyatomic oxides are monitored daily by analyzing for CeO/Ce during the daily tuning of the instrument. Acceptable levels of the oxide species is less than three percent.

5.13 Physical interferences are associated with the physical processes which govern the transport of sample into the plasma, sample conversion processes in the plasma, and the transmission of ions through the plasma-mass spectrometer interface. These interferences may result in differences between instrument responses for the samples and calibration standards. These interferences are governed primarily by viscosity effects and the resulting surface tension of the aerosol as it is ionized in the plasma. High levels of dissolved solids in the samples may also contribute deposits of material on the skimmer cones reducing the effective diameter of the orifices and therefore ion transmission. These effects are minimized by diluting all samples and prepared quality control samples with a diluent containing internal standards with masses spanning the entire mass range of elements to be determined. Likewise, calibration standards and instrument quality control standards are spiked with the same internal standards as the samples at the same concentration. This will reduce the total dissolved solids of the samples and compensate for the transfer differences between samples and standards.

5.14 Memory interferences result when isotopes in a previous sample contribute to the signals measured in a new sample. Memory effects can result from sample deposition on the sampler and skimmer cones, and from the buildup of sample material in the plasma torch and spray chamber. Memory effects are minimized by dilution such that only relatively low level samples are analyzed. However, sufficient rinse time between samples is required to properly minimize these effects. In addition, results for all analytes must be monitored. Any analyte whose concentration is greater than that of the highest calibration standard must be checked in the next sample. If the integrated signal values of the replicates drop consecutively, than memory interferences are likely. Those samples should be re-analyzed for the affected analytes. All samples are to be analyzed in triplicate.

6.0 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.

6.1 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.

The ICP plasma emits strong UV light and is harmful to vision. All analysts must avoid looking directly at the plasma.

6.2 **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.
1 – Always add acid to water to prevent violent reactions.			
2 – Exposure limit refers to the OSHA regulatory exposure limit.			

7.0 SAMPLE CONTAINERS, COLLECTION AND PRESERVATION

7.1 Samples are stable when digestion is complete and need no preservation. Sample bottles are not to be reused.

7.2 Holding time is 180 days.

8.0 APPARATUS AND MATERIALS

8.1 Agilent 7500cx ICP-MS
8.2 Agilent ASX 500 Autosampler

8.3 Peristaltic Pump

8.4 Ultra high purity argon

8.5 Ultra high purity helium

8.6 Personal Computer

8.7 100 and 1,000 uL variable Eppendorf pipettes

8.8 10 ml graduated serological pipettes

8.9 12 mL capacity polypropylene sample tubes (Borosilicate glass must not be used for the analysis of Boron or Silicon in order to avoid sample contamination).

9.0 REAGENTS AND STANDARD PREPARATION

9.1 Reagent water - ASTM Type II, 18 megohm, deionized

9.2 Trace grade nitric acid.

9.3 Trace grade hydrochloric acid.

9.4 ICP stock standards: All standards are to be plasma grade and must come with certificates of analysis traceable to NIST standards. All analytes in the calibration verification standards must be from a different source than those found in the calibration standards.

9.4.1 Calibration Standards used for the curve, Cal. Std 1, Cal. Std 2, Cal. Std 3 and their concentrations for each element are listed in Table 2.0

9.4.2 Calibration Verification Standard (CCV) – Must be from a second source than that of the calibration curve standards. Typical concentrations are listed in Table 2.0 and should be near the midpoint of the calibration curve.

9.4.3 CRI Standard – Concentrations are listed in Table 2.0 and must be at or below the reporting limit. May be from the same source as the calibration standards.

9.4.4 ICSA and ICSAB – Interference check standard concentrations are listed in Table 2.0

9.4.5 Internal Standard stocks: 1000 ppm each Sc45, Y89, In115 and Bi209.

9.5 Working Standards: All working standards are made in volumetric flasks with a final acid concentration of 1 % nitric and 0.1 ppm in each of the internal standards. Standards that contain Boron or Silicon must be stored in plastic bottles. All standards that are not digested are also made to contain 0.2% HCl prior to analysis.

9.6 Diluent solution for waters: 112 ppb each Sc, Y, In and Bi in 0.5 % HNO3. All digested waters are to be diluted by a factor of 10 with this solution prior to analysis.

9.7 Diluent solution for soils: 106 ppb each Sc, Y, In and Bi in reagent water. All digested soils are to be diluted by a factor of 20 with this solution prior to analysis.

9.8 Tuning solution: 10 ppb each Li, Y, Ce, Tl, Co, Mg, In, Pb, Ba, and Be. Analyze daily to check for

sensitivity, oxides, doubly charged species and mass axis calibration. Refer to section 10.1 for specific tune requirements. Consult the operation manual on proper tuning procedures.

10.0 CALIBRATION

10.1 Tuning must be performed daily to check for sensitivity, oxides, doubly charged species and mass axis calibration, once every 12 hrs. The following requirements must be met before samples can be analyzed.

Sensitivity	Li > 30000cps	Y > 80000cps	Tl > 40000cps
Oxides	<3% CeO	<10% RPD	
Doubly Charge Species	<3% Ce++	<10% RPD	
Mass Axis	+/- 0.1amu for all measured masses		
Peak Width at 10%	0.6 – 0.9amu for all measured masses		

Method 200.8 requires 5 consecutive successful tunes prior to sample analysis, with the resulting standard deviations of absolute signals for all analytes shown to be less than 5%.
Method 6020 requires 1 successful tune prior to sample analysis.

10.2 Calibration must be performed once every 12 hours. The calibration sequence is as followed: calibration blank followed Calibration standards 1,2,3 in that order. Quality control solutions are then analyzed to verify calibration. See section 11 for quality control solutions.

10.3 The average of three readings is used for all calibration, quality control, and client samples. An aspiration time of 0.1 seconds per mass, per reading, is used. A rinse blank containing 1% nitric acid is used to flush the instrument between all samples and standards.

11.0 QUALITY CONTROL

11.1 The Initial Calibration Verification solution (ICV)/QCS is analyzed immediately following calibration. This solution is to be from a source different from that of the calibration standards. The results of the ICV are to agree within 10% of the true values. For Cosmetic Lotion samples the results of the ICV are to agree within 8% of the true values. If the ICV does not meet the above criteria, terminate the analysis, correct the problem, and recalibrate the instrument. All samples analyzed under an out of control calibration must be reanalyzed.

11.2 Initial Calibration Blank (ICB) is analyzed immediately following the ICV. The results of the ICB are to be less than one half the RL for 6020 and less than the MDL for 200.8. If not, terminate the analysis, correct the problem, and recalibrate the instrument.

11.3 Low Level QC Check Standard (CRI) is analyzed at the beginning of each analytical run. The results of the CRI are to agree within 30% of the true values.

11.4 Interference Check Solutions (ICSA and ICSAB) are analyzed at the beginning of each analytical run. The non-spiked B elements in the ICSA shall be less than the RL, unless they are verified trace impurities. The spike results are to agree within 20% of the true values.

11.5 Continuing Calibration Verification (CCV) is analyzed prior to any samples, after every ten samples, and at the end of an analytical run. The results of the CCV are to agree within 10% of the true values. If not, terminate the analysis, correct the problem, recalibrate the instrument, and reanalyze all samples that are not bracketed by acceptable CCV's.

11.6 Continuing Calibration Blank (CCB) is analyzed immediately following the CCV, after every ten samples, and at the end of an analytical run. The results of the CCB are to be less than one half the RL for 6020 and less than the MDL for 200.8. If not, terminate the analysis, correct the problem, and recalibrate the instrument. Reanalyze all samples not bracketed by acceptable CCB's except those where analyte concentrations are greater than 10 times the CCB level for the affected analytes.

11.7 Method Blank (PB) is analyzed along with the sample batch it was prepared with. The PB must be carried through the entire procedure and contain the same acid concentration as the samples being analyzed. The results of the PB are to be less than the RL for method 6020 and less than the MDL for method 200.8. If not, redigest all samples in the associated batch and reanalyze the samples. This does not apply to samples where analyte concentrations are greater than 10 times the PB level for the affected analytes.

11.8 Laboratory Control Sample (LCS) is also carried through the entire procedure as the samples being analyzed and should contain each analyte of interest. The results of the LCS are to agree within 20% of the true values for method 6020 and 15% for method 200.8. If not, redigest all samples in the associated batch and reanalyze samples.

11.9 Matrix Duplicate is analyzed at a frequency of one per matrix batch that does not exceed 20 actual samples for method 6020 and 10 actual samples for method 200.8. A duplicate sample is brought through the entire sample preparation and analytical process in duplicate. A control limit of 20% RPD is used for sample values greater than ten times the MDL. Recoveries outside the control limit are flagged.

11.10 Matrix Spike is analyzed at a frequency of one per matrix batch that does not exceed 20 actual samples for method 6020 and 10 actual samples for method 200.8. A control limit of 25% of the true value is used for sample values less than four times the spike added. Recoveries outside the control limit are flagged.

11.11 Post Digestion Spike (PDS) is analyzed for every matrix spike failure to check for matrix interferences. A portion of digested sample is spiked with the same spiking solutions as are used for digestion. A control limit of 25% of the true values is used for sample values less than four times the spike added. Recoveries outside the control limit are flagged.

11.12 Reporting limits for each element are established based on the initial MDL study and the ability of the laboratory to produce consistent preparation blanks with levels below the reporting limits for all analytes. The RL must be at least 3x the value of the current MDL. Reporting limits for both soil and waters are listed in Table 2.0

11.13 Method Detection Limit (MDL). A minimum of seven replicate standards are digested and analyzed following 40 CFR Part 136, Appendix B. This is done annually

11.14 Serial Dilution. One five fold serial dilution must be analyzed for each batch of 20 samples or one per matrix type, whichever is greater. A control limit of 10% is used. If the control limit is exceeded,

reanalyze the serial dilution. If still out of control, dilute sample until a successful serial dilution can be obtained.

11.15 A linear range must be established for each element during initial instrument setup. A standard must be analyzed at those concentrations and the results must be within 10% of the true values. Any sample that contains analytes above the established linear range must be diluted and reanalyzed. The linear range should be verified whenever, in the judgment of the analyst, a change in analytical performance caused by either a change in instrument hardware or operating conditions would dictate they be re-determined.

11.16 Initial demonstration of capability is required before actual samples can be processed. 4 replicates of a digested laboratory control sample are analyzed. Recoveries must be within regular LCS criteria (section 11.8) for each replicate.

11.17 Internal standard recoveries must be monitored for all samples and quality control standards. Recoveries must fall within 60-120% of the original responses. Samples that fall outside these limits must be diluted until the internal standard responses fall within the control limits. Internal standard assignments are as follows :

- Li, Be, B, Na, Mg, Al, Si, S, K, Ca, Ti, V, Cr, Mn and Fe all use Sc45.
- Co, Ni, Cu, Zn, As, Se, Zr and Mo use Y89.
- Ag, Cd, Sn, Sb and Ba use In115. Pb, Tl, Th and U use Bi209.

11.18 Instrument detection limits (IDL's) are to be determined quarterly and can be established by calculating the average of the standard deviations of three runs on three non-consecutive days from the analysis of a reagent blank with seven consecutive measurements per day.

12.0 INSTRUMENTAL PROCEDURES

12.1 WARNING: Ensure that no pacemaker users are in the vicinity of the spectrometer. RF generator radiation may interfere with pacemaker operation.

A) Prior to initiation of the plasma, the operator should be familiar with the operator's manual supplied by Agilent.

B) Before starting the instrument, check the exhaust vents and cooling water to ensure they are operating.

C) Check that the pressure of the argon supply is 100 psi.

D) Check that the drain tube is immersed in the plastic waste container. Ensure there are no crimps in this tubing.

E) Connect the rinse and sample tubing on the peristaltic pump. Check that there is sufficient rinse solution in the rinse supply container. Start the instrument and allow at least 30 minutes to warm up prior to tuning and calibration.

12.2 Sample Evaluation

A) Locate the sample digestates to be analyzed and compare them to the applicable prep log in

order to determine the analysis protocols. Check login to determine which elements are required and prepare the appropriate run sequence.

B) Visually inspect the sample digestates, noting which samples were in duplicate and which were spiked. Also note initial and final sample volumes, which laboratory control samples were used and also the sample weights and volumes. Check this against the sample prep log for completeness for all these mentioned items.

C) All water digestates with a 50ml final volume must be diluted 10x with solution from section 9.6 for matrix matching. This dilution is taken into account in the preparation batch in the form of an increased final volume. This dilution is performed on all QA and samples. Any dilution performed above a 1:10 is noted as such as a DF in the analytical batch.

D) All soil digestates with a 50ml final volume must be diluted 20x with solution from section 9.7 for matrix matching. This dilution is taken into account in the preparation batch in the form of an increased final volume. This dilution is performed on all QA and samples. Any dilution performed above a 1:20 is noted as such as a DF in the analytical batch.

13.0 CALCULATIONS

13.1 Calculations are performed by the analytical software with direct reading in ppb (ug/L). The conversion to mg/Kg is as follows:

$$\frac{\text{(Reading in ppb)(sample digestate volume in liters)}}{\text{Sample Wt.in grams X dec. \% solids}} = \text{results in mg/Kg}$$

14.0 ACCEPTANCE CRITERIA

Acceptance criteria can be found in Quality Control Section 11 for all QC samples.

15.0 REPORTING OF RESULTS

15.1 Samples are entered into the LIMS system. All appropriate sections must be completed.

15.2 A batch system is used. Only 20 samples are allowed in a batch. Each batch should have a duplicate sample, a spiked sample, spiked duplicate, a prep-blank and a laboratory control sample. All of these samples should be indicated in the system logbook page.

15.3 Record pH, matrix, initial volume and final volume for each sample.

15.4 Record lot number of all solutions and reagents added.

15.5 Record in comment column if sample is diluted, concentrated, limited, preserved, reprepped or other changes.

16.0 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). Employees must abide by the policies in Section 13 of the Corporate Safety Manual for "Waste Management and Pollution Prevention."

16.1 Pollution prevention is the reduction and/or elimination of wastes or the toxicity of wastes at the point of generation. The laboratory strives to keep all wastes at a minimum through a variety of techniques. These include the following.

16.1.1 Inventory control: Rotate stocks (first in, first out) and only purchase reasonable amounts of chemicals. Do not purchase excessive quantities, which will be discarded as wastes at some future point.

16.1.2 Material Handling and Storage: Keep containers properly labeled. Keep solvents covered. Store and handle all chemicals, reagents, standards, etc. to prevent contamination and degradation.

16.1.3 Personnel: All staff must be trained in the proper handling and disposal of waste.

16.1.4 Waste Reduction; Reduce the volume of waste generated wherever possible.

16.1.5 Chemical/material substitution: Use less toxic chemicals whenever possible. Avoid the use of chromic acid (Chromerge) solutions.

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 Tracking and Collection of Hazardous Waste SOP. The following waste streams are produced when this method is carried out.

17.1 All waste shall be managed in accordance with all state and federal requirements. See the TestAmerica CT Hazardous Waste Management Plan.

17.2 All personnel who handle or generate waste must be trained within six months of employment in proper waste handling and requirements.

17.3 All acid waste is disposed of into a 55 gallon polyethylene drum marked acid waste.

18.0 SUPPLEMENTAL DOCUMENTS

18.1 **None**

19.0 REFERENCES

19.1 USEPA SW846 Third edition Method No. 6020 and EPA method 200.8.

20.0 SUBSTANTIVE REVISIONS

20.1 None – Original issue 7/7/08

20.2 10/14/08 – Added Table 2.0 to be referenced throughout Section 9. Sect 9.4.1 and 10.2 – added 3rd calibration level. Sect 10.1 – Tuning requirements added. 10.3 – aspiration time included. Sect 11.10 and 11.11 – changed criteria. Sect 11.12 – added RL's and specified requirements. Sect 11.14 – clarified serial dilution. Sect 11.17 – removed method specific criteria and changed to tighter criteria. Sections 6, 16, 17 updated to include more current safety, pollution control and waste mgmt. Sect. 5.11 & 5.12 updated percentages for acceptable levels.

20.3 Added in Section 11.1 the following statement to meet client requirements: For Cosmetic Lotion samples the results of the ICV are to agree within 8% of the true values. If the ICV does not meet the above criteria,". 11/14/08.

20.4 5/5/09 – 2.2 – Added 4 elements. Table 2.0 – Added 4 elements.

20.5 Sect 2.1 clarified prep procedures used. Sect 7.1 removed preservative requirements. Sect 9.4.5 and 11.17 added ISTD identification and associations. Sect 12.2 C and D added to clarify dilutions performed prior to analysis. Table 2 – Masses added and RL's updated. Updated 11.2, 11.6 and Table 1 for CCB/ICB criteria 10/13/09.

Table #1

Quality Control Sample	Control Limit	Failure Action
ICV	± 10 %	Recalibrate
ICB	< 1/2 RL(6020), <MDL(200.8)	Recalibrate
CRI	± 30 %	Recalibrate
ICSA	± 20 %	Recalibrate
ICSAB	± 20 %	Recalibrate
CCV	± 10 %	Rerun Samples
CCB	< 1/2 RL(6020), <MDL(200.8)	Rerun Samples
Duplicate	± 20 % RPD	Flag Sample
Sample Spike	± 25 % Recovery	Flag Sample
Prep Blank	< RL 6020 < MDL 200.8	Reprep Samples
Lab Control Sample	± 20 % 6020 +/- 15 % 200.8	Reprep Samples

Table 2.0
Standard solution Concentrations and Reporting Limits
(ug/L) unless otherwise noted

Elements/Masses	Cal Std 1	Cal Std 2	Cal Std 3	CCV	CRI	ICSA	ICSAB	RL's Waters ug/L (100ml:500ml)	RL's Soils mg/Kg (1.25g:1000ml)
Ag107	50	100	500	60	0.5	-	20	2.5	0.4
As75	50	100	500	200	0.5	-	10	2.5	0.4
Ba137	50	100	500	60	0.5	-	50	2.5	0.4
Be9	50	100	500	200	0.5	-	50	2.5	0.4
Cd111	50	100	500	60	0.5	-	100	2.5	0.4
Cr53	50	100	500	60	0.5	-	50	5	0.8
Co59	50	100	500	60	0.5	-	50	2.5	0.4
Cu63	50	100	500	60	0.5	-	50	5	0.8
Pb207	50	100	500	200	0.5	-	5	2.5	0.4
Mn55	50	100	500	40	0.5	-	50	6	1.0
Ni60	50	100	500	60	0.5	-	100	2.5	0.4
Se78	50	100	500	100	0.5	-	5	5	0.8
Tl205	50	100	500	200	0.5	-	10	3.5	0.6
V51	50	100	500	60	0.5	-	50	2.5	0.4
Zn66	50	100	500	60	0.5	-	100	25	4.0
B11	50	100	500	200	15	-	-	75	12
Mo95	50	100	500	200	2.5	400	400	15	2.4
Si29	50	100	500	200	n/a	-	-	250	40
Sn118	50	100	500	200	2.5	-	-	15	2.4
Ti47	50	100	500	200	2.5	400	400	15	2.4
Zr90	50	100	500	-	-	-	-	7.5	1.2
Sb121	100	200	1000	200	0.5	-	60	4	0.7
Al27	250	500	2500	1000	25	20000	20000	125	20
Fe57	250	500	2500	1000	25	50000	50000	125	20
Na23	500	1000	5000	2000	50	50000	50000	250	40
K39	500	1000	5000	4000	50	20000	20000	250	40
Ca44	500	1000	5000	2000	50	60000	60000	250	40
Mg24	500	1000	5000	2000	50	20000	20000	250	40
Chloride	-	-	-	-	-	360000	360000	n/a	n/a
Carbon	-	-	-	-	-	40000	40000	n/a	n/a
S34	1000	5000	20000	10000	n/a	20000	20000	5000	800
Li7	50	100	500	75	0.5	-	-	5	0.8
Th232	50	100	500	75	0.5	-	-	5	0.8
U238	50	100	500	75	0.5	-	-	2.5	0.4

Title: SOP for Mercury - Aqueous, Hot Block Digestion
[Method SW846 7470A]

Approvals (Signature/Date):

10/14/09	10/15 /09
Technical Manager Date	Health & Safety Manager/Coordinator Date
10/13/09	10/26/09
Quality Assurance Manager Date	Laboratory Director Date

This SOP was previously identified as CT-MES-31_8.

Facility Distribution No. Electronic **Distributed To: Facility Intranet**

1.0 APPROVALS

The approval of the Laboratory Director, the Facility QA Manager, Group Leader and Health and Safety Officer signify that the document has been prepared in accordance with the format requirements that have been established in this document. In addition, if the document is a Method SOP the signature signifies that it meets the specified requirements.

2.0 SCOPE AND APPLICATION

2.1 This SOP defines the analysis of samples by cold-vapor atomic absorption for samples of an aqueous matrix. Samples are digested and then analyzed for mercury by reduction with stannous chloride, which is added in-line by a mercury analyzer.

2.2 The element determined is defined as "Total Mercury" for an unfiltered sample and as "Dissolved Mercury" for a filtered sample. All samples, calibration standards and quality control samples must be digested prior to analysis.

2.3 The document control number for this document – CT-MES-31, Rev 9.

3.0 TERMS AND DEFINITIONS

3.1 There are many definitions used within the laboratory, which may be generic to all laboratory analyses, or more specific for certain methods. For the most recent terms and definitions used within the laboratory, reference the SOP for Terms and Definitions.

4.0 SUMMARY OF METHOD

4.1 The flameless AA procedure is based on the absorption of radiation at 253.7 nm by mercury vapor. Organic mercury compounds are oxidized, additional potassium permanganate will be added to ensure that the purple color persists for at least 15 minutes; the mercury is reduced to the elemental state, and aerated from solution in a closed system. The mercury vapor passes through a cell positioned in the light path of a mercury analyzer. Absorbance (peak height) is measured as a function of mercury concentration and recorded in the usual manner.

4.2 This method is based on SW846 Method 7470A and EPA Method 245.1.

5.0 INTERFERENCES

5.1 Potassium permanganate is added to eliminate possible interference from sulfide. Concentrations as high as 20 mg/L of sulfide do not interfere with the recovery of added mercury.

5.2 Copper has been reported to interfere; however, copper concentrations as high as 10 mg/L have no effect on mercury recovery. Samples suspected of containing appreciably greater amounts must be diluted prior to digestion.

5.3 High levels of free chlorine interfere with the analysis because chlorine also absorbs at a wavelength of 253.7 nm. Hydroxylamine hydrochloride is added prior to analysis to eliminate chlorine and to reduce the excess permanganate.

5.4 Interference from certain volatile organic compounds that also absorb at 253.7 nm is possible. A preliminary run without reagents should be made if this is suspected.

6.0 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.

6.1 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.

6.2 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
Mercury (1,000 PPM in Reagent)	Oxidizer Corrosive Poison	0.1 Mg/M3 Ceiling (Mercury 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	2 ppm-TWA 4 ppm-STEL	Nitric acid is extremely hazardous; it is corrosive, reactive, an oxidizer, and a poison.

	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 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.			

7.0 SAMPLE CONTAINERS, COLLECTION AND PRESERVATION

7.1 SAMPLE COLLECTION: Samples are collected in 500-mL bottles. Sample bottles are not to be reused.

7.2 SAMPLE PRESERVATION: Sample preservation is with nitric acid and preserved to a pH of less than two.

7.3 Samples that are received unpreserved or have insufficient preservation must be acidified to pH <2 with nitric acid, mixed, and held for 16 hours. The pH will be verified at <2 prior to withdrawing an aliquot for processing.

7.4 HOLDING TIMES: Samples must be analyzed within 28 days of collection.

8.0 APPARATUS AND MATERIALS

8.1 Perkin Elmer FIMS 100 Flow Injection Mercury System.

8.2 Hot Block Digestion Unit (SC100)

8.3 Thermometer - glass, mercury, 1 degree increments, 100 degree calibrated thermometer.

8.3.1 The temperature of the digestor must be monitored during each batch. This is done by filling a digestion tube ¾ full with reagent water and placing it in a digestion slot. A calibrated thermometer is then placed in the tube and monitored throughout the digestion.

8.4 Polypropylene digestion tubes with caps (50 ml).

8.5 Eppendorf Pipets (Brinkmann 2000 series, variable, 100uL and 1000uL).

8.5.1 Eppendorf pipets must be calibrated quarterly at similar volumes used to dispense standards and reagents. The procedure for Eppendorf verifications can be found in the SOPfor Pipette calibration.

8.5.2 Reagent water stored at room temperature is pipetted into a tared disposable beaker. The milliliters dispensed should equal the weight in grams. Pipets used for standards should be accurate to 0.5% and those used for reagents should be accurate to 1%. If not, calibrate the eppendorf following the manufacturer's instructions. Calibrations are to be recorded in a log specific to each pipet.

8.6 Digestion Tube Verification

Tare the weight of a digestion tube from a newly opened lot of tubes. Fill the tube with reagent water that is room temperature to the 25mL mark and weigh. Record that weight on the certificate supplied with the tubes. Repeat this procedure for the 50 mL mark. Weights should be within 2 percent of the respective volume amounts. Certificates are compiled in a separate folder. All volumes to be used (initial and final volumes), shall be verified by either the analyst or the manufacturer.

9.0 REAGENTS AND STANDARD PREPARATION

9.1 Water used in the preparation of standards or reagents must have a specific conductivity of less than 1 µmho/cm (or specific resistivity of greater than 1.0 megohm-cm) at 25°C.

9.2 Concentrated Sulfuric Acid, Reagent Grade

9.3 Concentrated Nitric Acid, Trace Grade

9.4 Concentrated Hydrochloric Acid, Trace Grade

9.5 Stannous Chloride (1.1% solution)- Add 11g Tin Chloride Dihydrate (SnCl2.2H2O) to a 1L plastic bottle. Add approximately 700 ml reagent water followed by 30 ml concentrated HCl. Fill to the top with reagent water, cap and mix by inversion. Store this solution at 4 C and discard if it turns green. Always make sure that there is enough reagent for the entire analytical run. Never mix batches of reagent during a run. However, batches can be combined prior to a run.

9.6 Sodium chloride-Hydroxylamine hydrochloride solution - Dissolve 120 g of each in 1 L of reagent water.

9.7 Potassium permanganate (5% solution) - Dissolve 125 g of potassium permanganate in 2.5 L of reagent water.

9.8 Potassium persulfate (5% solution) - Dissolve 125 g of potassium persulfate in 2.5L of reagent water.

9.9 3% HCl - To approximately 700 ml reagent water add 30 ml concentrated HCl. Add reagent water to make 1L total volume.

9.10 1000 ppm Mercury calibration standard – SCP, catalog #140-051-801(or equivalent). This standard is subsequently used to prepare the Intermediate standard, Initial calibration levels 1-5 and MS for water.

9.11 Second source Mercury standard (100 ppm) – Inorganic Ventures, Catalog # MSHG-100ppm (or equivalent). This standard is subsequently used to prepare the 2nd source Intermediate standard, the ICV, CCV, and LCS.

9.12 500 ppb intermediate standards for each source –
Source Intermediate - Add 50.0 ul of 1000 ppm mercury to a 100ml volumetric flask containing approximately 70 ml of reagent water and 2 ml of concentrated nitric acid. Add reagent water to the mark, stopper and mix by inverting the flask several times.
2nd Source Intermediate - Add 500 ul of 100 ppm mercury to a 100ml volumetric flask containing approximately 70 ml of reagent water and 2 ml of concentrated nitric acid. Add reagent water to the mark, stopper and mix by inverting the flask several times.

9.13 Table for Standard Preparation – Intermediates and Working Standards.

Mercury Standard Preparation

INTERMEDIATE STANDARD PREPARATION

Source=1000ug/ml Mercury (SCP, catalog #140-051-801(or equivalent))	Initial Conc ug/ml	Amount Used uls	Final Volume mls	Final Conc. ug/L
Intermediate - Hg	1000	50	100	500
Source=100ug/ml Mercury(2nd source)	Initial	Amount	Final	Final

	Conc ug/ml	Used uls	Volume mls	Conc. ug/L
2nd source Intermediate - Hg	100	500	100	500

Intermediate standards are diluted to volume using Reagent water and 2mls of concentrated nitric acid.

WORKING STANDARD PREPARATION

Source = Intermediate Hg(500ug/L) (Inorganic Ventures, catalog #MSHG-100ppm(or equivalent))	Initial Conc ug/L	Amount Used uls	Final Volume mls	Final Conc. ug/L
Hg Ical - Level 1	500	20	50	0.20
Hg Ical - Level 2	500	100	50	1.0
Hg Ical - Level 3	500	200	50	2.0
Hg Ical - Level 4	500	500	50	5
Hg Ical - Level 5	500	1000	50	10
MS Hg	500	100	50	1.0

Source = 2nd source Intermediate Hg(500ug/L)	Initial Conc ug/L	Amount Used uls	Final Volume mls	Final Conc. ug/L
ICV Hg	500	500	50	5.0
CCV Hg	500	500	50	5.0
LCS Hg	500	500	50	5.0

Working standards are diluted to volume using Reagent Water

10.0 CALIBRATION

10.1 Standards are to be run in ascending order. A five-point linear calibration curve is run and the correlation coefficient must be equal to or greater than 0.995 (i.e., $r \geq 0.995$) before actual samples can be run.

11.0 QUALITY CONTROL

11.1 Method detection limits are calculated according to 40 CFR Appendix B Part 136 and are performed annually.

11.2 The Practical Quantitation Limit (PQL) for mercury is 0.20 ug/L.

11.3 All stock solutions and standard preparations are logged and coded. All solutions are labeled with the following: analyte, concentration, analyst's initials, date prepared, and expiration date.

11.4 All chemicals will conform to minimum specifications set by the Reagent Chemicals Committee of the American Chemical Society. All chemical inventories are used on a first in first out basis.

11.5 A method blank or prep blank (PB) must be analyzed for every batch of 20 samples or every day,

which ever is more frequent. Method blanks are prepared using blank reagent water. All samples with an unacceptable method blank are to be reprepped and rerun.

11.6 One sample duplicate must be analyzed from each batch of 20 or fewer samples. The samples identified as field blanks are not to be used for duplicate analysis.

11.7 One sample spike is performed on every batch of 20 or fewer samples. Samples identified as field blanks may not be used for sample spike analysis. Add 100 ul of intermediate standard listed in section 9.13 to sample prior to the addition of any reagents. Either the calibration or the calibration verification intermediate may be used. The resulting spike amount will be 1 ug/L when sample is brought to final volume.

11.8 Upon client request, a sample matrix spike duplicate can be performed along with a sample matrix spike and at the same frequency.

11.9 One independent standard or laboratory control standard (LCS) for mercury is to be analyzed with each batch of 20 samples. ERA's mercury standard catalog #666, or the LCS can be prepared from the same source standard as the initial calibration standard. All samples with an unacceptable LCS are to be reprepped and rerun.

11.10 All digestates with concentrations above 10.0 ug/L must be diluted and reanalyzed.

11.11 Initial and Continuing Calibration:

Activity	Frequency
Initial Calibration Verification (ICV)	Immediately after the calibration curve
Initial Calibration Blank (ICB)	Immediately following the ICV
Continuing Calibration Verification (CCV)	After every ten samples, and after the last analytical sample.
Continuing Calibration Blank (CCB)	Immediately following each CCV

If a standard or CCB fails then, the 10 samples prior to that standard must be reanalyzed as well as the samples after that QC.

12.0 SAMPLE PREPARATION AND INSTRUMENTAL PROCEDURES

12.1 Sample preparation: Pour a well shaken sample into a 50 ml digestion tube accurately to the 25 ml mark. For dilutions, pipet the appropriate amount of well shaken sample to the digestion tube and add reagent water accurately to the 25 ml mark.

- Add 1.25 ml concentrated sulfuric acid and 0.6 ml concentrated nitric acid to each tube, mixing between additions.
- Add 4.0 ml of potassium permanganate solution to each tube and let stand for at least 15 minutes. Samples with high reductive properties will reduce the permanganate and cause the purple color to disappear. Add additional permanganate until the purple color persists. If

more than 5 additional ml of permanganate is needed, than a stronger permanganate solution will be needed. Likewise, if after digestion a sample loses its purple color, than it must be redigested using a larger addition of permanganate. Always include an additional reagent blank that contains the same permanganate concentration as the highest sample.

- Add 2.0 ml of potassium persulfate to each tube and heat for two hours in a hot block maintained at 95 C.
- Allow samples to cool to room temperature.
- Add 1.5 ml of hydroxylamine solution and swirl to mix.
- Add additional hydroxylamine solution to eliminate the purplish color if needed.
- Add reagent water accurately to the 50 ml mark, cap and mix the tubes by inverting them several times.

12.2 Standard preparation: Transfer 20, 100, 200, 500 and 1000 ul aliquots of the 500 ppb mercury calibration standard to a series of digestion tubes. This will correspond to 0.2, 1, 2, 5 and 10 ppb mercury in the final diluted digestates(Refer to sect 9.13). Add reagent water to the 25 ml mark and swirl to mix. Prepare enough calibration verification standards that will be required for the entire run from the second source intermediate, as dictated in section 11.10. Similarly prepare all necessary calibration blanks and laboratory control samples.

- To each tube add 1.25 ml of concentrated sulfuric acid and 0.6 ml of concentrated nitric acid, mixing after each addition.
- Add 4.0 ml of permanganate solution and allow to stand for at least 15 minutes.
- Add 2.0 ml of potassium persulfate solution to each tube and heat in a hot block maintained at 95 C.
- Allow samples to cool to room temperature.
- Add 1.5 ml of hydroxylamine solution and swirl to mix.
- Add additional hydroxylamine solution to eliminate the purplish color if needed.
- Add reagent water accurately to the 50 ml mark, cap and mix the tubes by inverting them several times.

Note: Always make sure that there is enough of all the reagents that will be needed for the entire batch of digestions. Reagent batches can only be combined prior to sample preparation.

12.3 Instrument Operation

A) Turn on instrument and printer.

B) Open FIMS Folder, open AA winlab analyst folder.

C) On workspace screen open automated analysis.

D) Open sample information and fill in all sample criteria.

E) Open File and Save As using the date the samples are being analyzed.

F) Print sample info file and close widow.

G) Open Automated analysis window, select browse, open file to be analyzed and click OK.

H) Click "use entire sample" box.

I) Click method STLHG at top of screen and click OK

J) Open FIAS window and click pump 1, let run for two minutes, shut off pump 1 and close window.

K) In automated analysis window select "analyze all" to start analysis.

L) Run Sequence proceeds as follows:

Standards in ascending order.

ICV, ICB

10 samples (includes prep blanks, duplicates, and spikes).

CCV, CCB

Repeat the above two steps until all samples are analyzed.

12.5 Digestates that exceed 10 ppb mercury must be diluted with dilution solution and reanalyzed.

12.6 Digestates must be free of particulates before being analyzed. Therefore, filtering and or diluting must be employed to limit particulates.

12.6.1 If filtering is needed prior to analysis; inspection of disposable filter and syringe coupling points to verify syringe and filter will be properly connected is required before actual filtration. Once verified that the filter and syringe are suitable for filtering, the sample to be filtered will be drawn up into the syringe and the filter will be attached to the end of the syringe by hand, with good force, so as to facilitate a strong and proper seal between the filter/syringe. The filter/syringe will be pressed against the analysis tube, with the tip of the filter physically inside the tube. The filtration will be done with the filter/syringe held pointing downward, and not at an angle that would allow a 'looking down the barrel' position in relation to the individual.

13.0 CALCULATIONS

13.1 A linear calibration curve is used to calculate results.

13.2 Sample Spike Recovery: $\%\ R = [(SSR - SR)/SA] \times 100$

SSR is Sample spike recovery in ug/L
SR is Sample result in ug/L
SA is Spike added in ug/L

13.3 $\%\ RPD = \{(| A - B |)/ [(A + B)/2]\} \times 100$

A is the sample result in ug/L
B is the duplicate result in ug/L

14.0 ACCEPTANCE CRITERIA

Initial Calibration Verification ± 10%
Continuing Calibration Verification ± 20%
Initial Calibration Blank: (<MDL)
Method Blank: (< ½ reporting limit)

Duplicates	± 20%
	± PQL if sample concentration < 5 x Reporting Limit
Spikes	± 25%
Laboratory Control Sample:	± 20%

15.0 REPORTING OF RESULTS

Practical Quantitation Limits:	0.20
Units of measure:	ug/L
Significant Figures:	3 figures

16.0 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). Employees must abide by the policies in Section 13 of the Corporate Safety Manual for "Waste Management and Pollution Prevention."

16.1 Pollution prevention is the reduction and/or elimination of wastes or the toxicity of wastes at the point of generation. The laboratory strives to keep all wastes at a minimum through a variety of techniques. These include the following.

16.1.1 Inventory control: Rotate stocks (first in, first out) and only purchase reasonable amounts of chemicals. Do not purchase excessive quantities, which will be discarded as wastes at some future point.

16.1.2 Material Handling and Storage: Keep containers properly labeled. Keep solvents covered. Store and handle all chemicals, reagents, standards, etc. to prevent contamination and degradation.

16.1.3 Personnel: All staff must be trained in the proper handling and disposal of waste.

16.1.4 Waste Reduction: Reduce the volume of waste generated wherever possible.

16.1.5 Chemical/material substitution: Use less toxic chemicals whenever possible. Avoid the use of chromic acid (Chromerge) solutions.

17.0 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 Tracking and Collection of Hazardous Waste SOP. The following waste streams are produced when this method is carried out.

17.1 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."

17.2 All acid waste is disposed into a 55-gallon plastic drum located in the satellite accumulation area.

18.0 SUPPLEMENTAL DOCUMENTS

18.1 None

19.0 REFERENCES

19.1 USEPA SW846 Test Methods for Evaluating Solid and Hazardous Wastes, Third Edition, method 7470, including revisions.

19.2 Methods for Chemical Analysis of Water and Wastes, EPA-600/4-82-055; December 1982, Method 245.1.

20.0 SUBSTANTIVE REVISIONS

20.1 Used daily working standard preparation from section 9 to section 12. Revised entire section 12 to include instrument procedures for the PE FIMS Mercury analyzer, added dilution solution to section 9, expanded QC sections 11.5, 11.8, 11.10; 4/28/03 MKC.

20.2 Updated section 6 to include the corporate health and safety SOP January 28, 2004.

20.3 Updated section 7 to remove glass and plastic bottles. Updated holding time to 28 days form time of collection.

20.4 Updated sections 10 and 13 to include linear calibration calculation, Added new TestAmerica SOP header and control number; 09/27/07.

20.5 Added section 12.6.1. Added MSD into QC section and Table 1. Updated safety, pollution control and waste management sections. 2/12/09.

20.6 Added section 7.3 to address samples received with insufficient preservation. Updated 8.5.1 for eppendorf verification. Updated sect 9.1 for conductivity requirements. Clarified 9.10 and 9.11 standard use. Removed LCS source standard and dilution solution from sect 9. Added 9.13 std prep table. Sect 8.6 Digestion tube verification added. 10/12/09.

Table #1

Quality Control Sample	Control Limit	Failure Action
ICV	± 10 %	Recalibrate
ICB	< MDL	Recalibrate
CCV	± 20 %	Rerun Samples
CCB	< ½ reporting limit	Rerun Samples
Duplicate	± 20 % RPD	Flag Sample
Sample Spike	± 25 % Recovery	Flag Sample
Matrix Spike Duplicate	± 20 % RPD	Flag Sample
Prep Blank	< ½ reporting limit	Reprep Samples

Title: SOP for Mercury – Solids, Hot Block Digestion
[Method SW846 7471A]

Approvals (Signature/Date):

10/14/09	10/15 /09
Technical Manager Date	Health & Safety Manager/Coordinator Date
10/13/09	10/26/09
Quality Assurance Manager Date	Laboratory Director Date

This SOP was previously identified as CT-MES-32_6.

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 APPROVALS

The approval of the Laboratory Director, the Facility QA Manager, Group Leader and Health and Safety Officer signify that the document has been prepared in accordance with the format requirements that have been established in this document. In addition, if the document is a Method SOP the signature signifies that it meets the specified requirements.

2.0 SCOPE AND APPLICATION

2.1 This SOP defines the analysis of samples by cold-vapor atomic absorption for soils, sediments, bottom deposits and sludge–type materials. Samples are digested and then analyzed for mercury by reduction with stannous chloride, which is added in-line by a mercury analyzer.

2.2 The document control number for this document is CT-MES-32, Rev 7.

3.0 TERMS AND DEFINITIONS

3.1 There are many definitions used within the laboratory, which may be generic to all laboratory analyses, or more specific for certain methods. For the most recent terms and definitions used within the laboratory, reference the SOP for Terms and Definitions.

4.0 SUMMARY OF METHOD

4.1 The flameless AA procedure is based on the absorption of radiation at 253.7 nm by mercury vapor. Organic mercury compounds are oxidized; the mercury is reduced to the elemental state, and aerated from solution in a closed system. The mercury vapor passes through a cell positioned in the light path of a mercury analyzer. Absorbance (peak height) is measured as a function of mercury concentration and recorded in the usual manner. All samples, calibration standards and quality control samples must be digested prior to analysis.

4.2 This method is based on SW846 Method 7471A.

5.0 INTERFERENCES

5.1 Potassium permanganate is added to eliminate possible interference from sulfide. Concentrations as high as 20 mg/L of sulfide do not interfere with the recovery of added mercury.

5.2 Copper has been reported to interfere; however, copper concentrations as high as 10 mg/L have no effect on mercury recovery.

5.3 High levels of free chlorine interfere with the analysis because chlorine also absorbs at a wavelength of 253.7 nm. Hydroxylamine hydrochloride is added prior to analysis to eliminate chlorine and to reduce the excess permanganate.

5.4 Interference from certain volatile organic compounds that also absorb at 253.7 nm is possible. A preliminary run without reagents should be made if this is suspected.

6.0 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.

6.1 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.

6.2 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
Mercury (1,000 PPM in Reagent)	Oxidizer Corrosive Poison	0.1 Mg/M3 Ceiling (Mercury 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 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.
Aqua Regia	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.			
2 – Exposure limit refers to the OSHA regulatory exposure limit.			

7.0 SAMPLE CONTAINERS, COLLECTION AND PRESERVATION

7.1 SAMPLE COLLECTION: Samples are collected in glass bottles. Sample bottles are not to be reused.

7.2 SAMPLE PRESERVATION: Cool to 4 C.

7.3 HOLDING TIMES: Samples must be analyzed within 28 days of collection. Samples must be analyzed within 26 days of collection if following NYSDEC methodology.

8.0 APPARATUS AND MATERIALS

8.1 Perkin Elmer FIMS 100 Flow Injection Mercury System.

8.2 Environmental Express Hot Block Digestion Unit (SC100)

8.3 Calibrated Thermometer

8.3.1 The temperature of the digestor must be monitored during each batch. This is done by filling a digestion tube ¾ full with reagent water and placing it in a digestion slot. A calibrated thermometer is then placed in the tube and monitored throughout the digestion. Polypropylene digestion tubes with caps (50 ml)

8.4 Eppendorf Pipets (Brinkmann 2000 series, variable, 100uL and 1000uL).

8.4.1 Eppendorf pipets must be calibrated quarterly at similar volumes used to dispense standards and reagents. The procedure for Eppendorf verifications can be found in the SOPfor Pipette calibration.

8.5 Reagent water stored at room temperature is pipetted into a tared disposable beaker. The milliliters dispensed should equal the weight in grams. Pipets used for standards should be accurate to 0.5% and those used for reagents should be accurate to 1%. If not, calibrate the eppendorf following the manufacturer's instructions. Calibrations are to be recorded in a log specific to each pipet.

8.6 Digestion Tube Verification

Tare the weight of a digestion tube from a newly opened lot of tubes. Fill the tube with reagent water that is room temperature to the 25mL mark and weigh. Record that weight on the certificate supplied with the tubes. Repeat this procedure for the 50 mL mark. Weights should be within 2 percent of the respective volume amounts. Certificates are compiled in a separate folder. All volumes to be used (initial and final volumes), shall be verified by either the analyst or the manufacturer.

9.0 REAGENTS AND STANDARD PREPARATION

9.1 Water used in the preparation of standards or reagents must have a specific conductivity of less than 1 µmho/cm (or specific resistivity of greater than 1.0 megohm-cm) at 25°C.

9.2 Concentrated Sulfuric Acid, Reagent Grade

9.3 Concentrated Nitric Acid, Trace Grade

9.4 Concentrated Hydrochloric Acid, Trace Grade

9.5 Stannous Chloride (1.1% solution)- Add 11g Tin Chloride Dihydrate (SnCl2.2H2O) to a 1L plastic bottle. Add approximately 700 ml reagent water followed by 30 ml concentrated HCl. Fill to the top with reagent water, cap and mix by inversion. Store this solution at 4 C and discard if it turns green. Always make sure that there is enough reagent for the entire analytical run. Never mix batches of reagent during a run. However, batches can be combined prior to a run.

9.6 Sodium chloride-Hydroxylamine hydrochloride solution - Dissolve 120 g of each in 1 L of reagent water.

9.7 Potassium permanganate (5% solution) - Dissolve 125 g of potassium permanganate in 2.5 L of reagent water.

9.8 3% HCl - To approximately 700 ml reagent water add 30 ml concentrated HCl. Add reagent water to make 1L total volume.

9.9 1000 ppm Mercury calibration standard – SCP, catalog #140-051-801(or equivalent). This standard is subsequently used to prepare the Intermediate standards, Initial calibration levels 1-5, CCV, LCS, MS for water and MS for soils.

9.10 Second source Mercury standard (100 ppm) – Inorganic Ventures, Catalog # MSHG-100ppm (or equivalent). This standard is subsequently used to prepare the ICV Intermediate standard and the ICV working standard.

9.11 Soil LCS is prepared using table 9.14, with the addition of glass beads to represent the soil matrix.

9.12 1000 ppm Mercury calibration standard – SCP, catalog #140-051-801(or equivalent). This standard is subsequently used to prepare the Intermediate standards, Initial calibration levels 1-5 and MS.

9.13 Second source Mercury standard (100 ppm) – Inorganic Ventures, Catalog # MSHG-100ppm (or equivalent). This standard is subsequently used to prepare the 2nd source Intermediate standard, the ICV, CCV, and LCS.

9.14 500 ppb intermediate standards for each source –
Source Intermediate - Add 50.0 ul of 1000 ppm mercury to a 100ml volumetric flask containing approximately 70 ml of reagent water and 2 ml of concentrated nitric acid. Add reagent water to the mark, stopper and mix by inverting the flask several times.
2nd Source Intermediate - Add 500 ul of 100 ppm mercury to a 100ml volumetric flask containing approximately 70 ml of reagent water and 2 ml of concentrated nitric acid. Add reagent water to the mark, stopper and mix by inverting the flask several times.

9.15 Aqua Regia – 400ml Type II Water, 100ml concentrated Nitric acid, and 300ml concentrated HCL.

Solution can be prepared at different volumes as long as the ratios are kept consistent.

10.0 CALIBRATION

10.1 Standards are to be run in ascending order. A five-point linear calibration curve is run and the correlation coefficient must be equal to or greater than 0.995 (i.e., $r \geq 0.995$) before actual samples can be run.

11.0 QUALITY CONTROL

11.1 Method detection limits are calculated according to 40 CFR Appendix B Part 136 and are performed annually.

11.2 The instrument Practical Quantitation Limit (PQL) for mercury is 0.20 ug/L. The final concentration reporting limit is 0.050mg/Kg.

11.3 All stock solutions and standard preparations are logged and coded. All solutions are labeled with the following: analyte, concentration, analyst's initials, date prepared, and expiration date.

11.4 All chemicals will conform to minimum specifications set by the Reagent Chemicals Committee of the American Chemical Society. All chemical inventories are used on a first in first out basis.

Mercury Standard Preparation

INTERMEDIATE STANDARD PREPARATION

Source=1000ug/ml Mercury (SCP, catalog #140-051-801(or equivalent))	Initial Conc ug/ml	Amount Used uls	Final Volume mls	Final Conc. ug/L
Intermediate - Hg	1000	50	100	500
Source=100ug/ml Mercury(2nd source)	Initial Conc ug/ml	Amount Used uls	Final Volume mls	Final Conc. ug/L
2nd source Intermediate - Hg	100	500	100	500

Intermediate standards are diluted to volume using Reagent water and 2mls of concentrated nitric acid.

WORKING STANDARD PREPARATION

Source = Intermediate Hg(500ug/L) (Inorganic Ventures, catalog #MSHG-100ppm(or equivalent))	Initial Conc ug/L	Amount Used uls	Final Volume mls	Final Conc. ug/L
Hg Ical - Level 1	500	20	50	0.20
Hg Ical - Level 2	500	100	50	1.0
Hg Ical - Level 3	500	200	50	2.0
Hg Ical - Level 4	500	500	50	5.0
Hg Ical - Level 5	500	1000	50	10
MS Hg	500	200	50	2.0
Source = 2nd source Intermediate Hg(500ug/L)	Initial Conc ug/L	Amount Used uls	Final Volume mls	Final Conc. ug/L
ICV Hg	500	500	50	5.0
CCV Hg	500	500	50	5.0
LCS Hg	500	500	50	5.0

Working standards are diluted to volume using Reagent Water

11.5 A method blank or prep blank (PB) must be analyzed for every batch of 20 samples or every day, which ever is more frequent. Method blanks are prepared using blank reagent water. All samples with an unacceptable method blank are to be reprepped and rerun.

11.6 One sample duplicate must be analyzed from each batch of 20 or fewer samples. The samples identified as field blanks are not to be used for duplicate analysis.

11.7 One sample spike is performed on every batch of 20 or fewer samples. Samples identified as field blanks may not be used for sample spike analysis. Add 200 ul of intermediate standard listed in section 9:12 to sample prior to the addition of any reagents. Either calibration or calibration verification intermediate may be used. The resulting spike amount will be 0.167 mg/kg when 0.6 g of sample is brought to a final volume of 50 ml.

11.8 Upon client request, a sample matrix spike duplicate can be performed along with a sample matrix spike and at the same frequency.

11.9 One independent standard or laboratory control standard (LCS) for mercury is to be analyzed with each batch of 20 samples. All samples with an unacceptable LCS are to be reprepped and rerun.

11.10 All digestates with concentrations above 10.0 ug/L must be diluted and reanalyzed.

11.11 Initial and Continuing Calibration :

Activity	Frequency
Initial Calibration Verification (ICV)	Immediately after the calibration curve
Initial Calibration Blank (ICB)	Immediately following the ICV
Continuing Calibration Verification (CCV)	After every ten samples, and after the last analytical sample.
Continuing Calibration Blank (CCB)	Immediately following each CCV

If a standard or CCB fails then, the 10 samples prior to that standard must be reanalyzed as well as the samples after that QC.

12.0 SAMPLE PREPARATION and ANALYSIS

12.1 Sample preparation: Weigh approximately 0.6g of sample, using three separate portions, and place in the bottom of a digestion tube. Make sure that very little sample is stuck to the sides of the tube.

- Add 10 ml half strength aqua regia and heat for 2 minutes in a hot block at 95 C.
- Cool and add reagent water to the 25 ml mark.
- Next add 5 ml of potassium permanganate solution and swirl to mix and let stand for at least 15 minutes. Samples with high reductive properties will reduce the permanganate and cause the purple color to disappear. Add additional permanganate until the purple color persists. If more than 5 additional ml of permanganate is needed, than a stronger permanganate solution will be needed. Likewise, if after digestion a sample loses its purple color, than it must be redigested using a larger addition of permanganate. Always include an additional reagent blank that contains the same permanganate concentration as the highest sample.
- Heat for 30 minutes in a hot block maintained at 95 C.
- Allow samples to cool to room temperature.
- Add 2 ml of hydroxylamine solution and swirl to mix.
- Add additional hydroxylamine solution to eliminate the purplish color if needed.
- Add reagent water accurately to the 50 ml mark, cap and mix the tubes by inverting them several times.

12.2 Standard preparation: Transfer 20, 100, 200, 500 and 1000 ul aliquots of the 500 ppb mercury calibration standard to a series of digestion tubes. This will correspond to 0.2, 1, 2, 5 and 10 ppb mercury in the final diluted digestates(refer to sect 9.14). Add reagent water to the 5 ml mark and swirl to mix. Prepare enough calibration verification standards that will be required for the entire run from the second source intermediate, as dictated in section 11.10. Similarly prepare all necessary calibration blanks and laboratory control samples.

- To each tube add 10 ml of half strength aqua regia solution and heat for 2 minutes in a hot block at 95 C.
- Cool and add reagent water to the 25 ml mark.

- Next add 5 ml of potassium permanganate solution and swirl to mix.
- Heat for 30 minutes in a hot block maintained at 95 C.
- Allow samples to cool to room temperature.
- Add 2 ml of hydroxylamine solution and swirl to mix.
- Add additional hydroxylamine solution to eliminate the purplish color if needed.
- Add reagent water accurately to the 50 ml mark, cap and mix the tubes by inverting them several times.

Note: Always make sure that there is enough of all the reagents that will be needed for the entire batch of digestions. Reagent batches can only be combined prior to sample preparation.

12.3 Instrument Operation

A) Turn on instrument and printer.

B) Open FIMS Folder, open AA winlab analyst folder.

C) On workspace screen open automated analysis.

D) Open sample information and fill in all sample criteria.

E) Open File and Save As using the date the samples are being analyzed.

F) Print sample info file and close widow.

G) Open Automated analysis window, select browse, open file to be analyzed and click OK.

H) Click “use entire sample” box.

I) Click method STLHG at top of screen and click OK

J) Open FIAS window and click pump 1, let run for two minutes, shut off pump 1 and close window.

K) In automated analysis window select “analyze all” to start analysis.

L) Run Sequence proceeds as follows:

Standards in ascending order.

ICV, ICB

10 samples (includes prep blanks, duplicates, and spikes).

CCV, CCB

Repeat the above two steps until all samples are analyzed.

12.2 Digestates that exceed 10 ppb mercury must be diluted with dilution solution and reanalyzed.

12.3 Dilution solution: To a 1L plastic bottle add approximately 500 ml of reagent water followed sequentially by 12.5 ml of concentrated nitric acid, 37.5 ml of concentrated hydrochloric acid, 100 ml of 5% permanganate solution and 40 ml of hydroxylamine solution, mixing after each addition. Fill the bottle completely with reagent water, cap and mix by inversion.

12.3 Digestates must be free of particulates before being analyzed. Therefore, filtering and or diluting must be employed to limit particulates.

12.3.1 If filtering is needed prior to analysis; inspection of disposable filter and syringe coupling points to verify syringe and filter will be properly connected is required before actual filtration. Once verified that the filter and syringe are suitable for filtering, the sample to be filtered will be drawn up into the syringe and the filter will be attached to the end of the syringe by hand, with good force, so as to facilitate a strong and proper seal between the filter/syringe. The filter/syringe will be pressed against the analysis tube, with the tip of the filter physically inside the tube. The filtration will be done with the filter/syringe held pointing downward, and not at an angle that would allow a 'looking down the barrel' position in relation to the individual.

13.0 CALCULATIONS

13.1 A linear calibration curve is used to calculate results.

13.2 Sample Spike Recovery: $\%\ R = [(SSR - SR)/SA] \times 100$

SSR is Sample spike recovery in ug/L
SR is Sample result in ug/L
SA is Spike added in ug/L

13.3 $\%\ RPD = \{(| A - B |)/ [(A + B)/2]\} \times 100$

A is the sample result in ug/L
B is the duplicate result in ug/L

13.4 All data is downloaded to the labnet data system. Open C: find the data file to be downloaded and drag file to the L: directory. Open L: find the data to be downloaded and drag file to MERC1. Data is now ready to be imported for reporting.

14.0 ACCEPTANCE CRITERIA

Initial Calibration Verification $\pm$ 10%
Continuing Calibration Verification $\pm$ 20%

Initial Calibration Blank: (<MDL)
Method Blank: (< ½ reporting limit)
Duplicates: ± 20%
± PQL if sample concentration < 5 x Reporting Limit
Spikes: ± 25%
Laboratory Control Sample: within manufacturers control limits.

15.0 REPORTING OF RESULTS

Practical Quantitation Limits:	0.050
Units of measure:	Mg/Kg
Significant Figures:	3 figures

16.0 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). Employees must abide by the policies in Section 13 of the Corporate Safety Manual for "Waste Management and Pollution Prevention."

16.1 Pollution prevention is the reduction and/or elimination of wastes or the toxicity of wastes at the point of generation. The laboratory strives to keep all wastes at a minimum through a variety of techniques. These include the following.

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16.1.2 Material Handling and Storage: Keep containers properly labeled. Keep solvents covered. Store and handle all chemicals, reagents, standards, etc. to prevent contamination and degradation.

16.1.3 Personnel: All staff must be trained in the proper handling and disposal of waste.

16.1.4 Waste Reduction: Reduce the volume of waste generated wherever possible.

16.1.5 Chemical/material substitution: Use less toxic chemicals whenever possible. Avoid the use of chromic acid (Chromerge) solutions.

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waste streams are produced when this method is carried out.

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17.2 All acid waste is disposed into a 55-gallon plastic drum located in the satellite accumulation area.

18.0 SUPPLEMENTAL DOCUMENTS

18.1 None

19.0 REFERENCES

19.1 USEPA SW846 Test Methods for Evaluating Solid and Hazardous Wastes, Third Edition, method 7471, including revisions.

19.2 Methods for Chemical Analysis of Water and Wastes, EPA-600/4-82-055 December 1982, Method 245.1.

20.0 SUBSTANTIVE REVISIONS

20.1 Used daily working standard preparation from section 9 to section 12. Revised entire section 12 to include instrument procedures for the PE FIMS Mercury analyzer, added dilution solution to section 9, expanded QC sections 11.5,11.8, 11.10; 4/28/03 MKC.

20.2 Updated section 6 to include the corporate health and safety SOP January 28, 2004.

20.3 Modified section 12.1 to use 10ml aqua regia, 03/18/2005.

20.4 Modified section 14 and Table 1 to meet method requirements, 03/18/2005.

20.5 Updated sections 10 and 13 to include linear calibration calculation, Added new TestAmerica SOP header and control number; 09/27/07.

20.6 Added section 12.3.1. Updated Safety, pollution control and waste management sections. Corrected Final RL in section 11.2 and 15.0. Corrected Sect 12.2 – 10ml. 2/12/09

20.7 Updated 8.4.1 for Eppendorf verification. Updated sect 9.1 for conductivity requirements. Updated 9.11 for use of glass beads. Added 9.13 std prep table. Sect 8.6 added for digestion tube verification. Sect 9.15 aqua regia preparation and added to safety section. 10/27/09.

Table #1

Quality Control Sample	Control Limit	Failure Action
ICV	± 10 %	Recalibrate
ICB	< MDL	Recalibrate
CCV	± 20 %	Rerun Samples
CCB	< ½ reporting limit	Rerun Samples
Duplicate	± 20 % RPD	Flag Sample
Sample Spike	± 25 % Recovery	Flag Sample
Matrix Spike Duplicate	± 20 % RPD	Flag Sample
Prep Blank	< ½ reporting limit	Reprep Samples