Remediation

ISLIP RESOURCE RECOVERY AGENCY

REMEDIAL INVESTIGATION / FEASIBILITY STUDY

SAMPLING ANALYSIS PLAN

SONIA ROAD LANDFILL
WEST BRENTWOOD, NEW YORK
SITE REGISTRY NO. 152013

Dvirka and Bartilucci
Consulting Engineers

VOLUME II OF IV

FEBRUARY 1997

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HAZARDOUS WASTE REMEDIATION

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SAMPLING AND ANALYSIS PLAN

REMEDIAL INVESTIGATION/FEASIBILITY STUDY
FOR
SONIA ROAD LANDFILL
WEST BRENTWOOD
SUFFOLK COUNTY, NEW YORK

(SITE REGISTRY NO. 152013)

VOLUME II OF IV

PREPARED FOR
ISLIP RESOURCE RECOVERY AGENCY
TOWN OF ISLIP, NEW YORK

BY
DVIRKA AND BARTILUCCI
CONSULTING ENGINEERS
WOODBURY, NEW YORK

FEBRUARY 1997

SAMPLING AND ANALYSIS PLAN
SONIA ROAD LANDFILL

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1.0 FIELD SAMPLING PLAN

Provided in the following sections is a detailed description of the field sampling program proposed for the Remedial Investigation (RI) to be conducted at the Sonia Road Landfill located in West Brentwood, Suffolk County, New York. The focus of the sampling program is to meet the objectives of the RI, which include collecting the data necessary to determine the nature and extent of contamination and to implement a remediation plan for the site within a short time frame. The field program discussed below includes program elements that will allow for a single phase RI. Obtaining the necessary environmental information together with pre-design data for a Presumptive Remedy/capping of the landfill, within a single phase investigation, will allow for an accelerated remediation program.

Although the focus of the investigation is the Sonia Road Landfill, the Town is interested in obtaining pertinent information with respect to groundwater quality up and downgradient of the site in order to fully characterize groundwater quality and identify other sources of contamination in the vicinity of the landfill. The identification of other sources of contamination will allow for a more comprehensive understanding of environmental impacts pertaining to all of the contributing sites. Therefore, in addition to the field sampling program being conducted under Title 3 EQBA Program, which provides 75% State funding of approved costs, portions of the program may be funded entirely by the Town. These portions include installation of monitoring wells in the industrial area northwest of the landfill and conducting a soil vapor survey on-site.

The elements of the RI field program are presented in summary format in Table 1-1. For a more detailed description of the sampling procedures see Section 2.0, Quality Assurance/Quality Control Plan.

1.1 Site Management

Primary access to the Sonia Road Landfill site is from Corbin Avenue. The site is entirely fenced and access can be made through one of three gates. One gate is located to the

Table 1-1

SONIA ROAD LANDFILL
REMEDIAL INVESTIGATION AND FEASIBILITY STUDY
REMEDIAL INVESTIGATION FIELD PROGRAM SUMMARY

Program Element	Rationale	Number of Samples	Equipment	Analysis
Aerial Photograph Review	Historical aerial photographs from 1940 to 1990 will be reviewed in order to evaluate the historical excavation and filling activities to define the approximate limits of waste up until 1974, and on-site activities, including grading, which took place after the landfill was closed.	Not Applicable.	Not Applicable.	Not Applicable.
Grid Network Survey	A 300-foot by 300-foot grid network will be established on-site for construction of test pits, positioning of on-site test borings and conducting the soil vapor survey.	Not Applicable.	Grid network survey will be conducted by a licensed New York State surveyor and will be incorporated into the existing topographic map. Each grid point will be identified with a stake and flagging pole.	Not Applicable.
Surface Soil Sampling	Surface soil samples (0-2 inches) will be collected to characterize surface soil quality on-site and off-site.	Four samples will be collected on-site and three samples will be collected off-site (one upgradient and two downgradient).	Surface soil samples will be collected using disposable polyethylene scoops	Each sample will be analyzed for TCL + 30, TAL parameters.

Table 1-1 (continued)

SONIA ROAD LANDFILL
REMEDIAL INVESTIGATION AND FEASIBILITY STUDY
REMEDIAL INVESTIGATION FIELD PROGRAM SUMMARY

Program Element	Rationale	Number of Samples	Equipment	Analysis
Soil Vapor Survey	Soil vapor samples will be collected to determine the presence of methane and volatile organic compounds.	Approximately 30 soil vapor measurements will be collected based on a 150-foot by 300-foot grid.	Soil vapor samples will be collected by driving a soil vapor probe 3 feet into the ground. The probe will be purged and once equilibrium has been reached, the probe will be monitored and measured with the PID/FID and combustible gas indicator. Samples for laboratory analysis may be collected using a vacuum pump and sorbent tubes.	Each sample will be screened with a PID/FID and combustible gas indicator. If elevated levels of VOCs are detected on the PID/FID, up to six samples will be collected and analyzed for volatile organics by USEPA Method T01/T02.
Test Trench Construction	Test trenches will be constructed in order to define the limits of the waste for cap design and depth of waste around perimeter of landfill to evaluate potential for waste consolidation.	Approximately 30 test trenches will be constructed based on 150-foot intervals around the perimeter of the landfill.	Each test trench will be constructed utilizing a backhoe. The test trench will be approximately 20 feet long, 3 feet wide and 10 feet deep or until waste or clean soil is encountered.	No samples will be collected for chemical analysis.
Test Pit Construction	Test pits will be constructed in the interior of the landfill to define the thickness of existing soil cover for use as part of capping.	Approximately 20 test pits will be constructed in the interior portion of the landfill based upon the 150-foot by 300-foot grid.	Each test pit will be constructed utilizing a backhoe. The test pit dimensions shall be approximately 6 feet long by 6 feet wide and 6 feet deep.	No samples will be collected for chemical analysis.

Table 1-1 (continued)

SONIA ROAD LANDFILL
REMEDIAL INVESTIGATION AND FEASIBILITY STUDY
REMEDIAL INVESTIGATION FIELD PROGRAM SUMMARY

Program Element	Rationale	Number of Samples	Equipment	Analysis
On-site Test Borings	Test borings will be constructed in order to define depth of waste in the landfill and below groundwater.	Four borings will be constructed to a depth of approximately 50 feet or to the depth of waste. Split spoon samples will be collected at the water table interface and the base of the borehole to confirm the depth of waste. Four samples will be collected (one from each boring with at least two at the water table interface) for chemical analysis.	Borings will be constructed using 4 1/4 I. D. inch hollow stem augers. Soil samples will be collected using split spoon samplers.	Two samples will be analyzed for TCL + 30, TAL parameters and two samples will be analyzed for TCLP parameters.
Upgradient Monitoring Wells (MW-9, MW-10, MW-11, MW-12)	Four monitoring well clusters (shallow, intermediate and deep) will be installed upgradient of the site and immediately downgradient of the industrial areas located north and northwest of the landfill in order to further define upgradient groundwater quality and identify other sources of contamination.	Twelve monitoring wells will be installed upgradient of the site, and one sample will be collected from each well.	Monitoring wells will be constructed of 4-inch diameter, flush joint, schedule 40 PVC with 10 slot stainless steel screen. The 15-foot screen will be placed 5 feet above the water table in the shallow well. All remaining wells will have 10-foot screens. Deep wells will be screened just above the confining unit.	None.

Table 1-1 (continued)

SONIA ROAD LANDFILL
REMEDIAL INVESTIGATION AND FEASIBILITY STUDY
REMEDIAL INVESTIGATION FIELD PROGRAM SUMMARY

Program Element	Rationale	Number of Samples	Equipment	Analysis
Perimeter Hydropunch/ Groundwater Screening	Groundwater samples will be collected along the southern border of the landfill utilizing a Hydropunch sampler in order to locate the perimeter monitoring wells.	Seven Hydropunch screening points will be installed along the southern perimeter of the landfill. The screening point will be based on the 300-foot grid developed for the site. Groundwater samples will be collected from each screening point at 10 foot intervals initiating at the water table to the top of the Gardiners clay (approximately 120 feet) for a total of about 70 samples.	Groundwater samples will be collected directly from the Hydropunch sampler.	Each sample will be analyzed in the field using a portable gas chromatograph. Analysis for select volatile organic compounds of concern will be conducted. Ten percent of the samples will be analyzed in an off-site laboratory for confirmatory TCL volatile organics.

Table 1-1 (continued)

SONIA ROAD LANDFILL
REMEDIAL INVESTIGATION AND FEASIBILITY STUDY
REMEDIAL INVESTIGATION FIELD PROGRAM SUMMARY

Program Element	Rationale	Number of Samples	Equipment	Analysis
Perimeter Monitoring Wells (MW-2 through MW-8)	Monitoring wells will be installed immediately downgradient and upgradient of the site in order to supplement the existing monitoring wells and to further define downgradient groundwater quality. Clusters will be installed at up to four new locations (shallow, intermediate and deep) and three existing locations will be completed with the addition of three deep and two intermediate wells. The shallow wells will be installed at the water table and deep wells will be constructed to the depth of the Gardiners Clay (approximately 120 feet). Intermediate depth wells will be installed dependent upon the results of the Hydropunch screening. The final locations of clusters will be predicated based upon the results of the Hydropunch results. For planning purposes, up to four clusters screened throughout the aquifer may be installed.	Up to 17 monitoring wells will be installed upgradient and downgradient along the site perimeter, in addition to the four existing wells, and one sample will be collected from each well.	Monitoring wells will be constructed of 4 inch diameter, flush joint, schedule 40 PVC with 10 slot stainless steel screen. The 15-foot screen will be placed 5 feet above the water table for the shallow wells. All remaining wells will have 10-foot screens. Deep wells will be screened just above the confining unit.	None.

Table 1-1 (continued)

SONIA ROAD LANDFILL
REMEDIAL INVESTIGATION AND FEASIBILITY STUDY
REMEDIAL INVESTIGATION FIELD PROGRAM SUMMARY

Program Element	Rationale	Number of Samples	Equipment	Analysis
Downgradient Monitoring Wells (MW-13 and MW-14)	Two monitoring well clusters (shallow, intermediate and deep) will be installed downgradient of the site in order to characterize and further define downgradient ground water quality.	Six monitoring wells will be installed downgradient of the site. Attempts will be made to utilize existing SCDHS intermediate wells. One sample will be collected from each well.	Monitoring wells will be constructed of 4-inch diameter flush joint, schedule 40 PVC with 10 slot stainless steel screen. The 15-foot screen will be placed 5 feet above the water table in the shallow wells. All remaining wells will have 10-foot screens. Deep wells will be screened above the confining unit.	None.
Monitoring Well Groundwater Sampling	Groundwater samples will be collected in order to characterize groundwater quality.	Groundwater samples will be collected from the 35 wells installed as part of this investigation and from the four existing wells for a total of 39 samples.	Monitoring wells will be purged of three to five well volumes prior to collection of the samples. Wells will be purged with a submersible pump or disposable polyethylene bailer. Purged water will be monitored for pH, conductivity, temperature and turbidity. Once each of the parameters has stabilized, groundwater samples will be collected. Groundwater samples will be obtained utilizing disposable polyethylene bailers.	Each sample will be analyzed for TCL +30, TAL parameters and leachate parameters as identified on the Part 360 Expanded Parameter List.
Water Level Measurements	Water level measurements will be collected to evaluate groundwater flow on-site and off-site.	Two rounds of groundwater level measurements will be collected during the investigation.	An electronic water level indicator will be utilized to obtain the groundwater level measurements.	None.

Table 1-1 (continued)

SONIA ROAD LANDFILL
REMEDIAL INVESTIGATION AND FEASIBILITY STUDY
REMEDIAL INVESTIGATION FIELD PROGRAM SUMMARY

Program Element	Rationale	Number of Samples	Equipment	Analysis
Public/Private Well Survey	The survey will be conducted in order to determine potential receptors that may be exposed to groundwater contamination that may be migrating from the landfill.	None.	None.	None.

west of the site on Corbin Avenue. One gate is located on the southeast side of the site on Deer Park Avenue and one gate is located on the east side of the site off of Sonia Road. Access for Dvirka and Bartilucci Consulting Engineers and its subconsultants will be made by the Town of Islip.

All equipment, materials and supplies used for drilling and excavation of test pits/trenches, equipment and supplies for decontamination will be stored on the western portion of the site (see Figure 1-1). The decontamination area and soil cuttings, decontamination, development and purge water disposal area will also be located on this portion of the site.

1.2 Base Mapping

The existing aerial photograph, and topographic survey and mapping of the site will be used to the furthest extent possible.

All site survey controls, topographic features and topography, and other requisite information, as applicable, will be verified by a New York State Licensed Surveyor. A final base map for the site and environs will be prepared for use with the Geographic Information System (GIS) program.

The base map will be prepared at various scales for report and presentation purposes. All mapping will be in the New York State coordinate system.

1.3 Grid Network Survey

A site-wide grid system will be set up based on a 300-foot grid (see Figure 1-1). The grid network will be used to locate soil borings, test pits and conduct the soil vapor/methane gas survey. The grid network survey will be conducted by a New York State Licensed Surveyor and will be incorporated into the base map prepared as described in Section 1.2 above. The grid will be established by surveying and staking the locations prior to initiation of the field program. All sample locations will be located based on this grid system.

1.4 Soil Vapor/Explosive Gas Survey and Sampling

Due to recent historical data indicating elevated levels of methane in several perimeter monitoring wells on the landfill and the need to obtain current information on landfill gas emissions for on-site activities and future landfill gas venting, a soil gas survey will be conducted on-site to monitor for the presence of volatile organic compounds (VOCs) and methane. The information obtained from the survey will aid in design of the capping system and emission control system, if necessary, and will address possible community health concerns raised during the investigation.

Based on the grid system presented on Figure 1-1, approximately 30 node points will be screened for soil vapors. The majority of the screening points are located toward the center of the landfill where soil vapor levels are likely to be elevated. At each node point, a stainless steel probe with a removable inner rod will be driven into the ground to a depth of 3 feet using a slide hammer or other hydraulically or pneumatically driven hammers. The inner rod prevents soil from entering the probe during the installation process. Soil gas measurements will be taken from a depth of 3 feet to reduce the effect of surface contamination, changes in barometric pressure, temperature and precipitation. Upon reaching the completion depth, the inner rod will be removed and a stainless steel cap will be placed immediately on the top of the steel probe. Total VOC and methane measurements will be obtained by use of a photoionization detector (which does not detect methane) and an explosive gas meter. If elevated levels are detected on the PID, up to six samples will be collected from the probes using a vacuum pump and sorbent tubes. Samples will be analyzed for VOCs in accordance with USEPA/600/4-89/017 method T01/T02.

Specific measurement procedures are provided in Section 2.0. During soil gas measurement, site-specific information regarding temperature, wind direction, wind speed, barometric pressure and humidity will be obtained. The survey will be conducted during a period of low barometric pressure to simulate "worst case" conditions.

1.5 Surface Soil Sampling

In an attempt to address concerns identified by the New York State Department of Health regarding potential surficial contamination, four surface soil samples will be collected on-site. At least two of the samples will be collected in the vicinity of the former ballfields on the eastern portion of the site, and the other two samples will be collected on the western portion of the site. Three additional samples will be collected off-site (one upgradient and two downgradient) to obtain off-site surface soil quality information (see Figure 1-1).

Each sample will be collected from 0 to 2 inches below ground surface utilizing a disposable polyethylene scoop. Each sample will be analyzed for Target Compound List (TCL) +30 and Target Analyte List (TAL) parameters.

1.6 Test Trench Construction

In order to define the limits of waste approximately 30 test trenches will be constructed on-site. As shown on Figure 1-1, the test trenches will be constructed around the perimeter of the site to determine/confirm the limits of waste determined during this investigation through an evaluation of historical aerial photographs. In addition, this perimeter test trench program will define the depth of waste for purposes of waste consolidation to reduce the cost of capping. Based on experience with similar projects, generally a depth of waste to 4-6 feet is a break even point for capping versus consolidation.

Test trenches provide a cost-effective alternative to drilling on sites such as this, that are anticipated to require shallow subsurface characterization. Test trench excavation will allow for greater and quicker visual observation of the subsurface soil than borings. Test trenches for determination of the limits of waste will be constructed every 150 feet around the landfill perimeter and will be extended in length as necessary to determine the limits of the fill material in that area. Test trenches will be excavated to a depth of approximately 10 feet and will be approximately 20 feet long and 3 feet wide. No samples are proposed to be collected for chemical analysis from these test trenches. The trenches will be logged by a qualified geologist on test pit/trench logs, and observations regarding color, odor and PID/explosive gas measurements, will be made. Observations for elevated levels on the PID/explosive gas meter will be made in order to assess levels of health and safety protection. Refer to the Health and Safety Plan (Volume III) for specific Health and Safety Procedures to be undertaken as part of this investigation. Community air monitoring will also be conducted during all intrusive activities. Refer to Appendix A for the Community Air Monitoring Plan.

The protocol for test trench excavation and backfill is the following:

- Soil from the top 1 to 2 feet of the test pit/trench will be removed and placed separately.
- If drums or significantly elevated organic vapor readings are encountered during test pit/trench construction, deeper excavation will be terminated.
- Excavated soil and waste material will be replaced in reverse order of removal.
- Records of excavation and sample collection will be maintained (see Section 2.0).
- Final backfill and grading will use the soil removed from the surface of the test pit/trench.

The entire backhoe will be decontaminated upon arrival at and the departure from the Sonia Road Landfill. Decontamination is not planned between test trenches. Clean soil from the surrounding area will be placed on top of the trench, if necessary.

1.7 Test Pit Construction

In order to provide information on existing cover material, approximately 20 test pits will be constructed on-site. As shown on Figure 1-1, the test pits will be constructed in the center portion of the landfill to determine the nature and thickness of the existing cover material for use in construction of the final remedial action.

Test pits will be excavated to a depth of approximately 6 feet and will be approximately 6 feet wide and 6 feet long. No samples are proposed to be collected for chemical analysis from these test pits. The test pits will be logged by a qualified geologist on test pit logs, and observations regarding color, odor and PID/explosive gas measurements will be made. Observations for elevated levels on the PID/explosive gas meter will be made in order to assess levels of health and safety protection. (Refer to Health and Safety Plan and Community Air Monitoring Plan.)

The protocol for test trench excavation noted in the above section will be used for test pit excavation. The backhoe will only be decontaminated upon arrival at and departure from the Sonia Road Landfill.

1.8 Soil Borings, Hydropunch and Monitoring Well Installation

1.8.1 Hydropunch Installation and Sampling

In order to evaluate the most appropriate locations and intermediate screen depths for monitoring wells to be installed along the southern perimeter of the landfill, seven hydropunch screening points will be installed (see Figure 1-1). The screening points will be installed at an approximately 300 foot spacing interval. Up to 10 groundwater samples will be collected from each screening point. Samples will be collected every 10 feet starting from the water table (approximately 20 feet below ground surface) and terminating at the top of the Gardiners Clay (approximately 120 feet below ground surface). The borings will be constructed using 4 1/4 inch hollow stem augers. Once the boring has been constructed to the water table, the Hydropunch sampler will be placed in the borehole and driven approximately four feet into the sample interval. The sampler will be retracted to expose the decontaminated Hydropunch screen and allow groundwater to enter the sampler. After allowing sufficient time to pass to allow the groundwater to enter the sampler, the sampler will be brought to the surface and groundwater will be transferred to appropriate sample container. The Hydropunch boreholes will be constructed without the addition of any potable water to the borehole during drilling. Should running sands be encountered, the addition of water to maintain positive head inside the borehole will only be used with the approval of the NYSDEC project manager.

Groundwater samples will be collected and screened on-site with a portable Gas Chromatograph (GC). Based on available information from the NYSDEC Geoprobe sampling program conducted at the site in 1994, elevated levels of vinyl chloride, chloroethane, 1,1-dichloroethene, 1,1-dichloroethane, 1,2-dichloroethene, 1,1,1-trichloroethane, trichloroethene, tetrachloroethene, benzene, chlorobenzene and xylene have been detected. The portable GC will be calibrated to detect each of these constituents. This will also allow for quantification of these constituents. However, if the portable GC indicates the presence of elevated levels of constituents other than those listed above, these constituents cannot be quantified in the field. At least 10 percent, or up to 10 of the samples collected, will be shipped to an off-site laboratory for confirmatory analysis. These samples will be analyzed for TCL volatile organics. If non-quantifiable constituents are detected on the GC, the sample can be forwarded to the off-site laboratory for analysis as one of the 10 samples.

Each sample will also be screened for pH, temperature and conductivity utilizing portable equipment.

In addition to the groundwater samples, split spoon samples will be collected every five feet starting from 100 feet below ground surface to confirm the presence of the Gardiners Clay.

Based on the results of the screening, recommendations for the placement of the southern perimeter mid-depth monitoring wells will be made.

1.8.2 Borehole Construction for On-site Soil Borings

Four soil borings will be constructed on-site to the base of the landfill material, which has been encountered up to 35 feet below ground surface and 15 feet below the water table based on the results of previous investigations, and the reported depth of the sand and gravel mixing operation prior to landfilling (see Figure 1-1). If refuse is encountered, borings will be terminated. These borings are located at nodes on the 300-foot grid. The borings will provide adequate coverage of the site to evaluate, in general, depth of the fill material. Each of the borings will be constructed using 4 1/4 inch hollow stem augers. Split spoon samples will be collected every at the water table interface and the base of the borehole. Borings will provide stratigraphic information on the site, in particular, depth of the waste material.

One sample will be collected from each boring for chemical analysis. Two of the samples collected will be analyzed for TCL +30 parameters and two of the samples will be analyzed for Toxicity Characteristic Leaching Procedure (TCLP) parameters. Two of the samples will be collected just above the water table.

Samples obtained from the split spoons will be observed and logged for physical/geologic characteristics, odors and staining, and screened with a PID. Due to the expected presence of methane, continuous monitoring will be performed with a combustible gas indicator.

All drill cuttings resulting from the installation of the test borings will be managed in accordance with NYSDEC Technical and Administrative Guidance Memorandum (TAGM) No. 4032 - Disposal of Drill Cuttings (see Appendix B). Drill cuttings associated with borings will be replaced into the borehole to the extent possible or disposed on-site in the designated area. Remaining cuttings not able to be placed in the borehole will be disposed of on-site in the designated area.

1.8.3 Borehole Construction for Monitoring Wells

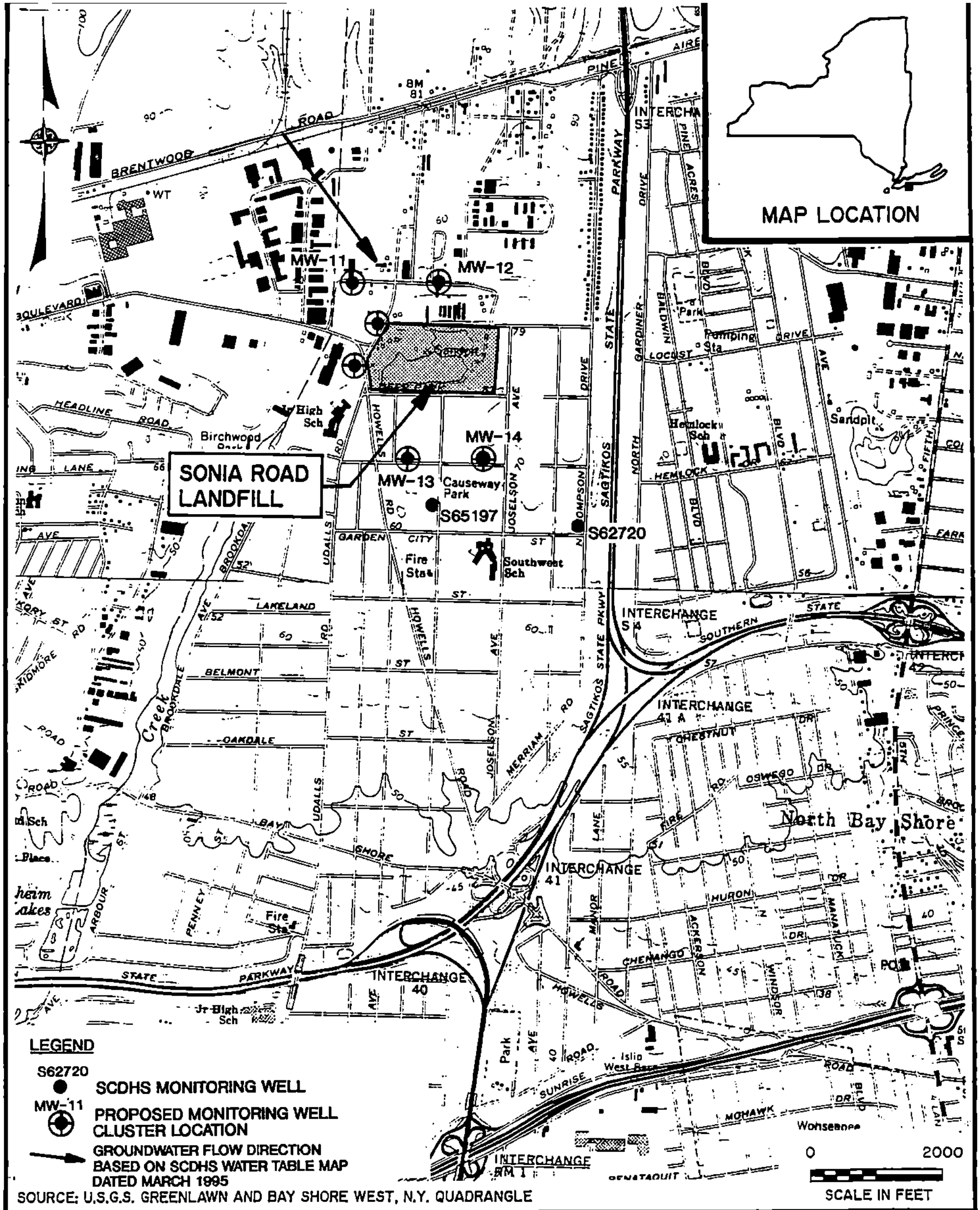
Up to thirty-five groundwater monitoring wells will be installed at thirteen cluster locations during this investigation capable of monitoring upgradient, on-site and downgradient water quality.

Based upon the results of the Hydropunch screening, up to seventeen monitoring wells will be installed along the perimeter of the landfill site to further define immediate upgradient and downgradient groundwater quality (see Figure 1-1 for the location of the wells). Four new clusters will be installed and three existing monitoring well locations will be completed as clusters with the addition of three deep and two intermediate wells.

Each cluster will include one shallow, one intermediate and one deep well. The shallow well will be installed at the water table (approximately 20 feet below ground surface). The intermediate well will be installed to approximately 80 feet below ground surface and the deep well will be installed at the base of the Upper Glacial aquifer at approximately 120 feet below ground surface.

The perimeter monitoring wells will be installed on the edges of the landfill (see Figure 1-1) to aid in providing on-site water quality information, as well as provide long-term groundwater quality information as part of long-term/post-closure monitoring of groundwater. Further definition of the groundwater flow direction will also be obtained from these wells.

Four additional clusters will be installed further upgradient and off-site to better define upgradient groundwater quality and other sources of contamination. Based on the site visit, existing monitoring well, MW-1, may no longer be accessible due to construction and demolition debris processing operations being conducted to the north of the landfill site. Therefore, one of the proposed upgradient monitoring well clusters will replace the well (formerly) at this location and will be installed on the western side of Corbin Avenue. See Figure 1-2 for the approximate locations of the off-site monitoring wells. The exact locations of these off-site wells will be based



SOURCE: U.S.G.S. GREENLAWN AND BAY SHORE WEST, N.Y. QUADRANGLE

ISLIP RESOURCE RECOVERY AGENCY
SONIA ROAD LANDFILL

OFFSITE MONITORING WELL LOCATIONS

Dvirka and Bartilucci
Consulting Engineers
A Division of William F. Cosulich Associates, P.C.

FIGURE 1-2

on the results of the review of information regarding upgradient industrial sources being conducted as part of this investigation.

The focus of the placement of the upgradient off-site wells will be to define groundwater quality migrating from the industrial park north and northwest of the landfill. Since the earlier NYSDEC Geoprobe investigation indicated the highest level of VOCs at the southwestern edge of the landfill, the source of the contamination may be the upgradient industrial area. Little information is available on specific potential sources in this area. As a result, wells will be placed downgradient of this industrial park and upgradient of the landfill.

In order to define downgradient groundwater quality, two monitoring well clusters will be installed downgradient of the landfill (see Figure 1-2). Attempts will be made to obtain downgradient information from SCDHS wells S62720 and S65197. However, the integrity of these wells is unknown.

Each of the monitoring wells will be drilled using 6-5/8 inch hollow stem augers. If difficulties with running sands are encountered which hinder soil sampling, potable water will be added to the hollow stem augers to maintain a positive hydrostatic head.

Similar to the on-site test borings, all drill cuttings resulting from the installation of the monitoring wells will be managed in accordance with NYSDEC TAGM No. 4032. Cuttings generated from the construction of the boreholes will be disposed on the landfill site.

1.8.4 Borehole Soil Sampling

Split spoon samples will be collected every 5 feet in the saturated zone in the deep wells installed at each cluster. Samples will provide stratigraphic information for the study area. Samples obtained from the split spoons will be observed and logged for geologic characteristics. Each sample will be screened with a PID. No samples will be collected for chemical analysis.

1.8.5 Borehole and Monitoring Well Logging

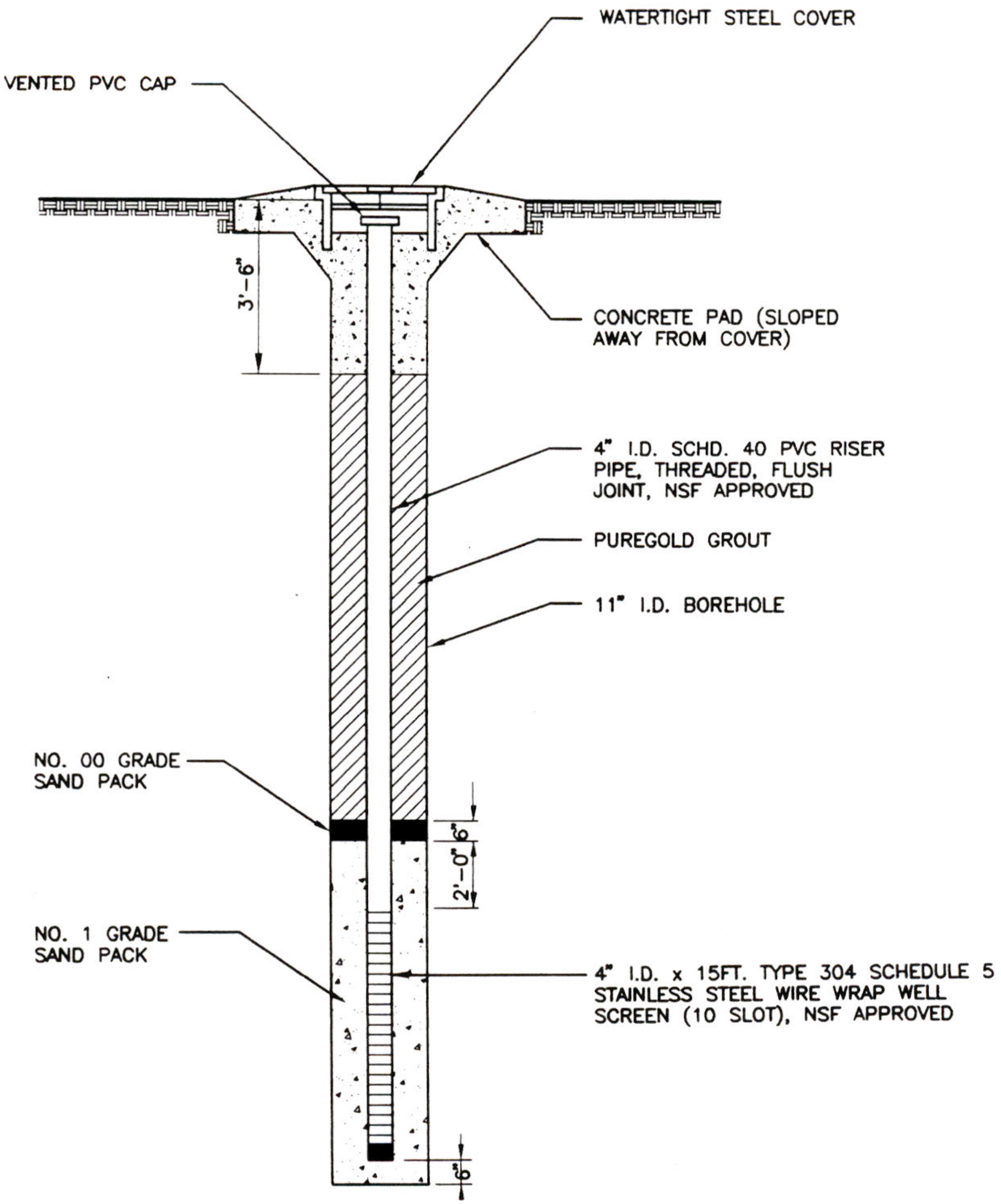
All boreholes and monitoring wells will be logged and documented by a geologist. Notes will be kept in both bound field books and on a Boring Log and Well Construction Log (see Section 2.22 of QA/QC Plan). The Modified Burmeister Classification System and/or appropriate descriptions for inclusion in the GIS/Key Program will be used to describe soil samples recovered from the borings. A Daily Field Activity Report will be completed whenever there are drilling activities (or any other field activities) undertaken as part of the investigation.

1.8.6 Monitoring Well Construction

Prior to constructing the monitoring wells, well casing and screens will be decontaminated as described in Section 2.8 of the QA/QC Plan.

To be consistent with the existing on-site monitoring wells, the wells installed as part of this investigation will be constructed with 4-inch diameter, flush joint, schedule 40 PVC casing and 10 slot stainless steel screen. A 10-foot screen will be installed in each intermediate and deep well. For the shallow wells, a 15-foot screen will be installed to allow for fluctuation in the water table. Approximately 5 feet of the screen will be placed above the water table and the remaining 10 feet below the water table. (See Figures 1-3 and 1-4.)

At the completion of borehole construction and soil sampling, the well screen and riser pipe will be inserted into the hollow stem auger and set at the desired depth. Number one grade sand pack will be tremied into the annular space to a minimum height of 2 feet above the top of the well screen. During this time, the augers will be slowly removed. The well pipe will also be pulled up no more than 1/2 foot to allow sand material to fill the borehole beneath the well screen. Upon completing the placement of the sand pack, a minimum 2-foot thick bentonite pellet or slurry seal will be tremied in the annular space. Bentonite slurry is to be used in the intermediate and deep depth wells. The pellets (if used) will be allowed to swell for a minimum of 1 hour before introducing PUREGOLD bentonite grout in the remaining annular space. If the



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TOWN OF ISLIP
SONIA ROAD LANDFILL

SHALLOW MONITORING WELL CONSTRUCTION DIAGRAM

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Consulting Engineers
A Division of William F. Cosulich Associates, P.C.

FIGURE 1-3

Section 2

2.0 QUALITY ASSURANCE/QUALITY CONTROL PLAN

2.1 Project Identification

Project Name:	Sonia Road Landfill Remedial Investigation and Feasibility Study
Project Requested by:	Town/Agency and New York State Department of Environmental Conservation (NYSDEC)
Project Manager:	Brian Maglienti (Agency) Jim Bologna (NYSDEC) Maria Wright (D&B Consulting Engineers)
Quality Assurance Officer:	Robbin Petrella (D&B Consulting Engineers)
Field Operations Manager:	Caroline Yates (D&B Consulting Engineers)

2.2 Objective and Scope

The purpose of this Remedial Investigation and Feasibility Study (RI/FS) is to collect necessary data to define the source(s) of contamination, determine the nature and extent of contamination, and to develop a cost-effective, environmentally sound, long-term Remedial Action/ Presumptive Remedy for the site, as well as determine the need for additional remedial measures, if necessary. The goals will be achieved by conducting a field sampling program addressing the following matrices: soil vapor, surface and subsurface soil, and groundwater.

The purpose of this Quality Assurance/Quality Control (QA/QC) Plan is to develop and describe the detailed sample collection and analytical procedures that will ensure high quality, valid data for use in the RI/FS.

2.3 Data Usage

The data generated from the field sampling program will be used to monitor for health and safety of workers at the site and the health and safety of persons off-site. As described above, it will also be utilized to determine the nature, extent and source(s) of contamination, prepare a qualitative risk/exposure and environmental assessment and to identify, evaluate, recommend and design a cost-effective environmentally sound, long-term remedial action for the landfill site.

2.4 Sampling Program Design and Rationale

The following presents a general discussion of the sampling to be conducted during the remedial investigation.

- Soil Vapor: Soil vapor samples will be collected during the soil vapor survey to monitor for the presence of volatile organic compounds (VOCs) and methane. Up to six samples will be collected for chemical analysis.
- Surface Soil: Six samples will be collected from areas both on-site and off-site.
- Subsurface Soil: Four subsurface soil samples will be collected from the test borings constructed on-site. One sample will be collected from each of the borings.
- Groundwater: One round of groundwater samples will be collected. The field program will consist of one sample from each of 35 newly installed wells and 4 existing wells adjacent to and in the vicinity of the Sonia Road Landfill. In addition, 70 groundwater samples will be collected from seven hydropunch locations.

For a detailed discussion of the sampling program, and selection of sample matrices and locations, see the Field Sampling Plan (Section 1.0).

2.5 Analytical Parameters

Sample analysis for the surface soil, subsurface soil and groundwater samples collected will consist of the Target Compound List (TCL) +30 substances and Target Analyte List (TAL) identified in the 1995 NYSDEC Analytical Services Protocol (ASP). Groundwater samples will also be analyzed for leachate parameters as identified in the Part 360 Expanded Parameters List. Select subsurface soil samples will be analyzed for Toxicity Characteristic Leaching Procedure (TCLP) parameters. The soil vapor samples will be analyzed for TCL volatile organic compounds.

Tables 2-1 and 2-2 present a summary of the parameters/sample fractions to be analyzed together with the sample location, type of sample, sample matrix, number of samples, frequency of sample collection, type of sample container, method of sample preservation, holding time and analytical method.

2.6 Data Quality Requirements and Assessment

Data quality requirements and assessments are provided in the 1995 NYSDEC ASP, which includes the detection limit for each parameter and sample matrix. Note that quantification limits, estimated accuracy, accuracy protocol, estimate precision and precision protocol are determined by the laboratory and will be in conformance with the requirements of the 1995 NYSDEC ASP, where applicable. Table 2-3 presents a summary of the data quality requirements.

In addition to meeting the requirements provided in the 1995 NYSDEC ASP, the data must also be useful in evaluating the nature, extent and impact of contamination. Data obtained during the remedial investigation will be compared to specific Standards, Criteria and Guidelines (SCGs) as discussed in the RI/FS Work Plan, copies of which are contained in the appendix of the RI/FS Work Plan. The SCGs to be utilized include:

Matrix	SCG
Soil Vapor	Draft New York State Air Guide-1, Guidelines for the Control of Toxic Ambient Air Contaminants, dated 1991.*
Surface and Subsurface Soil	NYSDEC Technical and Administrative Guideline Memorandum (TAGM) HWR-94-4046 for the Determination of Soil Cleanup Objectives and Cleanup Levels, dated January 1994.
Groundwater	Division of Water Technical and Operational Guidance Series (TOGs) (1.1.1) - Ambient Water Quality Standards and Guidance Values, dated October 1993.

* Including complete and HAP Listings, AGCs, SGCs and Air Quality Standards for the Air Guide-1 Software Program.

Table 2-1

SONIA ROAD LANDFILL REMEDIAL INVESTIGATION AND FEASIBILITY STUDY SUMMARY OF MONITORING PARAMETERS

Sample Location	Sample Type	Sample Matrix	Sample Fraction	Number of Samples	Frequency	Container Type/Size/No.	Sample Preservation	Maximum Holding Time*	Analytical Method
On-site	Grab	Soil Vapor	Volatile Organics	6	1	Tenax tube/1	Cool to 4°C	7 days for analysis	EPA/600/4-89/017 Method T01/T02
Site/Study Area	Field Blank	Air	Volatile Organics	1	1	Tenax tube/1	Cool to 4°C	7 days for analysis	EPA/600/4-89/017 Method T01/T02

*Holding time based upon VTSR (Verified Time of Sample Receipt).

Table 2-1 (continued)

SONIA ROAD LANDFILL REMEDIAL INVESTIGATION AND FEASIBILITY STUDY
SUMMARY OF MONITORING PARAMETERS

Sample Location	Sample Type	Sample Matrix	Sample Fraction	Number of Samples	Frequency	Container Type/Size/No.	Sample Preservation	Maximum Holding Time*	Analytical Method
Surface Soil	Grab	Surficial Soil	Volatile Organics	7	1	Glass, amber/ 4 oz/2 ICHEM 200 series or equivalent	Cool to 4°C	7 days for analysis	10/95 NYSDEC ASP Method 95-1
	Grab	Surficial Soil	Base Neutral and Acid Extractable Organics	7	1	Glass, amber/ 150 mL/1 ICHEM 200 series or equivalent	Cool to 4°C	5 days for extraction, 40 days after extraction for analysis	10/95 NYSDEC ASP Method 95-2
	Grab	Surficial Soil	Pesticides/PCBs	7	1	Glass, amber/ 150 mL/1 ICHEM 200 series or equivalent	Cool to 4°C	5 days for extraction, 40 days after extraction for analysis	10/95 NYSDEC ASP Method 95-3
	Grab	Surficial Soil	Metals	7	1	Glass, amber/ 150 mL/1 ICHEM 200 series or equivalent	Cool to 4°C	26 days for Hg analysis, 6 months for analysis of others	10/95 NYSDEC ASP Method 200.7**
	Grab	Surficial Soil	Cyanide	7	1	Glass, amber/ 150 mL/1 ICHEM 200 series or equivalent	Cool to 4°C	12 days for analysis	10/95 NYSDEC ASP Method 335.2

*Holding times based upon VTSR (Verified Time of Sample Receipt).

**and SW-846 Methods for:

	Method
Mercury	7470

Table 2-1 (continued)

SONIA ROAD LANDFILL REMEDIAL INVESTIGATION AND FEASIBILITY STUDY
SUMMARY OF MONITORING PARAMETERS

Sample Location	Sample Type	Sample Matrix	Sample Fraction	Number of Samples	Frequency	Container Type/Size/No.	Sample Preservation	Maximum Holding Time*	Analytical Method
Soil Borings	Grab	Borehole Soil	Volatile Organics	2	1	Glass, clear/ 4 oz./2 ICHEM 200 series or equivalent	Cool to 4°C	7 days for analysis	10/95 NYSDEC ASP Method 95-1
	Grab	Borehole Soil	Base Neutral and Acid Extractable Organics	2	1	Glass, amber/ 150 mL/1 ICHEM 200 series or equivalent	Cool to 4°C	5 days for extraction, 40 days after extraction for analysis	10/95 NYSDEC ASP Method 95-2
	Grab	Borehole Soil	Pesticides/PCBs	2	1	Glass, amber/ 150 mL/1 ICHEM 200 series or equivalent	Cool to 4°C	5 days for extraction, 40 days after extraction for analysis	10/95 NYSDEC ASP Method 95-3
	Grab	Borehole Soil	Metals	2	1	Glass, amber/ 150 mL/1 ICHEM 200 series or equivalent	Cool to 4°C	26 days for Hg analysis, 6 months for analysis of others	10/95 NYSDEC ASP Method 200.7*
	Grab	Borehole Soil	Cyanide	2	1	Glass, amber/ 150 mL/1 ICHEM 200 series or equivalent	Cool to 4°C	12 days for analysis	10/95 NYSDEC ASP Method 335.2

*Holding times based upon VTSR (Verified Time of Sample Receipt)

**and SW-846 Methods for:	Method
Mercury	7470

Table 2-1 (continued)

SONIA ROAD LANDFILL REMEDIAL INVESTIGATION AND FEASIBILITY STUDY
SUMMARY OF MONITORING PARAMETERS

Sample Location	Sample Type	Sample Matrix	Sample Fraction	Number of Samples	Frequency	Container Type/Size/No.	Sample Preservation	Maximum Holding Time*	Analytical Method
Soil Borings	Grab	Borehole Soil	Toxicity Characteristic Leaching Procedure (TCLP)	2	1	Glass/amber/ 150mL/1 ICHEM 200 Sreries or equivalent	Cool to 4°C	5 days for extraction**	USEPA SW-846 Method 1311
	Grab	TCLP Extract	Volatile Organics	2	1	Glass, clear/ 4o mL/3 ICHEM 300 series or equivalent	Cool to 4°C	7 days for analysis after TCLP extraction	10/95 NYSDEC ASP SW-846 Method 8240
	Grab	TCLP Extract	Base Neutral and Acid Extractable Organics	2	1	Glass, amber/ 1L/2 ICHEM 300 series or equivalent	Cool to 4°C	5 days after TCLP extraction for extraction, 40 days after extraction for analysis	10/95 NYSDEC ASP SW-846 Method 8270
	Grab	TCLP Extract	Pesticides/ Herbicides	2	1	Glass, amber/ 1L/2 ICHEM 300 series or equivalent	Cool to 4°C	5 days after TCLP extraction for extraction, 40 days after extraction for analysis	10/95 NYSDEC ASP SW-846 Method 8015 and 8080
	Grab	TCLP Extract	Metals	2	1	Plastic/ 1L/1 ICHEM 300 series or equivalent	HNO, to pH <2 Cool to 4°C	26 days after TCLP extraction for Hg analysis, 6 months for analysis of others	10/95 NYSDEC ASP Method 6010***

*Holding times based upon VTSR (Verified Time of Sample Receipt).

**Holding time for TCLP extraction is 5 days for SVOC and Pest/Herb fraction, 7 days for VOC and 180 days for metals.

***and SW-846 Methods for:

	Method
Mercury	7470

Table 2-1 (continued)

SONIA ROAD LANDFILL REMEDIAL INVESTIGATION AND FEASIBILITY STUDY
SUMMARY OF MONITORING PARAMETERS

Sample Location	Sample Type	Sample Matrix	Sample Fraction	Number of Samples	Frequency	Container Type/Size/No.	Sample Preservation	Maximum Holding Time*	Analytical Method
Monitoring Wells (Thirty-five new wells, four existing wells)	Grab	Groundwater	Volatile Organics	39	1	Glass, clear/ 40 mL/3 ICHEM 300 series or equivalent	Cool to 4°C	7 days for analysis	10/95 NYSDEC ASP Method 95-1
	Grab	Groundwater	Base Neutral and Acid Extractable Organics	39	1	Glass, amber/ 1L/2 ICHEM 300 series or equivalent	Cool to 4°C	5 days for extraction, 40 days after extraction for analysis	10/95 NYSDEC ASP Method 95-2
	Grab	Groundwater	Pesticides/PCBs	39	1	Glass, amber/ 1L/2 ICHEM 300 series or equivalent	Cool to 4°C	5 days for extraction, 40 days after extraction for analysis	10/95 NYSDEC ASP Method 95-3
	Grab	Groundwater	Total Metals***	39	1	Plastic/1L/1 ICHEM 300 series or equivalent	HNO_3 to pH <2 Cool to 4°C	26 days for Hg analysis, 6 months for analysis of others	10/95 NYSDEC ASP Method 200.7**
	Grab	Groundwater	Cyanide	39	1	Plastic/1L/1 ICHEM 300 series or equivalent	NaOH to pH >12 Cool to 4°C	12 days for analysis	10/95 NYSDEC ASP Method 335.2
	Grab	Groundwater	Leachate Parameters	39	1	See Table 2-2	See Table 2-2	See Table 2-2	1995 NYSDEC ASP See Table 2-2

*Holding times based upon VTSR (Verified Time of Sample Receipt).

**and SW-846 Methods for:

	Method
Mercury	7470

***Dissolved metals may be required if the turbidity of the samples is >50 NTUs.

Table 2-1 (continued)

SONIA ROAD LANDFILL REMEDIAL INVESTIGATION AND FEASIBILITY STUDY
SUMMARY OF MONITORING PARAMETERS

Sample Location Method	Sample Type	Sample Matrix	Sample Fraction	Number of Samples	Type/Size/No.	Container Preservation	Sample Holding Time	Maximum Analytical
Hydropunch Locations	Grab	Groundwater	Volatile Organics	70	Glass, clear/ 40 ml/3 ICHEM 300 series or equivalent	Cool to 4°C	7 days after VTSR for analysis	Headspace by Portable GC* Method 601
Hydropunch Locations	Grab	Groundwater	Volatile Organics	10**	Glass, clear/ 40 ml/3 ICHEM 300 series or equivalent	Cool to 4°C	7 days after VTSR for analysis	10/95 NYSDEC ASP Method 95-1

VTSR - Verfiied time of sample receipt at the laboratory

*GC: Gas Chromatograph

**Ten Percent of the hydropunch samples (up to a total of 10) will be sent to a laboratory for confirmatory analysis.

Table 2-1 (continued)

SONIA ROAD LANDFILL REMEDIAL INVESTIGATION AND FEASIBILITY STUDY
SUMMARY OF MONITORING PARAMETERS

Sample Location	Sample Type	Sample Matrix	Sample Fraction	Number of Samples	Frequency	Container Type/Size/No.	Sample Preservation	Maximum Holding Time*	Analytical Method
Site/Study Area	Trip Blank	Water	Volatile Organics	10**	1	Glass, clear/ 40 mL/1 ICHEM 300 series or equivalent	Cool to 4°C	7days for analysis	10/95 NYSDEC ASP Method 95-1

*Holding time based upon VTSR (Verified Time of Sample Receipt).

**One trip blank will accompany each shipment of aqueous samples requiring volatile organic analysis.

Table 2-1 (continued)

SONIA ROAD LANDFILL REMEDIAL INVESTIGATION AND FEASIBILITY STUDY
SUMMARY OF MONITORING PARAMETERS

Sample Location	Sample Type	Sample Matrix	Sample Fraction	Number of Samples	Frequency	Container Type/Size/No.	Sample Preservation	Maximum Holding Time*	Analytical Method
Site/Study Area	Matrix Spike and Matrix Spike Duplicate	Groundwater	Volatile Organics	3**	1	Glass, clear/ 40 mL/3 ICHEM 300 series or equivalent	Cool to 4°C	7 days for analysis	10/95 NYSDEC ASP Method 95-1
	Matrix Spike and Matrix Spike Duplicate	Groundwater	Base Neutral and Acid Extractable Organics	2**	1	Glass, amber/ 1L/2 ICHEM 300 series or equivalent	Cool to 4°C	5 days for extraction, 40 days after extraction for analysis	10/95 NYSDEC ASP Method 95-2
	Matrix Spike and Matrix Spike Duplicate	Groundwater	Pesticides/PCBs	2**	1	Glass, amber/ 1L/2 ICHEM 300 series or equivalent	Cool to 4°C	5 days for extraction, 40 days after extraction for analysis	10/95 NYSDEC ASP Method 95-3
	Matrix Spike and Matrix Spike Duplicate	Groundwater	Metals	2**	1	Plastic/1L/1 ICHEM 300 series or equivalent	HNO_3 to pH <2 Cool to 4°C	26 days for Hg analysis, 6 months for analysis of others	10/95 NYSDEC ASP Method 200.7***
	Matrix Spike and Matrix Spike Duplicate	Groundwater	Cyanide	2**	1	Plastic/1L/1 ICHEM 300 series or equivalent	NaOH to pH >12 Cool to 4°C	12 days for analysis	10/95 NYSDEC ASP Method 335.2
	Matrix Spike and Matrix	Groundwater	Leachate	2**	1	See Table 2-2	See Table 2-2	See Table 2-2	See Table 2-2

*Holding times based upon VTSR (Verified Time of Sample Receipt).

**Two sets of MS/MSD based upon collection of 39 groundwater samples. Three sets of MS/MSD for VOA analysis based on a maximum of 49 GW samples.

***and SW-846 Methods for:

	Method
Mercury	7470

Table 2-1 (continued)

SONIA ROAD LANDFILL REMEDIAL INVESTIGATION AND FEASIBILITY STUDY
SUMMARY OF MONITORING PARAMETERS

Sample Location	Sample Type	Sample Matrix	Sample Fraction	Number of Samples	Frequency	Container Type/Size/No.	Sample Preservation	Maximum Holding Time*	Analytical Method
Soil Borings/ Surface Soil	Matrix Spike and Matrix Spike Duplicate	Borehole Soil/ Surface Soil	Volatile Organics	1**	1	Glass, clear/ 4oz./2 ICHEM 200 series or equivalent	Cool to 4°C	7 days for analysis	10/95 NYSDEC ASP Method 95-1
	Matrix Spike and Matrix Spike Duplicate	Borehole Soil/ Surface Soil	Base Neutral and Acid Extractable Organics	1**	1	Glass, amber/ 150 mL/1 ICHEM 200 series or equivalent	Cool to 4°C	5 days for extraction, 40 days after extraction for analysis	10/95 NYSDEC ASP Method 95-2
	Matrix Spike and Matrix Spike Duplicate	Borehole Soil/ Surface Soil	Pesticides/PCBs	1**	1	Glass, amber/ 150 mL/1 ICHEM 200 series or equivalent	Cool to 4°C	5 days for extraction, 40 days after extraction for analysis	10/95 NYSDEC ASP Method 95-3
	Matrix Spike and Matrix Spike Duplicate	Borehole Soil/ Surface Soil	Metals	1**	1	Glass, amber/ 150 mL/1 ICHEM 200 series or equivalent	Cool to 4°C	26 days for Hg analysis, 6 months for analysis of others	10/95 NYSDEC ASP Method 200.7***
	Matrix Spike and Matrix Spike Duplicate	Borehole Soil/ Surface Soil	Cyanide	1**	1	Glass, amber/ 150 mL/1 ICHEM 200 series or equivalent	Cool to 4°C	12 days for analysis	10/95 NYSDEC ASP Method 335.2

*Holding times based upon VTSR (Verified Time of Sample Receipt).

**One set of MS/MSD based on a maximum of two soil boring samples and seven surface soil samples analyzed for TCL +30 parameters.

***and SW-846 Methods for:

	Method
Selenium	7740
Lead	7421
Thallium	7841
Mercury	7470
Arsenic	7060

Table 2-1 (continued)

SONIA ROAD LANDFILL REMEDIAL INVESTIGATION AND FEASIBILITY STUDY
SUMMARY OF MONITORING PARAMETERS

Sample Location	Sample Type	Sample Matrix	Sample Fraction	Number of Samples	Frequency	Container Type/Size/No.	Sample Preservation	Maximum Holding Time*	Analytical Method
Soil Borings/ Surface Soil	Matrix Spike and Matrix Spike Duplicate	Borehole Soil	Toxicity Characteristic Leaching Procedure (TCLP)	1****	1	Glass/amber/ 150mL/1 ICHEM 200 Sreries or equivalent	Cool to 4°C	5 days for extraction**	USEPA SW-846 Method 1311
	Matrix Spike and Matrix Spike Duplicate	TCLP Extract	Volatile Organics	1	1	Glass, clear/ 4o mL/3 ICHEM 300 series or equivalent	Cool to 4°C	7 days for analysis after TCLP extraction	10/95 NYSDEC ASP SW-846 Method 8240
	Matrix Spike and Matrix Spike Duplicate	TCLP Extract	Base Neutral and Acid Extractable Organics	1	1	Glass, amber/ 1L/2 ICHEM 300 series or equivalent	Cool to 4°C	5 days after TCLP extraction for extraction, 40 days after extraction for analysis	10/95 NYSDEC ASP SW-846 Method 8270
	Matrix Spike and Matrix Spike Duplicate	TCLP Extract	Pesticides/ Herbicides	1	1	Glass, amber/ 1L/2 ICHEM 300 series or equivalent	Cool to 4°C	5 days after TCLP extraction for extraction, 40 days after extraction for analysis	10/95 NYSDEC ASP SW-846 Method 8015 and 8080
	Matrix Spike and Matrix Spike Duplicate	TCLP Extract	Metals	1	1	Plastic/ 1L/1 ICHEM 300 series or equivalent	HNO_3 to pH <2 Cool to 4°C	26 days after TCLP extraction for Hg analysis, 6 months for analysis of others	10/95 NYSDEC ASP Method 200.7***

*Holding times based upon VTSR (Verified Time of Sample Receipt).

**Holding time for TCLP extraction is 5 days for SVOC and Pest/Herb fraction, 7 days for VOC and 180 days for metals.

***and SW-846 Methods for: Method
Mercury 7470

****One set of MS/MSD based on two soil boring samples to be analyzed for TCLP parameters

Table 2-1 (continued)

SONIA ROAD LANDFILL REMEDIAL INVESTIGATION AND FEASIBILITY STUDY
SUMMARY OF MONITORING PARAMETERS

Sample Location	Sample Type	Sample Matrix	Sample Fraction	Number of Samples	Frequency	Container Type/Size/No.	Sample Preservation	Maximum Holding Time*	Analytical Method
Equipment Bank	Grab	Water	Volatile Organics	3	1	Glass, clear/ 40 mL/3 ICHEM 300 series or equivalent	Cool to 4°C	7 days for analysis	10/95 NYSDEC Method 95-1
	Grab	Water	Base Neutral and Acid Extractable Organics	3	1	Glass, amber/ 1L/2 ICHEM 300 series or equivalent	Cool to 4°C	5 days for extraction, 40 days after extraction for analysis	10/95 NYSDEC ASP Method 95-2
	Grab	Water	Pesticides/PCBs	3	1	Glass, amber/ 1L/2 ICHEM 300 series or equivalent	Cool to 4°C	5 days for extraction, 40 days after extraction for analysis	10/95 NYSDEC ASP Method 95-3
	Grab	Water	Total Metals	3	1	Plastic/1L/1 ICHEM 300 series or equivalent	HNO_3 to pH <2 Cool to 4°C	26 days for Hg analysis, 6 months for analysis of others	10/95 NYSDEC ASP Method 200.7***
	Grab	Water	Cyanide	3	1	Plastic/1L/1 ICHEM 300 series or equivalent	NaOH to pH >12 Cool to 4°C	12 days for analysis	10/95 NYSDEC ASP Method 335.2

*Holding times based upon VTSR (Verified Time of Sample Receipt).

**One equipment blank will be collected off a split spoon each day samples are collected.

***and SW-846 Methods for:	Method
Mercury	7470

Table 2-2

LEACHATE PARAMETERS
METHOD OF ANALYSIS, PRESERVATION AND HOLDING TIMES*

Parameter	Method of Analysis	Preservation**	Container	Holding Time****
Ammonia	Method 350.3	H_2SO_4 to pH <2; Cool to 4°C	Plastic or Glass	26 days
Total Organic Carbon	Method 415.1	H_2SO_4 to pH <2; Cool to 4°C	Plastic or Glass	26 days
Total Dissolved Solids	Method 160.1	Cool to 4°C	Plastic or Glass	5 days
Alkalinity	Method 310.1	Cool to 4°C	Plastic or Glass	12 days
Chloride	Method 325.3	Cool to 4°C	Plastic or Glass	26 days
pH	Method 150.1	None Required	Glass	Field Measurement
Specific Conductance	Method 120.1	None Required	Glass	Field Measurement
Total Kjedahl Nitrogen	Method 351.3	H_2SO_4 to pH <2; Cool to 4°C	Plastic or Glass	26 days
Nitrate	Method 352.1	H_2SO_4 to pH <2; Cool to 4°C	Plastic or Glass	26 days
BOD (5-day)	Method 405.1	Cool to 4°C	Plastic or Glass	24 hours
COD	Method 410.1	H_2SO_4 to pH <2; Cool to 4°C	Plastic or Glass	26 days
Sulfate	Method 375.4	Cool to 4°C	Plastic or Glass	26 days
Chromium (hexavalent)	Method 218.5	Cool to 4°C	Plastic or Glass	24 hours
Color	Method 110.2	Cool to 4°C	Plastic or Glass	24 hours
Hardness (total)	Method 130.2	HNO_3 to pH <2;	Plastic or Glass	6 months

Table 2-2 (continued)

LEACHATE PARAMETERS
METHOD OF ANALYSIS, PRESERVATION AND HOLDING TIMES*

Parameter	Method of Analysis	Preservation**	Container	Holding Time****
Turbidity	Method 180.1	None required	Plastic or Glass	Field Measurement
Boron	Method 212.3	None required	Plastic	26 days
Eh	---	None required	Glass	Field Measurement
Phenol	Method 420.1	H_2SO_4 to pH <2 Cool to 4°C	Glass	26 days
Bromide	Method 320.1	Cool to 4°C	Plastic or Glass	26 days

*Taken from the 1995 NYSDEC ASP.

**For soil samples: preservation is cool to 4°C only.

***Only required for expanded parameter list.

****Holding Times based on VTSR (Validated Time of Sample Receipt)

Table 2-3

SONIA ROAD LANDFILL REMEDIAL INVESTIGATION AND FEASIBILITY STUDY
DATA QUALITY REQUIREMENTS

Parameter	Sample Matrix	CRDL* (ug/l)	Estimated Accuracy	Accuracy Protocol	Estimated Precision	Precision Protocol
Volatile Organics	Liquid Solid	5-10 5-10	0.87 - 2.48 ug/l	Vol. IB, Chapter 4, Method 8240, Table 7	0.11 - 4.00 ug/l	Vol. IB, Chapter 4, Method 8240, Table 7
Base Neutrals	Liquid Solid	10-50 330-1600	0.29 - 1.23 ug/l	Vol. IB, Chapter 4, Method 8240, Table 7	0.13 - 1.05 ug/l	Vol. IB, Chapter 4, Method 8270, Table 7
Acid Extractables	Liquid Solid	10-50 330-1600	0.29 - 1.23 ug/l	Vol. IB, Chapter 4, Method 8240, Table 7	0.13 - 1.055 ug/l	Vol. IB, Chapter 4, Method 8270, Table 7
Pesticides/PCBs	Liquid Solid	0.5-1.0 8.0-160	0.69 - 10.79 ug/l	Vol. IB, Chapter 4, Method 8080, Table 4	0.16 - 3.50 ug/l	Vol. IB, Chapter 4, Method 8080, Table 4
Metals (except cyanide)	Liquid Solid	0.2-5000 0.2-5000	--	Vol. IA, Chapter 3, Method 6010**, Table 4	--	Vol. IA, Chapter 3, Method 6010**, Table 4
Cyanide	Liquid Solid	10 10	85% - 102% of recovery	Vol. IC, Chapter 5, Method 9010	±0.02 mg/l	Vol. IC, Chapter 5, Method 9010

*Contract Required Detection Limits

**and SW-846 Methods for:

	Method
Mercury	7470

Table 2-3 (continued)

SONIA ROAD LANDFILL REMEDIAL INVESTIGATION AND FEASIBILITY STUDY
DATA QUALITY REQUIREMENTS
OBJECTIVES FOR PRECISION, ACCURACY, AND COMPLETENESS

Matrix/Parameter	Precision (%)	Accuracy (%)
Soils		
VOCs[(a)]	See Table 2-2a	See Table 2-2a
Extractables[(a)]	See Table 2-2b	See Table 2-2b
Pesticides/PCBs	See Table 2-2c	See Table 2-2c
Metals[(b)]	± 25	75-125
Groundwater		
VOCs[(a)]	See Table 2-2a	See Table 2-2a
Extractables[(a)]	See Table 2-2b	See Table 2-2b
Pesticides/PCBs	See Table 2-2c	See Table 2-2c
Metals[(b)]	± 25%	75-125

NOTES:

(a) Accuracy will be determined as percent recovery of surrogate spike compounds and matrix spike compounds. Surrogate and matrix spike compounds for VOCs, extractables, and pesticides/PCBs are listed in Tables 2-2a, 2-2b and 2-2c, respectively. Precision will be estimated as the relative standard deviation of the percent recoveries per matrix.

(b) Accuracy will be determined as percent recovery of matrix spikes when appropriate or the percent recovery of a QC sample if spiking is inappropriate. Precision will be determined as relative percent difference of matrix spike duplicate samples, or duplicate samples if spiking is inappropriate.

(c) Precision will be determined as the average percent difference for replicate samples. Accuracy will be determined as the percent recovery of matrix spike samples or laboratory control samples, as appropriate.

Source: NYSDEC ASP

Table 2-3a

SONIA ROAD LANDFILL REMEDIAL INVESTIGATION AND FEASIBILITY STUDY
DATA QUALITY REQUIREMENTS
ACCURACY REQUIREMENTS FOR VOC

	Spike Recovery Limits (%)	
	Water	Low/Medium Soil
Surrogate Compound		
Toluene-d8	88-110	84-138
4-Bromofluorobenzene	86-115	59-113
1,2,Dichlorethane-d4	76-114	70-121
Matrix Spike Compound		
1,1-Dichloroethene	61-145	59-172
Trichloroethane	71-120	62-137
Chlorobenzene	75-130	60-133
Toluene	76-125	59-139
Benzene	76-127	66-142

Source: NYSDEC ASP

Table 2-3b

SONIA ROAD LANDFILL REMEDIAL INVESTIGATION AND FEASIBILITY STUDY
DATA QUALITY REQUIREMENTS
OBJECTIVES FOR PRECISION AND ACCURACY OF EXTRACTABLE COMPOUNDS BASED UPON RECOVERY OF SURROGATE AND MATRIX SPIKE COMPOUNDS*

Surrogate Compounds	Matrix	Precision	Accuracy %
d5-Nitrobenzene	Water	≤ 20	35-114
	Solid	≤ 25	23-120
2-Fluorobiphenyl	Water	≤ 20	43-116
	Solid	≤ 25	30-115
d14-Terphenyl	Water	≤ 20	33-141
	Solid	≤ 25	18-137
d5-Phenol	Water	≤ 20	10-110
	Solid	≤ 25	24-113
2-Fluorophenol	Water	≤ 20	21-110
	Solid	≤ 25	25-121
2,4,6-Tribromophenol	Water	≤ 20	10-123
	Solid	≤ 25	19-122
2-Chlorophenol-d4 (Advisory)	Water	≤ 20	33-110
	Solid	≤ 25	20-130
1,2-Dichlorobenzene-d4 (Advisory)	Water	≤ 20	16-110
	Solid	≤ 25	20-130

Table 2-3b (continued)

SONIA ROAD LANDFILL REMEDIAL INVESTIGATION AND FEASIBILITY STUDY
DATA QUALITY REQUIREMENTS
OBJECTIVES FOR PRECISION AND ACCURACY OF EXTRACTABLE COMPOUNDS BASED UPON RECOVERY OF SURROGATE AND MATRIX SPIKE COMPOUNDS*

	Matrix	Precision	Accuracy %
Matrix Spike Compounds			
1,2,4-Trichlorobenzene	Water	≤ 20	39-98
	Solid	≤ 25	38-107
Acenaphthene	Water	≤ 20	46-118
	Solid	≤ 25	31-137
2,4-Dinitrotoluene	Water	≤ 20	24-96
	Solid	≤ 25	28-89
Pyrene	Water	≤ 20	26-127
	Solid	≤ 25	35-142
N-Nitroso-Di-n-Propylamine	Water	≤ 20	41-116
	Solid	≤ 25	41-126
1,4-Dichlorobenzene	Water	≤ 20	36-97
	Solid	≤ 25	28-104
Pentachlorophenol	Water	≤ 20	9-103
	Solid	≤ 25	17-109
Phenol	Water	≤ 20	12-110
	Solid	≤ 25	26-90
2-Chlorophenol	Water	≤ 20	27-123
	Solid	≤ 25	25-102

Table 2-3b (continued)

SONIA ROAD LANDFILL REMEDIAL INVESTIGATION AND FEASIBILITY STUDY
DATA QUALITY REQUIREMENTS
OBJECTIVES FOR PRECISION AND ACCURACY OF EXTRACTABLE COMPOUNDS BASED UPON RECOVERY OF SURROGATE AND MATRIX SPIKE COMPOUNDS*

	Matrix	Precision	Accuracy %
Matrix Spike Compounds (continued)			
4-Chloro-3-methylphenol	Water	≤ 20	23-97
	Solid	≤ 25	26-103
4-Nitrophenol	Water	≤ 20	10-80
	Solid	≤ 25	11-114

* Accuracy will be determined as percent recovery of these compounds. Precision will be estimated as the relative standard deviation of the percent recoveries per matrix.

Source: NYSDEC ASP

Table 2-3c

SONIA ROAD LANDFILL REMEDIAL INVESTIGATION AND FEASIBILITY STUDY
ADVISORY RECOVERY LIMITS
SURROGATE AND MATRIX SPIKE COMPOUNDS
FOR PESTICIDES/PCBs*

	Advisory Recovery Limits (%)	
	Water	Soil/Sediment
Surrogate Compound		
Decachlorobiphenyl	60-150	60-150
Tetrachloro-m-xylene	60-150	60-150
Matrix Spike Compound		
Lindane	56-123	46-127
Heptachlor	40-131	35-130
Aldrin	40-120	34-132
Dieldrin	52-126	31-134
Endrin	56-121	42-139
4,4'-DDT	38-127	23-134

*Samples do not have to be reanalyzed if these recovery limits are not met.

Source: NYSDEC ASP

For groundwater samples collected from the hydropunch screening locations, select volatile organics well be analyzed via headspace. The standard operating procedures (SOP) for the portable GC and headspace analysis, as well as a copy of the training manual provided by Photovac for the Photovac 10S+ GC is provided in Appendix E.

The methods of analysis will be in accordance with the 1995 NYSDEC Analytical Services Protocols. Specific analytical procedures and laboratory QA/QC descriptions are not included in this QA/QC plan, but will be available upon request from the laboratory selected to perform the analyses. The laboratory will be New York State Department of Health (NYSDOH) Environmental Laboratory Approved Program (ELAP) certified for organic and inorganic analyses, and also NYSDOH Contract Laboratory Protocol (CLP) certified. The laboratory selected to perform the analysis shall submit a copy of their Standard Operating Procedures (SOPs) and Quality Assurance/Quality Control (QA/QC) Plan to the Agency.

2.6.1 Data Representativeness

Representative samples will be collected as follows:

- Soil Vapor - Stainless steel probes will be driven up to 3 feet below ground surface. Samples will be collected from decontaminated stainless steel soil probes after the soil vapor has reached equilibrium. Samples will be collected using a personal sampling pump and sorbent tube.

- Surface Soil - Samples will be collected at a depth of 0-2 inches using a sterile polystyrene scoop and/or sterile wooden tongue depressor.

- Subsurface Soil - Samples will be obtained using a decontaminated split spoon sampler during soil boring construction and transferred into the container with a sterile polystyrene scoop and/or wooden tongue depressor.

- Groundwater (Monitoring Well) - Samples will be obtained after the monitoring well has been purged of three to five well casing volumes until turbidity less than 50 NTUs and field measurements (pH, conductivity, temperature and turbidity) have stabilized, or until the well is purged dry (whichever occurs first) and allowed to recharge. Samples will be collected with a dedicated polyethylene bailer.

- Groundwater (Hydropunch) - Samples will be collected upon retrieval of the hydropunch sampler. Samples will be collected directly from the sampler.

- Equipment Calibration - Field equipment used for air monitoring will be calibrated daily before use according to the manufacturer's procedures.

- Equipment Decontamination - Non-sterile sampling equipment will be decontaminated prior to use at each location according to the NYSDEC approved procedures described on Section 2.8 of this QA/QC Plan.

2.6.2 Data Comparability

All data will be presented in the units designated by the methods specified by a NYSDOH ELAP and CLP certified laboratory, and the 1995 NYSDEC ASP. In addition, sample locations, collection procedures and analytical methods from earlier studies will be evaluated for comparability with current procedures/methods.

2.6.3 Data Completeness

The acceptability of 100% of the data is desired as a goal for this project. The acceptability of less than 100% complete data, meeting all laboratory QA/QC protocols/standards, will be evaluated on a case-by-case basis.

2.7 Detailed Sampling Procedures

Three types of environmental samples will be collected from different locations as part of the remedial investigation for the Sonia Road Landfill. These include soil vapor, soil and groundwater. Sample locations will consist of soil vapor points, surface soils, soil borings, monitoring wells and Hydropunch locations. Actual locations are described in Section 1.0. Sampling procedures and equipment are described in this section. A summary of the sampling program, including sample media, locations, depths, equipment, rationale and analytical parameters is provided in Table 2-4. Sample collection will be performed in conformance with Dvirka and Bartilucci Consulting Engineer's Quality Assurance Procedures and Field Investigations Manual

Table 2-4

SUMMARY OF SONIA ROAD LANDFILL RI/FS SAMPLING PROGRAM

Environmental Media	Sample Location	Sample Point	Number of Samples	Sample Depth	Equipment	Rationale	Sample Analysis
Soil Vapor	On-site	Soil vapor survey point	up to 6	3 feet below ground surface	Decontaminated or disposable soil vapor rods/tubing, sorbent tube and personal sampling pump	To monitor for the presence of VOCs and methane	TCL volatile parameters EPA 600/4-88/017
Soil	On-site and Off-site areas	Surface	6	0-2" below surface	Disposable sterile polystyrene scoop and/or wooden tongue depressor	To determine surface soil contamination	TCL +30 and TAL parameters (1995 NYSDEC ASP)
Soil	On-site and off-site areas	Soil Boreholes	4	Depth dependent on field screening. Two of the samples will be collected just above the water table.	Auger, split spoon and wooden tongue depressor	To determine subsurface soil contamination	Two for TCL +30 and TAL parameters (1995 NYSDEC ASP). Two samples for TCLP (USEPA SW-846)
Groundwater	On- and off-site monitoring wells	39 monitoring wells	39	At surface of water in well	Disposable polyethylene bailer (after purge of three volumes)	To determine groundwater contamination	TCL +30 and TAL parameters (1995 NYSDEC ASP) and leachate parameters as per Part 360 Expanded Parameters List
Groundwater	Off-site hydropunch locations	Hydropunch	70	At surface of water in screen	Disposable polyethylene bailer	To determine groundwater contamination	Select volatile organics by portable GC (EPA method 601)
Groundwater	Off-site hydropunch locations	Hydropunch	10	At surface of water in screen	Disposable polyethylene bailer	Confirmation of portable GC analysis	TCL VOA + 10 parameters (1995 NYSDEC ASP)

for Hazardous Waste Sites, which has been prepared in accordance with USEPA and NYSDEC requirements and guidelines.

When collecting soil samples, an attempt will be made to maintain sample integrity by preserving its physical form and chemical composition to as great an extent as possible. First, the mechanism used to collect the soil must be properly decontaminated. An appropriate sampling device (i.e., decontaminated scoop or sterile wooden tongue depressor) will be utilized to transfer the sample into the sample container. The sample should reflect and contain a good representation of the matrix it was taken from.

The sample will be transferred into the sample container as quickly as possible, with no mixing, to ensure that the volatile fraction is not lost.

The materials involved in groundwater sampling are critical to the collection of high quality monitoring information, particularly where the analyses of volatile, pH sensitive or reduced chemical constituents are of interest. Sterile disposable polyethylene bailers will be utilized for this project. The Grundfos pump will be equipped with dedicated polyethylene tubing and pump parts will be polytetrafluoroethylene (PTFE) (e.g., Teflon^R). The pump itself will be decontaminated prior to each location.

There will be several steps taken after the transfer of the soil or water sample into the sample container that are necessary to properly complete collection activities. Once the sample is transferred into the appropriate container, the container will be capped and, if necessary, the outside of the container will be wiped with a clean paper towel to remove excess sampling material. The container will not be submerged in water in an effort to clean it. Rather, if necessary, a clean paper towel moistened with distilled/deionized water will be used.

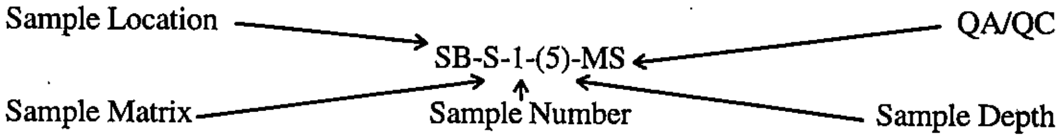
The sample container will then be properly labeled. Information such as sample number, location, collection time and sample description will be recorded in the field log book. Associated forms (e.g., Chain of Custody forms) will then be completed and will stay with the sample. The samples will be packaged in a manner that will allow the appropriate storage temperature (4°C) to be maintained during shipment to the laboratory. Samples will be delivered to the laboratory within 48 hours of collection.

2.7.1 Sample Identification

All samples collected during the field investigation at the Sonia Road Landfill will be labeled with a sample identification code. The code will identify the sample location, sample matrix and series numbers for sample locations with more than one sample. Samples will be labeled according to the following system:

- Sample Location:
    - Soil Vapor "SV"
    - Surface Soil "SS"
    - Soil Boring "SB"
    - Monitoring Well "MW"
    - Hydropunch "HP"
- Sample Matrix:
    - Soil Vapor "V"
    - Soil "S"
    - Water "W"
- Sample Number:
    - For circumstances where more than one sample of the same type and/or from the same location will be collected, a consecutive sample number will be assigned. When more than one sample is collected from a Hydropunch location in a sampling round at different depths, the depth will be indicated on the sample container and in the field log book.
- Quality Assurance/ Quality Control (QA/QC):
    - Matrix Spike "MS"
    - Matrix spike duplicate "MSD"
    - Trip Blank "TB"

Based upon the above sample identification procedures, an example of a sample label may be:



2.7.2 Sample Handling, Packaging and Shipping

All analytical samples will be placed in the appropriate sample containers as specified in the 1995 NYSDEC Analytical Services Protocol (ASP). The holding time criteria identified in the ASP will be followed, as specified in Table 2-1.

Prior to packaging any samples for shipment, the sample containers will be checked for proper identification and compared to the field log book for accuracy. The samples will then be wrapped with a cushioning material and placed in a cooler (or laboratory shuttle) with a sufficient amount of bagged ice or "blue ice" packs in order to keep the samples at 4°C until arrival at the laboratory.

All necessary documentation required to accompany the sample during shipment will be placed in a sealed plastic bag and taped to the underside of the cooler lid. The cooler will then be sealed with fiber (duct) tape, and custody seals will be placed in such a manner that any opening of the cooler prior to arrival at the laboratory can be detected.

All samples will be shipped to ensure laboratory receipt within 48 hours of sample collection in accordance with NYSDEC requirements. The laboratory will be notified prior to the shipment of the samples.

2.7.3 Soil Vapor

1. Be certain that the sample location is noted on Location Sketch (see Section 2.10).
2. Drive the decontaminated stainless steel probe with removable inner rod into the ground to the desired depth.

3. Remove inner rod and immediately replace with a stainless steel cap equipped with a sampling port.

4. Connect new silicon tubing to the probe and the personal sampling pump. Turn on pump and evacuate probe (approximately 2-3 minutes). Turn off pump - remove tubing, cap probe. Allow soil vapor to collect in probe.

5. Label sorbent tube and fill out Sample Information Record and Chain of Custody Form.

6. Connect silicon tubing to probe, tenax tube and personal sampling pump in series. Turn on pump and allow to run in order to collect the designated volume of air.*

7. Shut off pump and disconnect tubing.

8. Place rubber caps on each end of the sample tube and place in shipping container.

9. Extract probe from the ground and decontaminate according to the procedures in Section 2.8.

* The designated volume of air is dependent on the constituents of concern and the type of tube utilized.

2.7.4 Soil (Surface)

1. Be certain that the sample location is noted on Location Sketch (see Section 2.10).

2. Be certain that the sampling equipment (scoop) has been decontaminated, if necessary, utilizing the procedures outlined in Section 2.8.

3. Remove laboratory precleaned sample containers from sample cooler, label container with an indelible marker, fill out Sample Information Record and Chain of Custody Form (see Section 2.10).

4. At the desired location, clear surface debris (e.g., grass, rocks, twigs). Collect an adequate portion of soil from a depth of 0-2 inches using a decontaminated/sterile scoop and/or sterile wooden tongue depressor. Transfer the sample directly into the sample container.

5. Return the sample container to the cooler.

6. If reusable, decontaminate the sampling equipment according to the procedures described in Section 2.8.

2.7.7 Groundwater (Hydropunch)

1. Be certain sample location is noted on Location Sketch (see Section 2.20).
2. Using hydropunch equipment drive/punch screen to desired depth.
3. Remove inner sleeve and lower down decontaminated hydropunch bailer.
4. Remove the laboratory precleaned sample container from the sample cooler, label container with an indelible marker, fill out Sample Information Record and Chain of Custody form.
5. Obtain a volatile organic sample from hydropunch sampler. Gently pour the sample into the sample container taking care not to spill on the outside of the container or overfill container and replace cover on the sample container.
6. Obtain a sample and analyze for field parameters (pH, conductivity, temperature and turbidity).
7. Transfer the sample to the field GC analyst for analysis.
8. Punch down to next depth and repeat items 3 through 7.
9. Decontaminate hydropunch equipment as described in Section 2.8.

2.8 Decontamination Procedures

All field sampling equipment should be sterile and dedicated to a particular sampling point. In instances where this is not possible, a field cleaning (decontamination) procedure will be used in order to reduce the chances of cross-contamination between sample locations. A decontamination station will be established for all field activities. This will be an area located away from the source of contamination so as not to adversely impact the decontamination procedure, but close enough to the sampling area to keep equipment handling to a minimum.

2.8.1 Field Decontamination Procedures

All nondisposable equipment will be decontaminated at appropriate intervals (e.g., prior to initial use, prior to moving to a new sampling location and prior to leaving the site). Different decontamination procedures are used for various types of equipment that perform the field activities as discussed below. When using field decontamination, it is advisable to start sampling in the area of the site with the lowest contaminant probability and proceed through to the areas of highest suspected contamination.

2.8.2 Decontamination Procedure for Drilling/Test Trench/Pit Equipment

All equipment such as drill rigs, backhoes and other mobile equipment should receive an initial cleaning prior to use at the site. The equipment will then be decontaminated prior to leaving the site and each time it returns on-site. Unless otherwise specified and approved, all wash/rinse solutions should be collected and contained on-site. The actual fate of this material will be determined after review of analytical data generated from samples and on-site discharge impacts have been evaluated.

After the initial washing, cleaning may be reduced to those areas that are in close proximity to materials being sampled. Drill rig items such as auger flights, drill rods, and drill bits should be cleaned in between sample locations.

All decontamination generated wastes will be contained in 55-gallon drums and stored in the fenced area.

Drilling equipment will be decontaminated in the following manner:

- Scrub all surfaces thoroughly with nonresidual detergent (alconox) and tap water using a brush to remove particulate matter or surface film. This is necessary in order to remove any solids buildup on the back of the rig, auger flights, drill rods, drilling head, etc. Any loose paint chips, paint flakes and rust must also be removed.

- Steam clean (212°F).

Also, following the general cleaning procedures described above, all downhole/drilling items, such as split spoon samplers, Shelby tubes or any other item of equipment which will come in direct contact with a sample during drilling will be decontaminated by steam cleaning.

2.8.3 Decontamination Procedure for Sampling Equipment

Teflon, PVC, polyethylene, polystyrene and stainless steel sampling equipment decontamination procedures will be the following:

- Wash thoroughly with nonresidual detergent (alconox) and clean potable tap water using a brush to remove particulate matter or surface film.
- Rinse thoroughly with tap water.
- Rinse thoroughly with distilled water.
- Rinse in a well ventilated area with methanol (pesticide grade) and air dry.
- Rinse thoroughly with distilled water and air dry.
- Wrap completely in clean aluminum foil with dull side against the equipment. For small sampling items, such as scoops, decontamination will take place over a drum specifically used for this purpose.

The first step, a soap and water wash, is to remove all visible particulate matter and residual oils and grease. This is followed by a tap water rinse and a distilled/deionized water rinse to remove the detergent. Next, a high purity solvent rinse is designated for trace organics removal. Methanol has been chosen because it is not an analyte of concern in the Target Compound List (TCL). The solvent must be allowed to evaporate and then a final distilled/deionized water rinse is performed. This rinse removes any residual traces of the solvent. The aluminum wrap protects the equipment and keeps it clean until it is used at another sampling location.

2.8.4 Decontamination Procedure for Well Casing/ Screen and Development/Purging Equipment

Field cleaning of well casing and screen should consist of a manual scrubbing to remove foreign material and steam cleaning, inside and out, until all traces of oil and grease are removed. If precleaned certified well casing and screen can be obtained from the manufacturer this would also be acceptable. This material should then be stored in such a manner so as to preserve it in this condition. Special attention to threaded joints may be necessary to remove cutting oil or weld burn residues.

Materials and equipment that will be used within the monitoring well casing for the purposes of well development and purging shall also be decontaminated.

The submersible pump will be decontaminated by the following procedures:

1. Place pump in Alconox and water solution and wash the outside of the pump with a scrub brush.
2. Pump approximately five gallons of Alconox and water solution through the pump.
3. Place pump in bucket of clean water and pump out five gallons of water.
4. Wipe down the cable with deionized water and a paper towel.
5. Decontamination water will be contained in DOT-approved 55-gallon drums.

2.9 Laboratory Sample Custody Procedures

A NYSDOH ELAP and CLP certified laboratory meeting the requirements for sample custody procedures, including cleaning and handling sample containers and analytical equipment will be used. The laboratory's Standard Operating Procedures will be available upon request.

2.10 Field Management Documentation

Proper management and documentation of field activities is essential to ensure that all necessary work is conducted in accordance with the sampling plan and QA/QC Plan in an efficient and high quality manner. Field management procedures include following proper chain of custody procedures to track a sample from collection through analysis, noting when and how samples are to be composited (if required), preparing a Location Sketch, completing Sample Information Record Forms, Chain of Custody Forms and Boring, Well and Test Pit Construction Logs, maintaining a daily Field Log Book, preparing Daily Field Activity Reports, completing Field Change Forms and filling out a Daily Air Monitoring Form. Copies of each of these forms, with the exception of the Air Monitoring Forms, are provided in Section 2.22. Proper completion of these forms and the field log book are necessary to support the consequent actions that may result from the sample analysis. This documentation will support that the evidence was gathered and handled properly.

2.10.1 Location Sketch

Each sampling point shall have its own location sketch (found in Section 2.22) with permanent references, if possible.

2.10.2 Sample Information Record

At each sampling location, the Sample Information Record Form is filled out and maintained including, but not limited to, the following information:

- Site name
- Sample crew
- Sample location
- Field sample identification number
- Date

- Time of sample collection
- Weather conditions
- Temperature
- Sample matrix
- Method of sample collection and any factor that may affect its quality adversely
- Well information (groundwater only)
- Field test results
- Constituents sampled
- Remarks (Sample Compositing Information)

2.10.3 Chain of Custody

The Chain of Custody (COC) is initiated at the laboratory with bottle preparation and shipment to the site. The COC remains with the sample at all times and bears the name of the person assuming responsibility for the samples. This person is tasked with ensuring secure and appropriate handling of the bottles and samples. When the form is complete, it should indicate that there was no lapses in sample accountability.

A sample is considered to be in an individual's custody if any of the following conditions are met:

- It is in the individual's physical possession, or
- It is in the individual's view after being in his or her physical possession, or
- It is secured by the individual so that no one can tamper with it, or
- The individual puts it in a designated and identified secure area.

In general, Chain of Custody Forms are provided by the laboratory contracted to perform the analytical services. At a minimum, the following information shall be provided on these forms:

- Project name and address
- Project number
- Sample identification number
- Date
- Time
- Sample location
- Sample type
- Analysis requested
- Number of containers and volume taken
- Remarks
- Type of waste
- Sampler(s) name(s) and signature(s)
- Spaces for relinquished by/received by signature and date/time.

For this particular study, forms provided by the laboratory will be utilized. A copy of this form is contained in Section 2.22.

The Chain of Custody Form is filled out and signed by the person performing the sampling. The original of the form travels with the sample and is signed and dated each time the sample is relinquished to another party, until it reaches the laboratory or analysis is completed. The field sampler keeps one copy and a copy is retained for the project file. The sample container must also be labeled with an indelible marker with a minimum of the following information:

- Sample number
- Analysis to be performed
- Date of collection
- Compositing information

A copy of the completed form is returned by the laboratory with the analytical results.

2.10.4 Split Samples

Whenever samples are being split with another party, a Receipt for Samples Form must be completed and signed. A copy of this form can be found in Section 2.22. A copy of the COC Form will accompany this form. The present work plan does not provide for split samples.

2.10.5 Field Log Book

Field log books must be bound and should have consecutively numbered, water resistant pages. All pertinent information regarding the site and sampling procedures must be documented. Notations should be made in log book fashion, noting the time and date of all entries. Information recorded in this notebook should include, but not be limited to, the following:

The first page of the log contains the following information:

- Project name and address
- Name, address and phone number of field contact
- Waste generator and address, if different from above

- Type of process (if known), generating waste
- Type of waste
- Suspected waste composition, including concentrations

Daily entries are made for the following information:

- Purpose of sampling
- Location of sampling point
- Number(s) and volume(s) of sample(s) taken
- Description of sampling point and sampling methodology
- Date and time of collection, arrival and departure
- Collector's sample identification number(s)
- Sample distribution and method of storage and transportation
- References, such as sketches of the sampling site or photographs of sample collection
- Field observations, including results of field analyses (e.g., pH, temperature, specific conductance), water levels, drilling logs, and organic vapor and dust readings
- Signature of personnel responsible for completing log entries.

2.10.6 Daily Field Activity Report

At the end of each day of field work, the Field Operations Manager, or designee, completes this form noting personnel on-site and summarizing the work performed that day, equipment, materials and supplies used, results of field analyses, problems and resolutions. This form is then signed and is subject to review. A copy of the Daily Field Activity Report form is contained in Section 2.22.

2.10.7 Field Changes and Corrective Actions

Whenever there is a required or recommended investigation/sampling change or correction, a Field Change Form must be completed by the Field Operations Manager and NYSDEC on-site supervisor, and approved by the D&B Consulting Engineers, Town of Islip and NYSDEC Project Managers.

2.11 Calibration Procedures and Preventive Maintenance

The following information regarding equipment will be maintained at the project site:

1. Equipment calibration and operating procedures which will include provisions for documentation of frequency, conditions, standards and records reflecting the calibration procedures, methods of usage and repair history of the measurement system. Calibration of field equipment will be done daily at the sampling site so that any background contamination can be taken into consideration and the instrument calibrated accordingly.
2. A schedule of preventive maintenance tasks, consistent with the instrument manufacturer's specific operation manuals, that will be carried out to minimize down time of the equipment.
3. Critical spare parts, necessary tools and manuals will be on hand to facilitate equipment maintenance and repair.
4. Copies of the manufacturer's equipment manuals will be kept on-site and are contained in Appendix F.

Calibration procedures and preventive maintenance, in accordance with the NYSDEC 1995 ASP, for laboratory equipment is contained in the laboratory's standard operating procedures (SOP) and is available upon request.

2.12 Performance of Field Audits

During field activities, the QA/QC officer will accompany sampling personnel into the field to verify that the site sampling program is being properly implemented and to detect and define problems so that corrective action can be taken. All findings will be documented and provided to the Field Operations Manager. A copy of Dvirka and Bartilucci's Field Audit form is in Section 2.26.

2.13 Control and Disposal of Contaminated Material

During construction and sampling of the monitoring wells and borings installed during the remedial investigation, possibly contaminated waste, soil and water may be generated from drill cuttings, drilling fluids, decontamination water, development water and purge water. Drill cuttings will be handled in accordance with the NYSDEC Technical and Administrative Guidance Memorandum (TAGM) - No. 4032 - Disposal of Drill Cuttings (see Appendix B). Specifically, all soil and water associated with the wells or borings will be disposed of on-site. Decontamination water and sediment will also be disposed of on-site.

Soil/waste excavated during the test pit program will be placed back into the excavation pit. If necessary, clean soil from areas immediately adjacent to the test pit will be used to cover any waste material and/or contaminated soil returned to the ground. Soil cuttings generated from soil borings will be placed back into the boring (if possible) and, if determined to be necessary, capped with grout. At the present time, there is not provision in the work plan for off-site transport and disposal/treatment of excavated material.

All soiled protective clothing and disposable sampling equipment (i.e., bailers, tongue depressors) will be contained and stored on-site in a secure area. This material will be disposed off-site at the completion of the field program.

2.14 Documentation, Data Reduction and Reporting

A NYSDOH ELAP and CLP certified laboratory meeting requirements for documentation, data reduction and reporting will be used. All data will be cataloged according to sampling locations and sample identification nomenclature which is described in Section 2.7.1 of the QA/QC plan.

NYSDEC "Sample Identification and Analytical Requirement Summary" and "Sample Preparation and Analysis Summary" forms (for VOA Analysis, B/N-A Analysis, Pesticides/PCB Analysis and Inorganic Analysis) will be completed and included with each data package. These forms are contained in Section 2.23. The sample tracking forms are required and supplied by the 1995 NYSDEC ASP.

2.15 Data Validation

As described in Section 2.14 above, summary documentation regarding data validation will be completed by the laboratory using NYSDEC forms contained in Section 2.23, and submitted with the data package.

Data validation will be performed in order to define and document analytical data quality in accordance with NYSDEC requirements that project data must be of known and acceptable quality. The validation processes will be conducted in conformance with the USEPA Contract Laboratory Program National Functional Organic Data Review and Inorganic Analyses while ensuring that the QC requirements for the 95 NYSDEC ASP have been met. Copies of these guidelines are contained in Appendix G.

Because the NYSDEC Analytical Services Protocol is based on the USEPA CLP, the USEPA Functional Guidelines for Evaluating Organics and Inorganics Analyses for the Contract Laboratory Program (CLP) will assist in formulating standard operating procedures (SOPs) for the data validation process. The data validation process will ensure that all analytical requirements specific to this work plan, including the QA/QC Plan are followed. Procedures will address validation of routine analytical services (RAS) results based on the NYSDEC Target Compound List (TCL) for standard sample matrices.

The data validation process will provide an informed assessment of the laboratory's performance based upon contractual requirements and applicable analytical criteria. The report generated as a result of the data validation process will provide a base upon which the usefulness of the data can be evaluated by the end user of the analytical results. The overall level of effort and specific data validation procedure to be used will be equivalent to a "100% validation" of all analytical data in any given data package.

During the review process, it will be determined whether the contractually required laboratory submittals for sample results are supported by sufficient back-up data and QA/QC results to enable the reviewer to conclusively determine the quality of data. Each data package will be checked for completeness and technical adequacy of the data. Upon completion of the review, the reviewers will develop a QA/QC data validation report for each analytical data package.

"Qualified" analytical results for any one field sample are established and presented based on the results of specific QC samples and procedures associated with its sample analysis group or batch. Precision and accuracy criteria (i.e., QC acceptance limits) are used in determining the need for qualifying data. Where test data have been reduced by the laboratory, the method of reduction will be described in the report. Reduction of laboratory measurements and laboratory reporting of analytical parameters shall be verified in accordance with the procedures specified in the NYSDEC program documents for each analytical method (i.e., recreate laboratory calculations and data reporting in accordance with the method specific procedure). The standard operating guideline manuals and any special analytical methodology required are expected to specify documentation needs and technical criteria and will be taken into consideration in the validation process. Copies of the complete data package and the validation report, including the laboratory results data report sheets, with any qualifiers deemed appropriate by the data reviewer, and a supplementary field QC sample result summary statement, will be submitted to the NYSDEC.

Examples of standard organics and inorganics data validation reporting formats and completeness inventory lists which are proposed for use on this project are contained in Section 2.24. These report forms will be modified as necessary and appropriate for any project specific or NYSDEC requirements.

The following is a description of the two-phased approach to data validation planned to be used in this project. The first phase is called checklisting and the second phase is the analytical quality review, with the former being a subset of the latter.

- <u>Checklisting</u> - The data package is checked for correct submission of the contract required deliverables, correct transcription from the raw data to the required deliverable summary forms and proper calculation of a number of parameters.
- <u>Analytical Quality Review</u> - The data package is closely examined to recreate the analytical process and verify that proper and acceptable analytical techniques have been performed. Additionally, overall data quality and laboratory performance is evaluated by applying the appropriate data quality criteria to the data to reflect conformance with the specified, accepted QA/QC standards and contractual requirements.

At the completion of the data validation, a Summary Data Validation/Usability Report will be prepared and submitted to the NYSDEC.

2.16 Performance and System Audits

A NYSDOH ELAP and CLP certified laboratory which has satisfactorily completed performance audits and performance evaluation samples shall be used.

2.17 Corrective Action

A NYSDOH ELAP and CLP certified laboratory shall meet the requirements for corrective action protocols, including sample "clean up" to attempt to eliminate/mitigate "matrix interference."

The 1995 NYSDEC ASP protocols include both mandatory and optional sample cleanup and extraction methods. Cleanup is required by the 1995 NYSDEC ASP in order to meet contract required detection limits. There are several optional cleanup and extraction methods noted in the 1995 NYSDEC ASP protocol. These include: florisil column cleanup, silica gel column cleanup, acid-base partition, steam distillation and sulfuric acid cleanup for PCB analysis.

It should be noted, that if these optional cleanup and extraction methods are requested by NYSDEC, holding time requirements should not be exceeded due to negligence of the laboratory. However, subsequent to selection of the analytical laboratory for this project, a meeting will be scheduled among representatives of the NYSDEC, the Town, D&B and the laboratory to discuss these issues and establish procedures to ensure good and timely communications among all parties.

2.18 Trip Blanks (Travel Blanks)

The primary purpose of this type of blank is to detect additional sources of contamination that might potentially influence contaminant values reported in actual samples both quantitatively and qualitatively. The following have been identified as potential sources of contamination:

- Laboratory reagent water
- Sample containers
- Cross contamination in shipment
- Ambient air or contact with analytical instrumentation during preparation and analysis at the laboratory
- Laboratory reagents used in analytical procedures

A trip blank consists of a set of 40 ml sample vials filled at the laboratory with laboratory demonstrated analyte free water. Trip blanks should be handled, transported and analyzed in the same manner as the samples acquired that day, except that the sample containers themselves are not opened in the field. Rather, they just travel with the sample cooler. Trip blanks must accompany samples at a rate of one per shipment. The temperature of the trip blanks must be maintained at 4°C while on-site and during shipment. Trip blanks must return to the laboratory with the same set of bottles they accompanied in the field.

The purpose of a trip blank is to control sample bottle preparation and blank water quality as well as sample handling. Thus, the trip blank travels to the site with the empty sample bottles and back from the site with the collected samples in an effort to simulate sample handling conditions. Contaminated trip blanks may indicate inadequate bottle cleaning or blank water of questionable quality. Trip blanks are implemented only when collecting water samples, and analyzed for volatile organic compounds only.

2.19 Field Blank (Field Rinsate Blank)/Equipment Blank

Based upon discussion with the NYSDEC, field blanks are not required for this project, since disposable bailers and sterile scoops are being utilized for sample collection. However, one equipment blank is required for the split spoon since it will be decontaminated after each use. Equipment blanks will be collected at a rate of one per day and analyzed for the same parameters as that of the samples collected with that equipment. The equipment blank will be collected by pouring laboratory supplied deionized water over/through the decontaminated equipment.

2.20 Matrix Spikes/Matrix Spike Duplicates and Spiked Blanks

Matrix spike samples and blanks are quality control procedures, consistent with 1995 NYSDEC ASP specifications, used by the laboratory as part of its internal Quality Assurance/Quality Control program. The matrix and matrix spike duplicates are aliquots of a designated sample (water or soil) which are spiked with known quantities of specified compounds. They are used to evaluate the matrix effect of the sample upon the analytical methodology as well as to determine the precision of the analytical method used. A matrix spike blank is an aliquot of analyte-free water, prepared in the laboratory, and spiked with the same solution used to spike the

MS and MSD. The MSB is subjected to the same analytical procedure as the MS/MSD and used to indicate the appropriateness of the spiking solution by calculating the spike compound recoveries. The procedure and frequency regarding the MS, MSD and MSB are defined in the 1995 NYSDEC ASP. Site specific MS and MSDs should be collected at a frequency of one per 20 samples or every 7 days (one for each sample delivery group), for each sample matrix collected (i.e., water, soil, etc.). The laboratory is required to analyze an MSB at the same frequency as the MS/MSD.

2.21 Method Blanks

A method blank is an aliquot of laboratory water or soil which is spiked with the same internal and surrogate compounds as the samples. Its purpose is to define and determine the level of laboratory background contamination. Frequency, procedure and maximum laboratory containment concentration limits are specified in the 1995 NYSDEC ASP as follows:

The laboratory shall prepare and analyze one laboratory reagent blank (method blank) for each group of samples of a similar matrix (for water or soil samples), extracted by a similar method (separatory funnel, continuous liquid extraction or sonication) and a similar concentration level (for volatile and semivolatile soil samples only) for the following, whichever is most frequent:

- Each case of field samples received, or
- Each 20 samples in a case, including matrix spikes and reanalyses, or
- Each 7 calendar day period during which field samples in a case were received (said period beginning with the receipt of the first sample in that sample delivery group) or
- Whenever samples are extracted.

Volatile analysis requires one method blank for each 12-hour time period when volatile target compounds are analyzed.

Semivolatile and pesticide method blanks shall be carried through the entire analytical process from extraction to final GC/MS or GC/EC analysis, including all protocol performance/delivery requirements.

2.22 Field Management Forms

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DAILY FIELD ACTIVITY REPORT

Report Number: ______________ Project Number: ______________ Date: ______________

Field Log Book Page Number: ______________

Project: ______________

Address: ______________

Weather: (AM) ______________ Rainfall: (AM) ______ Inches
(PM): ______________ (PM) ______ Inches

Temperature: (AM) ______ °F Wind Speed: (AM) ______ MPH Wind Direction: (AM) ______
(PM) ______ °F (PM) ______ MPH (PM) ______

Site Condition: ______________

Personnel On Site:

Name	Affiliation	Arrival Time	Departure Time

Subcontractor Work Commencement: (AM) ______________ (PM) ______________

Subcontractor Work Completion: (AM) ______________ (PM) ______________

DB-DFAR

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DATE: ____________________

DAILY FIELD ACTIVITY REPORT

General work performed today by D&B: ____________________

List specific inspection(s) performed and results (include problems and corrective actions):

List type and location of tests performed and results (include equipment used and monitoring results):

Verbal comments received from subcontractor (include construction and testing problems, and recommendations/resulting action):

Prepared by: ____________________ Reviewed by: ____________________

DB-DFAR

Dvirka and Bartilucci
Consulting Engineers
A Division of William F. Cosulich Associates, P.C.

DAILY DRILLING REPORT

Weather:

Date:

Contractor:

Project Name:

Driller:

Project Number:

Inspector:

Client:

Other Personnel:

On-Site Equipment:

Time Log

	AM						PM														Borehole		
Category	6	7	8	9	10	11	12	1	2	3	4	5	6	7	8	9	10	11	12	Total Hours			
Mob/Setup																							
Drilling Soil																							
Drilling Rock																							
Roller Bit Reaming																							
Well Installation																							
Well Development																							
Test Pit Construction																							
Land Clearing																							
Decontamination																							
Standby Time																							
Down Time																							
Health and Safety (Level B)																							

Remarks:

Consumables

Materials	Well/Borehole	Size/Type	Contract Item #	Quantity	Well/Borehole	Size/Type	Contract Item #	Quantity
Well Riser								
Well Screen								
Grout								
Bentonite								
Sand Pack								
Split Spoons								
Rock Coring								
Protective Casing								
Locking Cap								
55 Gallon Drums								

Remarks:

Drilling Record

Borehole Number	From Feet	To Feet	Dia. Inches	Method	Notes

Remarks:

Driller Signature:______________________

Inspector Signature:______________________

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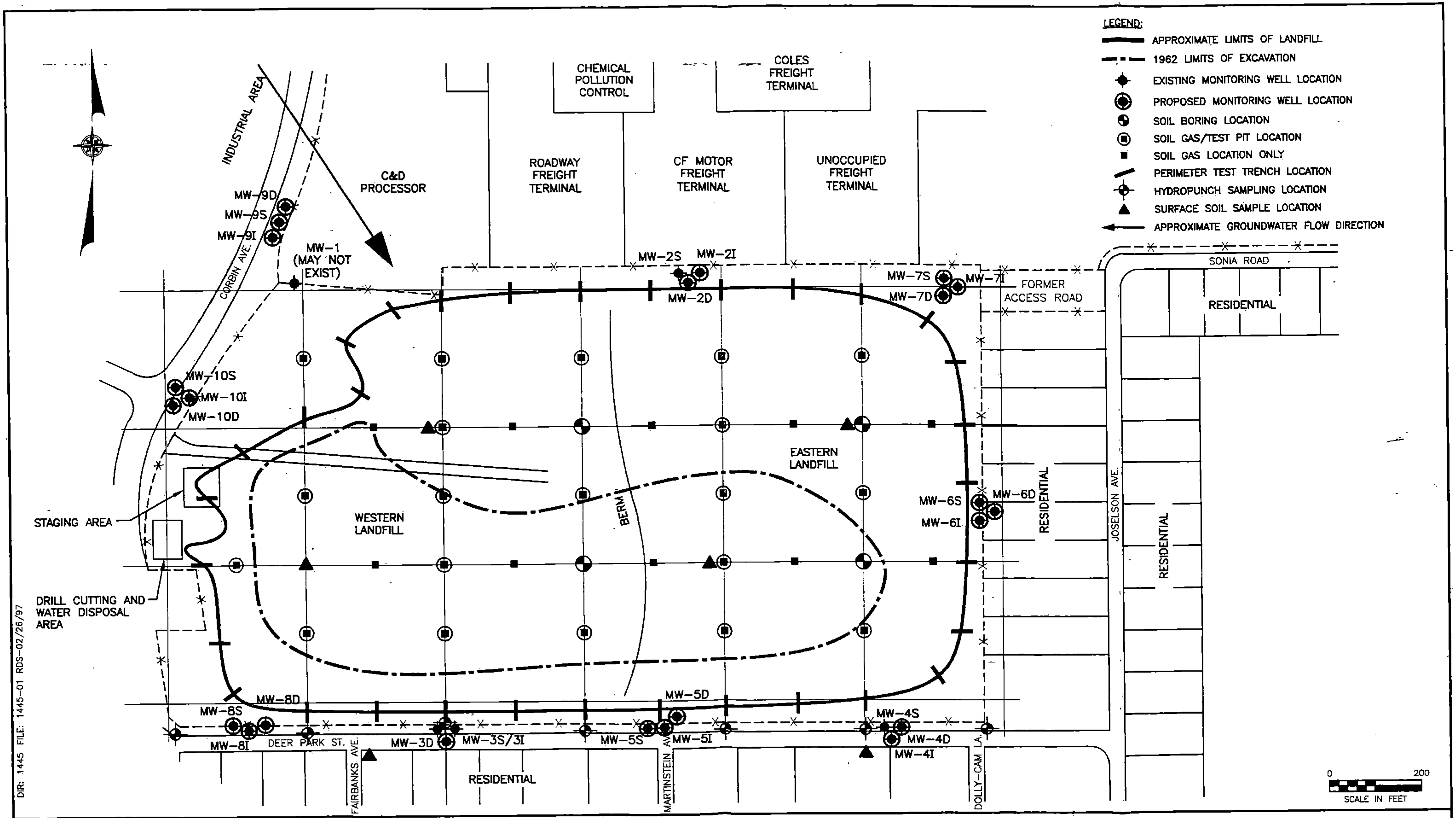

ISLIP RESOURCE RECOVERY AGENCY
SONIA ROAD LANDFILL

Dvirka and Bartilucci
Consulting Engineers
A Division of William F. Cosulich Associates, P.C.

FIELD SAMPLING PROGRAM

FIGURE 1-1

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DRILLING CONTRACTOR

Driller ____________

Inspector ____________

Rig Type ____________

Drilling Method ____________

Drive Hammer Weight ____________

DRILLING LOG

PROJECT NAME ____________

PROJECT # ____________

Location/Address ____________

BORING NUMBER ____________

Sheet ______ **of** ______

Boring Location ____________

GROUNDWATER OBSERVATIONS

Water Level			
Time			
Date			
Casing Depth			

Weather ____________

Date/Time Start ____________

Date/Time Finish ____________

Plot Plan

Sample Depth	Sample Number	SPT	PID/FID Reading	FIELD IDENTIFICATION OF MATERIAL	WELL SCHEMATIC	COMMENTS

SPT = STANDARD PENETRATION TEST

Soil Stratigraphy Summary ____________

DRILLCON.PM4

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TEST PIT LOG

TEST PIT LOCATION SKETCH MAP

N

TEST PIT NO.

PROJECT NO./NAME

LOCATION

EXCAVATOR/EQUIPMENT/OPERATOR

INSPECTOR/OFFICE

START/FINISH DATE

ELEVATION OF: GROUND SURFACE/BOTTOM OF PIT
(FT. ABOVE MSL)

CONDITION OF PIT

REMARKS:

DEPTH	SAMPLE INTERVAL	OVA SCREEN	DESCRIPTION OF MATERIALS	REMARKS
0				
1				
2				
3				
4				
5				
6				
7				
8				
9				
10				
11				
12				
13				
14				
15				

NJDEPTPL

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CONSULTING ENGINEERS

WELL CONSTRUCTION LOG

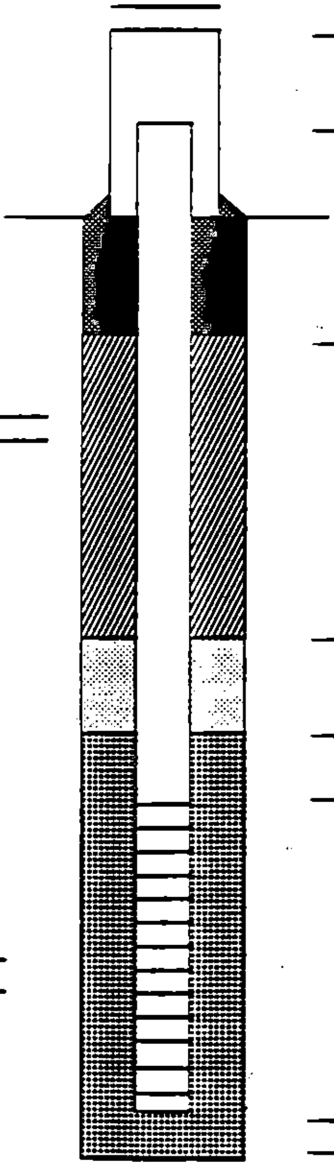
SITE ______________________________ JOB NO. _____ WELL NO. _____

TOTAL DEPTH _____ SURFACE ELEV. __________ TOP RISER ELEV. __________

WATER LEVELS (DEPTH, DATE, TIME) __________ DATE INSTALLED _______

RISER	DIA ___	MATERIAL ________	LENGTH _____	
SCREEN	DIA ___	MATERIAL ________	LENGTH _____	SLOT SIZE _______
PROT CSG	DIA ___	MATERIAL ________	LENGTH _____	

SCHEMATIC



___ Prot. Csg Stickup

___ Riser Stickup

Ground Surface

Surface Seal Type ______________
____

___ Bottom Surface Seal

Grout Type ______________
____

___ Top Seal

Seal Type ______________

___ Top Sand Pack

___ Top Screen

Sand Pack Type ______________
Size ______________

___ Bottom Screen

___ Total Depth of Boring

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WELL CONSTRUCTION LOG

SITE ______________________ JOB NO. ________ WELL NO. ________

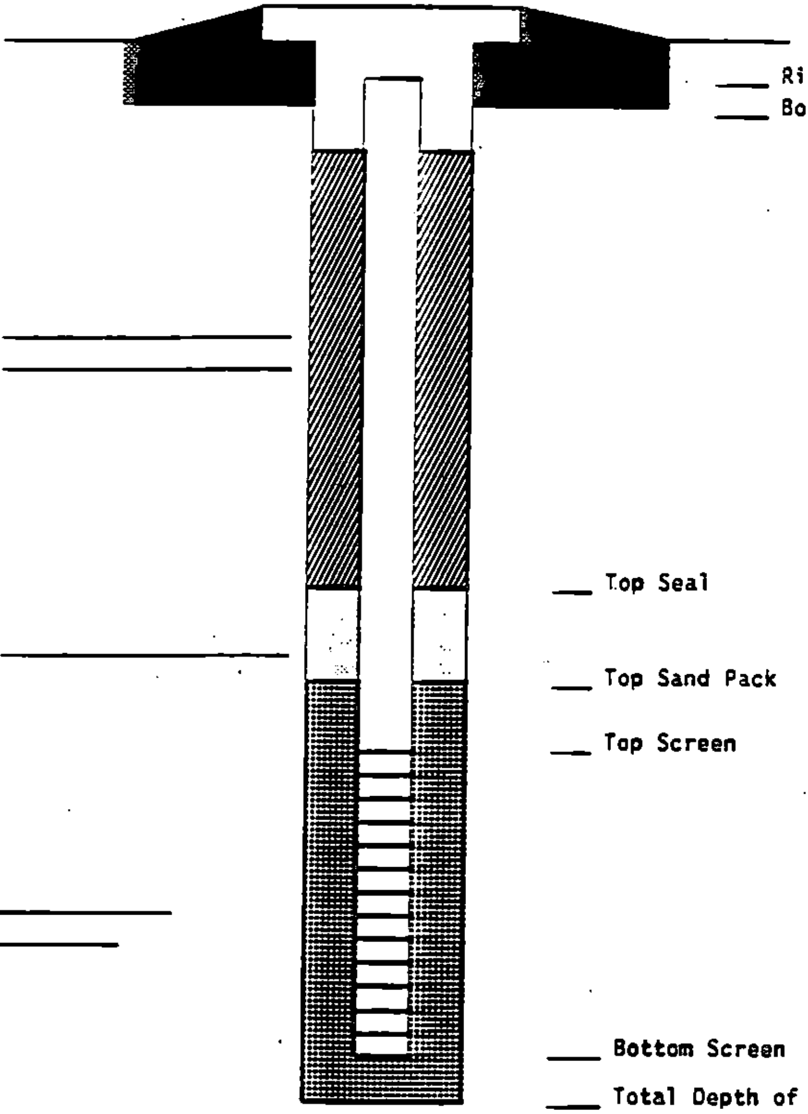
TOTAL DEPTH ________ SURFACE ELEV. ________ TOP RISER ELEV. ________

WATER LEVELS (DEPTH, DATE, TIME) ____________ DATE INSTALLED ________

RISER DIA ___ MATERIAL ________ LENGTH ____

SCREEN DIA ___ MATERIAL ________ LENGTH ____ SLOT SIZE ______

SCHEMATIC



Ground Surface

___ Riser Elevation

___ Bottom Surface Seal

Surface Seal Type
____
____

Grout Type ________________
____

___ Top Seal

Seal Type ________________

___ Top Sand Pack

___ Top Screen

Sand Pack Type ______________
Size ______________

___ Bottom Screen

___ Total Depth of Boring

**DVIRKA
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DAILY EQUIPMENT CALIBRATION LOG

Project Name: ______________________ Date: ______________________

Project Number: ______________________ Calibrated By: ______________________

Instrument Name and Model Number	Calibration Method	Time	Readings and Observations

DECL

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Receipt for Samples

Project Name: ____________________

Project Address: ____________________

Project Number: ____________________

Field Log Book Reference Number: ____________________

Sampled By: ____________________

Split With: ____________________

SAMPLE NUMBERS	DATE	TIME	COMP	GRAB	SPLIT SAMPLES	LOG BOOK PAGE NO.	TAG NUMBERS	SAMPLE LOCATION	NO. OF CONTAINERS	REMARKS

Transferred by (Signature)		Received by (Signature)		Telephone	
Date	Time	Title	Date	Time	

RFS

DVIRKA AND BARTILUCCI

LOCATION SKETCH

Project ______________________ Sample Crew ______________________

Sample(s) Location(s) ______________________

Sample(s) and/or Well Number(s) ______________________

____

Location of sample points, wells, borings, etc., with reference to three permanent reference points. Measure all distances, clearly label roads, wells and permanent features.

LS

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SAMPLE INFORMATION RECORD

SITE ______________________ SAMPLE CREW ______________________

SAMPLE LOCATION/WELLNO. ______________________

FIELD SAMPLE I.D. NUMBER ______________________ DATE ______________________

TIME ____________ WEATHER ______________________ TEMPERATURE ____________

SAMPLE TYPE:

GROUNDWATER ______________________ SEDIMENT ______________________

SURFACE WATER/STREAM ______________________ AIR ______________________

SOIL ______________________ OTHER (Describe, i.e., septage, leachate) ______________________

WELL INFORMATION (fill out for groundwater samples):

DEPTH TO WATER ______________________ MEASUREMENT METHOD ______________________

DEPTH OF WELL ______________________ MEASUREMENT METHOD ______________________

VOLUME REMOVED ______________________ REMOVAL METHOD ______________________

FIELD TEST RESULTS:

COLOR ______________________ pH ______________________ ODOR ______________________

TEMPERATURE (°F) ____________ SPECIFIC CONDUCTANCE (umhos/cm) ______________________

OTHER (OVA, Methane meter, etc.) ______________________

____

CONSTITUENTS SAMPLED:

____________ ____________ ____________ ____________

____________ ____________ ____________ ____________

REMARKS: ______________________

____

____

WELL CASING VOLUMES

GAL/FT	**1-1/4" = 0.077**	**2" = 0.16**	**3" = 0.37**	**4" = 0.65**
	1-1/2" = 0.10	**2-1/2" = 0.24**	**3-1/2" = 0.50**	**6" = 1.46**

SIR

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FIELD CHANGE FORM

Project Name: ______________________________

Project Number: ____________________ Field Change Number: ____________

Location: ______________________________ Date: ____________

Field Activity Description: ______________________________

____

____

____

Reason for Change: ______________________________

____

____

____

Recommended Disposition: ______________________________

____

____

____

______________________________ ____________

Field Operations Officer (D&B Consulting Engineers) (Signature) Date

Disposition: ______________________________

____

____

____

______________________________ ____________

On-site Supervisor (NYSDEC) (Signature) Date

Distribution: Project Manager (D&B)
Project Manager (NYSDEC)
Field Operations Officer
On-site Supervisor (NYSDEC)

Others as Required: ____________
____
____
____

DB-FCF

2.23 NYSDEC Sample Identification, Preparation and Analysis Summary Forms

To be included with all lab data and with each workplan

NEW YORK STATE DEPARTMENT OF ENVIRONMENTAL CONSERVATION

SAMPLE IDENTIFICATION AND ANALYTICAL REQUIREMENT SUMMARY

Customer Sample Code	Laboratory Sample Code	Analytical Requirements*					
		*VOA GC/MS	*BNA GC/MS	*VOA GC	*PEST PCB	*METALS	*OTHER

*Check Appropriate Boxes

* CLP, Non-CLP (Please indicate year of protocol)
* HSL, Priority Pollutant

NEW YORK STATE DEPARTMENT OF ENVIRONMENTAL CONSERVATION

SAMPLE PREPARATION AND ANALYSIS SUMMARY
B/N-A
ANALYSES

Laboratory Sample ID	Matrix	Date Collected	Date Rec'd At Lab	Date Extracted	Date Analyzed

NEW YORK STATE DEPARTMENT OF ENVIRONMENTAL CONSERVATION

SAMPLE PREPARATION AND ANALYSIS SUMMARY
PESTICIDE/PCB
ANALYSES

Laboratory Sample ID	Matrix	Date Collected	Date Rec'd At Lab	Date Extracted	Date Analyzed

NEW YORK STATE DEPARTMENT OF ENVIRONMENTAL CONSERVATION

SAMPLE PREPARATION AND ANALYSIS SUMMARY
VOA
ANALYSES

Laboratory Sample ID	Matrix	Date Collected	Date Rec'd At Lab	Low Level Med. Level	Date Analyzed

NEW YORK STATE DEPARTMENT OF ENVIRONMENTAL CONSERVATION
SAMPLE PREPARATION AND ANALYSIS FORM

B/N-A ORGANIC ANALYSES

Sample ID	Matrix	Analytical Protocol	Extraction Method	Auxilary Clean Up	Dil/Conc Factor

NEW YORK STATE DEPARTMENT OF ENVIRONMENTAL CONSERVATION

SAMPLE PREPARATION AND ANALYSIS SUMMARY

INORGANIC ANALYSES

Sample ID	Matrix	Metals Requested	Date Rec'd	Date Analyzed

2.24 Data Validation Reporting Forms

TO BE SUPPLIED BY DATA VALIDATOR

2.25 Data Quality Requirements and Assessment Summaries

Superfund Target Compound List (TCL) and Contract Required Quantitation Limits (CRQL)

			Quantitation Limits*			
	Volatiles	CAS Number	Water µg/L	Low Soil µg/Kg	Med Soil µg/Kg	On Column (ng)
1.	Chloromethane	74-87-3	10	10	1200	(50)
2.	Bromomethane	74-83-9	10	10	1200	(50)
3.	Vinyl chloride	75-01-4	10	10	1200	(50)
4.	Chloroethane	75-00-3	10	10	1200	(50)
5.	Methylene chloride	75-09-2	10	10	1200	(50)
6.	Acetone	67-64-1	10	10	1200	(50)
7.	Carbon Disulfide	75-15-0	10	10	1200	(50)
8.	1,1-Dichloroethylene	75-35-4	10	10	1200	(50)
9.	1,1-Dichloroethane	75-35-3	10	10	1200	(50)
10.	1,2-Dichloroethylene(total)	540-59-0	10	10	1200	(50)
11.	Chloroform	67-66-3	10	10	1200	(50)
12.	1,2-Dichloroethane	107-06-2	10	10	1200	(50)
13.	2-Butanone	78-93-3	10	10	1200	(50)
14.	1,1,1-Trichloroethane	71-55-6	10	10	1200	(50)
15.	Carbon tetrachloride	56-23-5	10	10	1200	(50)
16.	Bromodichloromethane	75-27-4	10	10	1200	(50)
17.	1,2-Dichloropropane	78-87-5	10	10	1200	(50)
18.	cis-1,3-Dichloropropene	10061-01-5	10	10	1200	(50)
19.	Trichloroethene	79-01-6	10	10	1200	(50)
20.	Dibromochloromethane	124-48-1	10	10	1200	(50)
21.	1,1,2-Trichloroethane	79-00-5	10	10	1200	(50)
22.	Benzene	71-43-2	10	10	1200	(50)
23.	trans-1,3-Dichloropropene	10061-02-6	10	10	1200	(50)
24.	Bromoform	75-25-2	10	10	1200	(50)
25.	4-Methyl-2-pentanone	108-10-1	10	10	1200	(50)
26.	2-Hexanone	591-78-6	10	10	1200	(50)
27.	Tetrachloroethene	127-18-4	10	10	1200	(50)
28.	Toluene	108-88-3	10	10	1200	(50)
29.	1,1,2,2-Tetrachloroethane	79-34-5	10	10	1200	(50)
30.	Chlorobenzene	108-90-7	10	10	1200	(50)
31.	Ethyl Benzene	100-41-4	10	10	1200	(50)
32.	Styrene	100-42-5	10	10	1200	(50)
33.	Total Xylenes	1330-20-7	10	10	1200	(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 protocol, will be higher.

Note that the CRQL values listed on the preceding page may not be those specified in previous Analytical Services Protocols. These values are set at concentrations in the sample equivalent to the concentration of the lowest calibration standard specified in Exhibit D, Part II. Lower quantitation limits may be achievable for water samples by employing the methods in Exhibit D, Part X for Low Concentration Water for Organic Analyses.

VOLATILES

Water Samples

A 5 mL volume of water is purged with an inert gas at ambient temperature. The volatiles are trapped on solid sorbents, and desorbed directly onto the GC/MS. For a sample with compound X at the CRQL of 10 µg/L:

(10 µg/L) (5 mL) (10^{-3} L/mL) = 50 x 10^{-3} ug = 50 ng on the GC column

Low Level Soil/Sediment Samples

A 5 g aliquot of the soil/sediment sample is added to a volume of water in a purge tube, heated, and purged with an inert gas. The volatiles are trapped, and later desorbed directly onto the GC/MS. For a sample with compound X at the CRQL of 10 µg/Kg:

(10 µg/Kg) (5 g) (10^{-3} Kg/g) = 50 x 10^{-3} µg = 50 ng on the GC column

Medium Level Soil/Sediment Samples

A 4 g aliquot of soil/sediment is extracted with 10 mL of methanol, and filtered through glass wool. Only 1 mL of the methanol extract is taken for screening and analysis. Based on the results of a GC/FID screen, an aliquot of the methanol extract is added to 5 mL of reagent water and purged at ambient temperature. The largest aliquot of extract considered in Exhibit D, Part III is 100 uL. For a sample with compound X at the CRQL of 1200 µg/Kg:

(1200 µg/Kg) (4 g) (10^{-3} Kg/g) = 4800 x 10^{-3} µg = 4800 ng

This material is contained in the 10 mL methanol extract:

(4800 ng) / 10 mL = 480 ng/mL

Of which, 100 µL are purged from the reagent water.

(480 ng/mL) (100 µL) (10^{-3} mL/µL) = 480 x 10^{-1} ng = 50 ng on the GC column

Note that for both low and medium soil/sediment samples, while it may affect the purging efficiency, the volume of reagent water used in the purging process does not affect the calculations.

Superfund Target Compound List (TCL) and Contract Required Quantitation Limits (CRQL)*

	Semivolatiles	CAS Number	Quantitation Limits* Water µg/L	 Low Soil µg/Kg	 Med Soil µg/Kg	 On Column (ng)
34.	Phenol	108-95-2	10	330	10,000	(20)
35.	bis(2-Chloroethyl) ether	111-44-4	10	330	10,000	(20)
36.	2-Chlorophenol	95-57-8	10	330	10,000	(20)
37.	1,3-Dichlorobenzene	541-73-1	10	330	10,000	(20)
38.	1,4-Dichlorobenzene	106-46-7	10	330	10,000	(20)
39.	1,2-Dichlorobenzene	95-50-1	10	330	10,000	(20)
40.	2-Methylphenol	95-48-7	10	330	10,000	(20)
41.	2,2'-oxybis(1-Chloropropane) #	108-60-1	10	330	10,000	(20)
42.	4-Methylphenol	106-44-5	10	330	10,000	(20)
43.	N-Nitroso-di-n-propylamine	621-64-7	10	330	10,000	(20)
44.	Hexachloroethane	67-72-1	10	330	10,000	(20)
45.	Nitrobenzene	98-95-3	10	330	10,000	(20)
46.	Isophorone	78-59-1	10	330	10,000	(20)
47.	2-Nitrophenol	88-75-5	10	330	10,000	(20)
48.	2,4-Dimethylphenol	105-67-9	10	330	10,000	(20)
49.	bis(2-Chloroethoxy) methane	111-91-1	10	330	10,000	(20)
50.	2,4-Dichlorophenol	120-83-2	10	330	10,000	(20)
51.	1,2,4-Trichlorobenzene	120-82-1	10	330	10,000	(20)
52.	Naphthalene	91-20-3	10	330	10,000	(20)
53.	4-Chloroaniline	106-47-8	10	330	10,000	(20)
54.	Hexachlorobutadiene	87-68-3	10	330	10,000	(20)
55.	4-Chloro-3-methylphenol	59-50-7	10	330	10,000	(20)
56.	2-Methylnaphthalene	91-57-6	10	330	10,000	(20)
57.	Hexachlorocyclopentadiene	77-47-4	10	330	10,000	(20)
58.	2,4,6-Trichlorophenol	88-06-2	10	330	10,000	(20)
59.	2,4,5-Trichlorophenol	95-95-4	25	800	25,000	(50)
60.	2-Chloronaphthalene	91-58-7	10	330	10,000	(20)
61.	2-Nitroaniline	88-74-4	25	800	25,000	(50)
62.	Dimethyl phthalate	131-11-3	10	330	10,000	(20)
63.	Acenaphthylene	208-96-8	10	330	10,000	(20)
64.	2,6-Dinitrotoluene	606-20-2	10	330	10,000	(20)
65.	3-Nitroaniline	99-09-2	25	800	25,000	(50)
66.	Acenaphthene	83-32-9	10	330	10,000	(20)

\# Previously known by the name bis(2-Chloroisopropyl) ether

Superfund Target Compound List (TCL) and Contract Required Quantitation Limits (CRQL)

			Quantitation Limits*			
	Semivolatiles	CAS Number	Water µg/L	Low Soil µg/Kg	Med Soil µg/Kg	On Column (ng)
67.	2,4-Dinitrophenol	51-28-5	25	800	25,000	(50)
68.	4-Nitrophenol	100-02-7	25	800	25,000	(50)
69.	Dibenzofuran	132-64-9	10	330	10,000	(20)
70.	2,4-Dinitrotoluene	121-14-2	10	330	10,000	(20)
71.	Diethylphthalate	84-66-2	10	330	10,000	(20)
72.	4-Chlorophenyl phenyl ether	7005-72-3	10	330	10,000	(20)
73.	Fluorene	86-73-7	10	330	10,000	(20)
74.	4-Nitroaniline	100-01-6	25	800	25,000	(50)
75.	4,6-Dinitro-2-methylphenol	534-52-1	25	800	25,000	(50)
76.	N-nitrosodiphenylamine	86-30-6	10	330	10,000	(20)
77.	4-Bromophenyl phenyl ether	101-55-3	10	330	10,000	(20)
78.	Hexachlorobenzene	118-74-1	10	330	10,000	(20)
79.	Pentachlorophenol	87-86-5	25	800	25,000	(50)
80.	Phenanthrene	85-01-8	10	330	10,000	(20)
81.	Anthracene	120-12-7	10	330	10,000	(20)
82	Carbazole	86-74-8	10	330	10,000	(20)
83.	Di-n-butyl phthalate	84-74-2	10	330	10,000	(20)
84.	Fluoranthene	206-44-0	10	330	10,000	(20)
85.	Pyrene	129-00-0	10	330	10,000	(20)
86.	Butyl benzyl phthalate	85-68-7	10	330	10,000	(20)
87.	3,3'-Dichlorobenzidine	91-94-1	10	330	10,000	(20)
88.	Benz(a)anthracene	56-55-3	10	330	10,000	(20)
89.	Chrysene	218-01-9	10	330	10,000	(20)
90.	bis(2-Ethylhexyl)phthalate	117-81-7	10	330	10,000	(20)
91.	Di-n-octyl phthalate	117-84-0	10	330	10,000	(20)
92.	Benzo(b)fluoranthene	205-99-2	10	330	10,000	(20)
93.	Benzo(k)fluoranthene	207-08-9	10	330	10,000	(20)
94.	Benzo(a)pyrene	50-32-8	10	330	10,000	(20)
95.	Indeno(1,2,3-cd)pyrene	193-39-5	10	330	10,000	(20)
96.	Dibenz(a,h)anthracene	53-70-3	10	330	10,000	(20)
97.	Benzo(g,h,i)perylene	191-24-2	10	330	10,000	(20)

* 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 Protocol, will be higher.

Water Samples

A 1 L volume of water is extracted in a continuous liquid-liquid extractor with methylene chloride at a pH of approximately 2. This extract is reduced in volume to 1.0 mL, and a 2 µL volume is injected onto the GC/MS for analysis. For a sample with compound X at the CRQL of 10 µg/L:

(10 µg/L) (1 L) = 10 µg in the original extract

When the extract is concentrated, this .material is contained in the 1 mL concentrated extract, of which 2 µL are injected into the instrument:

(10 µg/mL) (2 µL) (10^{-3} mL/µL) = 20 x 10^{-3} µg = 20 ng on the GC column

Low Soil Samples

A 30 g soil sample is extracted three times with methylene chloride/acetone at ambient pH, by sonication or Soxhlet. The extract is reduced in volume to 1.0 mL, and a 2 µL volume is injected onto the GC/MS for analysis. For a sample with compound X at the CRQL of 330 µg/Kg:

(330 µg/Kg) (30 g) (10^{-3} Kg/g) = 9900 x 10^{-3} µg = 9.9 µg

When the sample extract is to be subjected to Gel Permeation Chromatography (required) to remove high molecular weight interferences, the volume of the extract is initially reduced to 10 mL. This 10 mL is put through the GPC column, and only 5 mL are collected off the GPC. That 5 mL volume is reduced to 0.5 mL prior to analysis. Therefore:

(9.9 µg/10 mL) (5 mL) = 4.95 µg

This material is contained in the 0.5 mL extract, of which 2 µL are injected into the instrument:

(4.95 µg/0.5 mL) (2 µL) (10^{-3} mL/µL) = (1.98 x 10^{-2} µg) 20 ng on the GC column

Medium Soil Samples

A 1 g soil sample is extracted once with 10 mL of methylene chloride/acetone, which is filtered through glass wool to remove particles of soil. The filtered extract is then subjected to GPC clean up, and only 5 mL of extract are collected after GPC. This extract is reduced in volume to 0.5 mL, of which 2 µL are injected onto the GC/MS. For a sample with compound X at the CRQL of 10,000 µg/Kg:

(10,000 µg/Kg) (1 g) (10^{-3} Kg/g) = 10 µg

(continued)

Semivolatiles, Medium Soil, continued -

This material is contained in the 10 mL extract, of which only 5 mL are collected after GPC:

(10 µg) (5 mL/10 mL) = 5 ug

The volume of this extract is reduced to 0.5 mL, of which 2 µL are injected into the instrument:

(5 µg/0.5 mL) (2 µL) (10^{-3} mL/µL) = 20 x 10^{-3} ug = 20 ng on the GC column

Eight semivolatile compounds are calibrated using only a four point initial calibration, with the lowest standard at 50 ng. Therefore, the CRQL values for these eight compounds are 2.5 times higher for all matrices and levels.

Superfund Target Compound List (TCL) and Contract Required Quantitation Limits (CRQL)*

			Quantitation Limits*		
	Pesticides/Aroclors	CAS Number	Water µg/L	Soil µg/Kg	On Column (ng)
98.	alpha-BHC	319-84-6	0.05	1.7	5
99.	beta-BHC	319-85-7	0.05	1.7	5
100.	delta-BHC	319-86-8	0.05	1.7	5
101.	gamma-BHC (Lindane)	58-89-9	0.05	1.7	5
102.	Heptachlor	76-44-8	0.05	1.7	5
103.	Aldrin	309-00-2	0.05	1.7	5
104.	Heptachlor epoxide	1024-57-3	0.05	1.7	5
105.	Endosulfan I	959-98-8	0.05	1.7	5
106.	Dieldrin	60-57-1	0.10	3.3	10
107.	4,4'-DDE	72-55-9	0.10	3.3	10
108.	Endrin	72-20-8	0.10	3.3	10
109.	Endosulfan II	33213-65-9	0.10	3.3	10
110.	4,4'-DDD	72-54-8	0.10	3.3	10
111.	Endosulfan sulfate	1031-07-8	0.10	3.3	10
112.	4,4'-DDT	50-29-3	0.10	3.3	10
113.	Methoxychlor	72-43-5	0.50	17.0	50
114.	Endrin ketone	53494-70-5	0.10	3.3	10
115.	Endrin aldehyde	7421-36-3	0.10	3.3	10
116.	alpha-Chlordane	5103-71-9	0.05	1.7	5
117.	gamma-Chlordane	5103-74-2	0.05	1.7	5
118.	Toxaphene	8001-35-2	5.0	170.0	500
119.	AROCLOR-1016	12674-11-2	1.0	33.0	100
120.	AROCLOR-1221	11104-28-2	1.0	67.0	200
121.	AROCLOR-1232	11141-16-5	1.0	33.0	100
122.	AROCLOR-1242	53469-21-9	1.0	33.0	100
123.	AROCLOR-1248	12672-29-6	1.0	33.0	100
124.	AROCLOR-1254	11097-69-1	1.0	33.0	100
125.	AROCLOR-1260	11096-82-5	1.0	33.0	100

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

Water Samples

A 1 L volume of water is extracted three times with methylene chloride or by a continuous liquid-liquid extractor. This extract is reduced in volume to approximately 3 - 5 mL, and diluted up to 10.0 mL with clean solvent. When Gel Permeation Chromatography is performed, only 5 of the 10 mL of extract are collected after GPC.

Regardless of whether GPC is performed, either 1.0 or 2.0 mL of the 10.0 mL of the original extracts are taken through the remaining clean up steps (Florisil and sulfur removal). The volume taken through Florisil cleanup and the final volume of the extract after the clean up steps depends on the requirements of the autosampler. If the autosampler can handle 1.0 mL final extract volumes, this is the volume taken through Florisil and the final volume. If the autosampler cannot reliably handle 1.0 mL volumes, the volume is 2.0 mL. When using an autosampler, the injection volume may be 1.0 or 2.0 μL. Manual injections must use a 2.0 μL injection volume.

For a sample with compound X at the CRQL of 0.05 μg/L and an autosampler requiring a 1.0 mL volume:

(0.05 μg/L) (1 L) = 0.05 μg in the original extract

This material is contained in the 10.0 mL of extract:

(0.05 μg)/ (10.0 mL) = 0.005 μg/mL

Of which, only 1.0 mL is carried through the remaining clean up steps. For a final extract volume of 1.0 mL and a 1 μL injection volume:

(0.005 μg/L) (1 μL) (10^{-3} mL/μL) = 5 x 10^{-6} μg = 5 pg on the GC column

Soil Samples

There is no differentiation between the preparation of low and medium soil samples in this method for the analysis of pesticides/Aroclors. A 30 g soil sample is extracted three times with methylene chloride/acetone by sonication or Soxhlet extraction. The extract is reduced in volume to 10.0 mL and subjected to Gel Permeation Chromatography. After GPC, only 5.0 mL of extract are collected. However, as with the water sample described above, either 1.0 or 2.0 mL of that extract are subjected to the other clean up steps, so no loss of sensitivity results from the use of GPC. From this point on, the soil sample extract is handled in the same fashion as the extract of a water sample. For a sample with compound X at the CRQL of 1.7 μg/Kg:

(1.7 μg/Kg) (30 g) (10^{-3} Kg/g) = 51 x 10^{-3} μg = 51 ng in the original extract

This material is contained in the 10.0 mL of extract:

(51 ng)/10 mL = 5.1 ng/mL

(continued)

of which, only 1.0 or 2.0 mL are carried through the remaining cleanup steps For a final extract volume of 1.0 mL and a 1 µL injection volume:

(5.1 ng/mL)(1 µL)(10^{-3} mL/µL) = 5.1 x 10^{-3} ng = 5 pg on the GC column.

For either water or soil samples, if the autosampler used requires a 2.0 mL final volume, the concentration in the 10.0 mL of extract above remains the same.

Using a 2 µL injection volume, twice the total number of picograms are injected onto the GC column. However, because the injection volume must be the same for samples and standards, twice as much material is injected onto the column during calibration, and thus the amount of compound X injected from the sample extract is equivalent to the amount of compound X injected from the calibration standard, regardless of injection volume.

If a single injection is used for two GC columns attached to a single injection part, it may be necessary to use an injection volume greater than 2 µL.

SECTION II

SUPERFUND-CLP INORGANICS

Superfund Target Compound List (TCL) and Contract Required Quantitation Limit

Parameter	Contract Required Quantitation Level (μg/L)
1. Aluminum	200
2. Antimony	60
3. Arsenic	10
4. Barium	200
5. Beryllium	5
6. Cadmium	5
7. Calcium	5000
8. Chromium	10
9. Cobalt	50
10. Copper	25
11. Iron	100
12. Lead	3
13. Magnesium	5000
14. Manganese	15
15. Mercury	0.2
16. Nickel	40
17. Potassium	5000
18. Selenium	5
19. Silver	10
20. Sodium	5000
21. Thallium	10
22. Vanadium	50
23. Zinc	20
24. Cyanide	10

1: Any analytical method specified in Exhibit D, CLP-Inorganics may be utilized as long as the documented instrument or method detection limits meet the Contract Required Quantitation Level (CRQL) requirements. Higher quantitation levels may only be used in the following circumstance:

If the sample concentration exceeds five times the quantitation limit of the instrument or method in use, the value may be reported even though the instrument or method detection limit may not equal the Contract Required Quantitation Limit. This is illustrated in the example below:

For lead:
Method in use = ICP
Instrument Detection Limit (IDL) = 40
Sample concentration = 220
Contract Required Quantitation Level (CRQL) = 3

The value of 220 may be reported even though instrument detection limit is greater than Contract Required Quantitation Limit. The instrument or method detection limit must be documented as described in Exhibit E.

2: These CRQLs are the instrument detection limits obtained in pure water that must be met using the procedure in Exhibit E. The quantitation limits for samples may be considerably higher depending on the sample matrix.

Regulatory Promulgated Parameters

In addition to the preceding lists, the Laboratory may be asked to analyze for any or all of the conventional water quality parameters as listed in 40CFR Part 136 or for the hazardous waste parameters listed in 40CFR Part 260 through 270.

Quantitation limits to be achieved for these analyses are specified.

2.26 Field Audit Form

**DVIRKA
AND
BARTILUCCI**

FIELD AUDIT FORM

Site: ____________________ Date: ____________________

Persons On-site: ____________________ QA/QC Officer Conducting Audit: ____________________

Project: ____________________

1. Is safety equipment in use (hardhats, respirators, gloves etc.): YES NO

2. Is a decontamination station, equipment and supplies on site and in working order:

Methanol	YES	NO
Alconox	YES	NO
D.I. Water	YES	NO
Scrub Brushes	YES	NO
Steam Cleaner	YES	NO

Comments: ____________________

3. Is the site/investigation areas secured (fence, markers, etc.): YES NO

Comments: ____________________

4. Is contaminated material properly stored and in a secure area: YES NO

Comments: ____________________

DVIRKA
AND
BARTILUCCI

FIELD AUDIT FORM (continued)

5. Are forms filled out properly:

Field Log Book	YES	NO
Chain of Custody	YES	NO
Equipment Calibration Log	YES	NO
Daily Field Activity Report	YES	NO
Location Sketch	YES	NO
Sample Information Record	YES	NO
Equipment Usage Form	YES	NO

Comments: ______________________________

____

____

6. Is the proper sampling and field measurement equipment, including calibration supplies on site: YES NO

Comments: ______________________________

____

____

7. Are there adequate sample containers, including deionized water for QA/QC:

Field Blanks	YES	NO
Trip Blanks	YES	NO

Comments: ______________________________

____

____

8. Is the equipment decontaminated properly:

Sampling equipment	YES	NO
Construction equipment	YES	NO

Comments: ______________________________

____

____

FIELD AUDIT FORM (continued)

9. Is field measurement equipment calibrated:

Daily	YES	NO
Properly	YES	NO

Comments: ____________________

10. Are samples collected and labeled properly: YES NO

Comments: ____________________

11. Are samples stored at 4°C: YES NO

Comments: ____________________

12. Are coolers properly sealed and packed for shipment including Chain of Custody taped to underside of lid: YES NO

Comments: ____________________

13. Is a copy of the Field Investigation Work Plan available on site: YES NO

Comments: ____________________

FIELD AUDIT FORM (continued)

14. Is a copy of the QA/QC Plan available on site: YES NO

Comments:

15. Are investigation personnel familiar with the Work Plan and QA/QC Plan: YES NO

Comments:

16. Are quality control samples taken:

Trip Blanks YES NO
Field Blanks YES NO

Comments:

17. Are samples shipped in a timely and appropriate manner: YES NO

Comments:

18. Has the laboratory been contacted regarding planned shipment of samples: YES NO

Comments:

DVIRKA
AND
BARTILUCCI

FIELD AUDIT FORM (continued)

General Comments: ____________________

____

____

____

should be entitled; Information on Other Sites in the Area ok

SECTION 3.1
page 3-2 Under RI/FS Objectives, needs a lay-person's equivalent for receptor (can still use both terms, put one in parenthesis). ok

SECTION 3.2
page 3-2 Task 1 -Use Planning for or Planning Meetings since otherwise, people may anticipate these as public meetings. ok

SECTION 4.0
page 4-1 Instead of "local media", use local media and Newsday's Government Watch, here and elsewhere in the text (such as 4-3, newspapers...). ok

SECTION 4.1
Should note that the addresses/hours of the information repositories are provided in Section 7.0. ok

SECTION 4.2
Instead of groups (bullet 5), use groups, including civic and environmental, here and elsewhere in the text. ok

SECTION 6.0
The draft has too many contacts. This would be confusing for the public and cumbersome for the contacts. Two Town/Consultant contacts are certainly sufficient; Dr. William Graner and Thomas Maher, P.E.. Note that it is Joshua Epstein, Ph.D. ok

SECTION 7.0
Include the hours of operation of the information repositories. ok

APPENDIX A

As a first section, the names and addresses of the relevant Federal, State, and County Legislators should be included. ok

IV. should read Interested Agencies
and also include; South Farmingdale Water District, Langdary Road, Farmingdale, New York 11737, Att.: John H Bates, Commissioner ok

After Local Media (again, note 4.0) V, VI should read Public Interest Groups and Other Organizations ok

In addition to those already in the draft, the following need to be included (listing should be in alphabetical order);

Adelante of Suffolk County, PO Box 385, 10 3rd Street, Brentwood, New York 11717

Bayshore Community Forum, 393 Brook Avenue, Bayshore, New York 11706, Attn.: Mr. Powell

Bay-Brent Chapter, Long Island Progressive Coalition, 21 Frederick Avenue, Bay Shore, New York 11717, Attn.: Lou Olivera

St. Anne's Roman Catholic Church, 88 Second Avenue, Brentwood, New York 11717, Attn.: Rev. Michael O'Keefe

St. Patrick's School Board, c/o Virginia Lombardi, 27 West Lakeland Street, Brentwood, New York 11706

Brentwood Family Center, 1734 Brentwood Road, Brentwood, New York 11717

East Brentwood Civic Association, PO Box 471 Brentwood, New York 11717, Attn.: Mary Gleason; also Joseph Fritz, Esq., 135 West Main Street, East Islip, New York 11730

Brentwood/Bayshore Breast Cancer Coalitoon, 18 Stockton Street, Brentwood, New York 11717, Attn.: Elsa Ford

Linkage, 2000 Brentwood Avenue, Brentwood, New York 11717

Temple Beth Ann Sanctuary, 28 6th Avenue, Brentwood, New York 11717, Attn.: Ruth Rosenthal

Long Island Citizens Advisory Committee on Hazardous Waste, Co-Chairs; Jeff Fullmer, c/o Citizens Campaign for the Environment, 550 Smithtown Bypass, Suite 205, Hauppauge, New York 11788 and Rosemary Konatich, c/o NYS Legislative Commission, Long Island Water Needs, 11 Middle Neck Road, Suite 200, Great Neck, New York 11021

Americana Laundry, 1572 Fifth Avenue, Bay Shore, New York 11706

Hubbard Wilson Sand and Gravel, 1612 North 5th Avenue, Bay Shore, New York 11706

H.U.D., 1506 East Third Avenue, Bay Shore, New York 11706, Attn.: Secretary

K.P.K. Realty Corp., 1460 North Firfth Avenue, Bay Shore, New York 11706

La Tropical Market, 1617 Fifth Avenue, Bay Shore, New york 11706, Attn.: Rosalie Robolas.

Freshman Center, Leahy Avenue, Brentwood, New York 11717, Attn.: Peter Perlow, Principal

Twin Pines Elementary School, 2 Mur Place, Brentwood, New York

11717, Attn.: Alex Werner, Principal

Brentwood PTSA Council, Council President, Lauri Koerner, 179 Spur Drive South, Bay Shore, New York 11706 and Dorene Zurlo, Environmental Chair, at 99 Washington Avenue, Brentwood, New York 11717

Pine Park Kindergarten PTA, Rebecca Alvarado, President, at 980 Suffolk Avenue, Brentwood, New York 11717

Hemlock Park Elementary School PTA, Helen Coletti, President, at 1584 Brightwaters, Bay Shore, New York 11706

Twin Pines Elementary PTA, Dorathy Garverick, President, at 339 Hancock Street, Brentwood, New York 11717

Southwest Elementary PTA, Joann Gearino, President, at 1692 Pine Acres Blvd., Bay Shore, New York 11706

Islip School District (ascertain and include appropriate officials)

VII (VI in current draft) should read Residents in the Area of the Site and re-state that all residents and businesses within a 1/4 mile of the site will be included. The current sentence in the draft should be deleted.

Please inform me as to the disposition of these comments.

c.c. Robert Becherer
     Nina Knapp

Appendix B

APPENDIX C

GROUNDWATER MONITORING WELL DECOMMISSIONING PROCEDURES NYSDEC DIVISION OF HAZARDOUS WASTE REMEDIATION DATED MAY 1995

GROUNDWATER MONITORING WELL DECOMMISSIONING PROCEDURES

May 1995

EARTH DAY 25
1995
ENVIRONMENTAL PARTNERS

Prepared for:

New York State Department
of Environmental Conservation

Division of Hazardous Waste Remediation

Prepared by:

Malcolm Pirnie, Inc.

DISCLAIMER

May 8, 1995

RE: New York State Department of Environmental Conservation
Division of Hazardous Waste Remediation
Monitoring Well Decommissioning Procedures

Per your request, the enclosed referenced document is being made available to you for informational purposes. These procedures may be used as a guideline when decommissioning a monitoring well. Please note that this document does not address some site specific special situations that may be encountered while dealing with telescoped wells. These procedures have not been adopted by the Department of Environmental Conservation. Compliance with the procedures set out on this document does not relieve any party of the obligation to successfully and satisfactorily decommission a well.

If you have any questions, please contact A. K. Gupta, of my staff, at (518) 457-0927.

Sincerely,

Gerald J. Rider, Jr., P.E.
Chief, Operation, Maintenance and Support Section
Bureau of Hazardous Site Control
Division of Hazardous Waste Remediation
New York State Department of Environmental Conservation

Enclosure

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NYS SUPERFUND STANDBY CONTRACT
NPL SITE MONITORING WELL DECOMMISSIONING

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LIST OF FIGURES/LOGS & PLATES

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reviewed, and a pre-construction inspection of the monitoring well will be conducted to assist in determining the best-suited decommissioning method.

For maximum protection of human health and the environment, any material brought to the surface during the decommissioning process will be treated as a hazardous waste unless sample data indicates otherwise. The selection of disposal methods for these materials will depend on information reported in site investigation reports and analytical characterization of the retrieved materials for hazardous characteristics (see Sections 4.1.3 through 4.1.4). An appropriate procedure will be followed for the physical and hydrologic setting of the well that best protects the environment.

The following sections describe the procedures that will be implemented to properly decommission a well, including the procedure for selecting which decommissioning method will be used. There are eleven elements to be addressed in decommissioning a monitoring well at a hazardous waste site:

1) Reviewing Site Data
2) Selecting the Well Decommissioning Method
3) Preparing a Site-Specific Health and Safety Plan
4) Preparing a Materials Handling and Disposal Plan
5) Establishing Decontamination Procedures
6) Locating and Setting-Up on the Well
7) Removing the Protective Casing
8) Decommissioning of Screen and Riser
9) Selecting, Mixing, and Placing Grout
10) Backfilling and Site Restoration
11) Quality Assurance/Quality Control (QA/QC) Procedures

The proper well decommissioning methods and selection process are presented on the flow chart presented as Plate 1. For each decommissioning method, the specific procedures are determined by (1) geology, (2) contaminants, and (3) well design. For example, decommissioning a well that penetrates a confining layer may require a different approach than decommissioning an unconfined water table well.

1.0 REVIEWING SITE DATA

The first step in selecting the well decommissioning process consists of reviewing all pertinent site information; boring and well logs, field inspection sheets, and laboratory analytical results performed on site soil and groundwater samples. This site information will form the basis for decisions throughout the decommissioning process. Field inspection of the wells prior to decommissioning is also recommended to verify the characteristics and conditions of the wells and to identify special conditions, such as access problems. A sample Monitoring Well Field Inspection Log indicating the minimum information to be collected during field verification activities is included as Figure 1.

2.0 SELECTING THE WELL DECOMMISSIONING METHOD

The primary rationale for well decommissioning is to prevent contaminant migration along the disturbed construction zone created by the original well boring. This requires selection of a decommissioning procedure that takes into account factors such as:

- The hydrogeological conditions at the well site.
- The presence or absence of contamination in the groundwater.
- The original well construction details.

This section presents a summary of the well decommissioning methods and the selection process, which is illustrated in the flow chart presented as Plate 1. The primary well decommissioning procedures consist of:

- Casing pulling.
- Overdrilling.
- Grouting the casing in-place.
- Perforating the casing followed by grouting in-place.

A general discussion of each decommissioning procedure is presented in Sections 2.1 through 2.4.

2.1 CASING PULLING

In general, casing pulling is the preferred method for decommissioning wells where: no contamination is present; contamination is present but the well does not penetrate a confining layer; and when both contamination and a confining layer are present but the contamination cannot cross the confining layer. Additionally, the well construction materials and well depth must be such that pulling can be effected without breaking the riser.

Casing pulling involves removing the well casing by lifting. The procedure for removing the casing must allow grout to be added during pulling. The grout will fill the space once occupied by the material being withdrawn. Grout mixing and placement must be performed according to the procedures in Section 9.0.

An acceptable procedure to remove casing involves puncturing the bottom of the casing, flushing with water to remove sand (if necessary to mitigate lock-up of the casing during pulling), filling the casing with grout tremied from the bottom of the well, using jacks to free casing from the hole, and lifting the casing out by using a drill rig, backhoe, crane, or other suitable equipment. Additional grout must be added to the casing as it is withdrawn. In the event that the casing or well screen is severed during casing pulling, the remaining portion should be removed by overdrilling using the conventional augering method described in Section 2.2.

2.2 OVERDRILLING

Overdrilling is used where casing pulling is determined to be infeasible, or where installation of a temporary casing is necessary to prevent cross-contamination, such as when a confining layer is present and contamination in the deeper aquifer could migrate to the upper aquifer as the well was pulled (see Section 2.5). The overdrilling method should:

- Follow the original well bore.
- Create a borehole of the same or greater diameter than the original boring.
- Remove all of the well construction materials.

TE NAME:

SITE ID.:
INSPECTOR:
DATE/TIME:
WEll ID.:

MONITORING WELL FIELD INSPECTION LOG
NYSDEC WELL DECOMMISSIONING PROGRAM

	YES	NO
ELL VISIBLE? (If not, provide directions below)		
ELL I.D. VISIBLE?		
ELL LOCATION MATCH SITE MAP? (If not, sketch actual location on back)		

ELL I.D. AS IT APPEARS ON PROTECTIVE CASING OR WELL:

	YES	NO
URFACE SEAL PRESENT?		
URFACE SEAL COMPETENT? (If cracked, heaved etc., describe below)		
ROTECTIVE CASING IN GOOD CONDITION? (If damaged, describe below)		

EADSPACE READING (ppm) AND INSTRUMENT USED
YPE OF PROTECTIVE CASING AND HEIGHT OF STICKUP IN FEET (If applicable)
ROTECTIVE CASING MATERIAL TYPE:
EASURE PROTECTIVE CASING INSIDE DIAMETER (Inches):

	YES	NO
OCK PRESENT?		
OCK FUNCTIONAL?		
ID YOU REPLACE THE LOCK?		
THERE EVIDENCE THAT THE WELL IS DOUBLE CASED? (If yes,describe below)		
ELL MEASURING POINT VISIBLE?		

EASURE WELL DEPTH FROM MEASURING POINT (Feet):
EASURE DEPTH TO WATER FROM MEASURING POINT (Feet):
EASURE WELL DIAMETER (Inches):
ELL CASING MATERIAL:
HYSICAL CONDITION OF VISIBLE WELL CASING:
TTACH ID MARKER (If well ID is confirmed) and IDENTIFY MARKER TYPE

ESCRIBE ACCESS TO WELL: (Include accessibility to truck mounted rig, natural obstructions, overhead pow
roximity to permanent structures, etc.); ADD SKETCH OF LOCATION ON BACK, IF NECESSARY.

ESCRIBE WELL SETTING (For example, located in a field, in a playground, on pavement, in a garden, etc.) AND ASSESS
THE TYPE OF RESTORATION REQUIRED.

DENTIFY ANY NEARBY POTENTIAL SOURCES OF CONTAMINATION, IF PRESENT (e.g. Gas station, salt pile, etc.):

REMARKS:

Acceptable methods for overdrilling include the following:

- Using conventional augering (i.e., a hollow stem auger fitted with a plug). The plug cutter will grind the well construction materials, which will be brought to the well surface by the auger.
- Using a conventional cable tool rig to advance casing having a larger diameter than the original boring. The cable tool kit is advanced within the casing to grind the well construction materials and soils, which are periodically removed with large diameter bailer. This method is not applicable to bedrock wells.
- Using an over-reaming tool with a pilot bit nearly the same size as the inside diameter of the casing and a reaming bit slightly larger than the original borehole diameter. This method can be used for wells with steel casings.
- Using a hollow-stem auger with outward facing carbide cutting teeth having a diameter two to four inches larger than the casing. Outward-facing cutting teeth will prevent severing the casing and drifting off center.
- Using a hollow-stem auger with a steel guide pipe inside. The casing guides the cutter head and remains inside the auger. The guide pipe should be firmly attached to the inside of the casing by use of a packer or other type of expansion or friction device.

In all cases above, overdrilling should advance through the original bore depth by a distance of 0.5 feet to ensure complete removal of the construction materials. when the overdrilling is complete, the casing and screen can be retrieved from the center of the auger (American Society for Testing and Materials, Standard D 5299-92, 1992), if one of the hollow stem auger methods described above is employed. After overdrilling is completed, the borehole must be grouted according to the procedures in Section 9.0 and the upper five feet of borehole must be restored according to the procedures in Section 10.0.

2.3 GROUTING IN-PLACE

Grouting in-place is the simplest decommissioning procedure, but offers the least long-term protection of all the methods. As discussed in Section 2.5, however, this method is preferred for the bedrock portion of bedrock wells, and is used for decommissioning cased wells in certain situations. For cased wells, the procedure involves filling the casing with grout to a level of five feet below the land surface, cutting the well casing at the five-foot depth, and removing the top portion of the casing and associated well materials from the ground. The casing must be grouted according to the procedures in Section 9.0. In addition, the upper five feet of the borehole is filled to land surface and restored according to the procedures described in Section 10.0.

For wells installed in bedrock, the procedure involves filling the casing (or open hole) with grout to the top of rock according to the procedures in Section 9.0. The grout mix, however, will vary according to the hydrogeological conditions as discussed in Section 2.5.

It should be noted that for wells located on landfills regulated under 6NYCRR Part 360, the screened interval of the well must be sealed separately and hydrostatically tested to ensure its adequacy before sealing the remaining borehole. The Standard Operating Procedure (SOP) for the hydrostatic test has been included under Appendix D.

2.4 CASING PERFORATION/GROUTING IN-PLACE

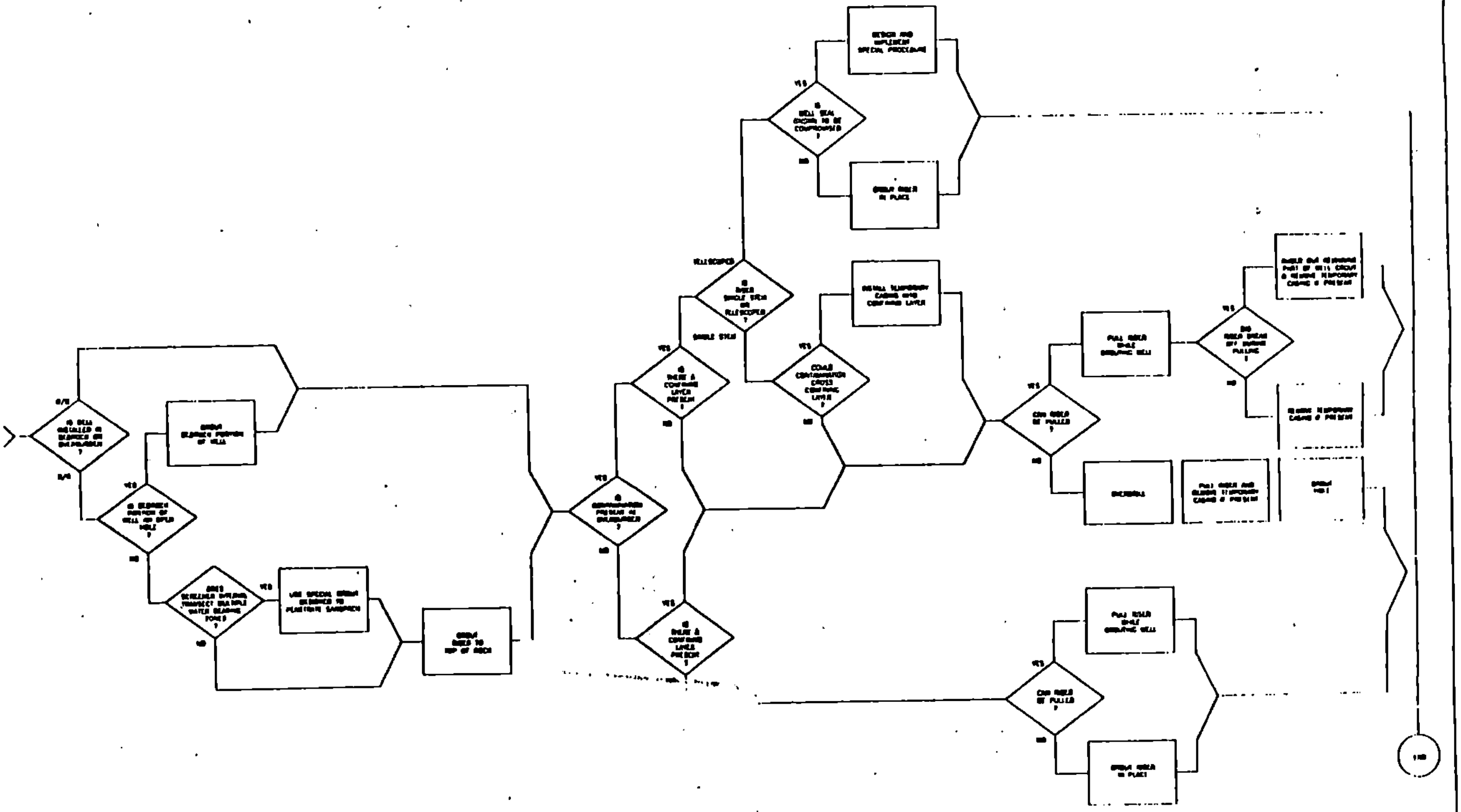
At this time, casing perforation is the preferred method for wells with four-inch or larger inside diameter which are designated to be grouted in-place in accordance with the selection flow chart. The procedure involves perforating the well casing and screen then grouting the well. A wide variety of commercial equipment is available for perforating casings and screens in wells with four-inch or larger inside diameters. Due to the diversity of application, experienced contractors must recommend a specific technique based on site-specific conditions. A minimum of four rows of perforations several inches long and a minimum of five perforations per linear foot of casing or screen is recommended (American Society for Testing and Materials, Standard D 5299-92, 1992).

After perforating is complete, the borehole must be grouted according to the procedures in Section 9.0 and the upper five feet of borehole must be restored according to the procedures in Section 10.0.

2.5 SELECTION PROCESS AND IMPLEMENTATION

Selection of the decommissioning method is governed by the flow chart presented as Plate 1. A discussion of the selection criteria and decommissioning methodology is presented below.

PLATE - 1



MALCOLM PIRNIE

MULTIPLE NPL SITES
WELL DECOMMISSIONING
DECOMMISSIONING PROCEDURE SELECTION
FLOWCHART

NYSDEC

MARCH 1995

2.5.1 Bedrock Wells

As illustrated on Plate 1, the initial decision point in the well decommissioning process considers whether the well is constructed within a bedrock formation. If so, the riser or the open hole portion of the well is grouted to the top of the bedrock. Note that if the bedrock well is cased, the riser should be perforated to the top of the rock if the inside diameter of the casing is 4-inches or larger. Furthermore, if the screened interval transects multiple water bearing zones the special grout mix discussed in Section 9.1.3 should be used to ensure penetration of the sand pack.

After the rock hole is grouted, the overburden portion of the well is decommissioned in accordance with the following sections. If the borehole extends to the surface, no further decommissioning procedures are required, however the boring should only be filled to within 5-feet of the ground surface and site restoration should be completed in accordance with Section 10.0.

2.5.2 Uncontaminated Overburden Wells

For overburden wells and the overburden portion of bedrock wells, the first decision point in determining the decommissioning method considers whether the overburden portion of the well exhibits evidence of contamination, as determined through historical groundwater and/or soil sampling results. If the overburden portion of the well is uncontaminated, the next criteria considers whether the well penetrates a confining layer. In the case that the overburden portion of the well does not penetrate a confining layer, the casing should be pulled (and tremie-grouted) if possible. As a general rule, PVC wells greater than 25-feet deep should not be pulled unless site-specific conditions or other factors indicate that the well can be pulled without breaking. If the well cannot be pulled, such as in the case that a bedrock portion of the well has already been grouted in place, or if the well materials and depth prohibit pulling or will likely result in breakage, the well should be grouted in-place as accordance with Section 2.3 (if the casing is less than 4-inch in diameter) or Section 2.4 (if the casing diameter is 4-inches or larger).

If the overburden portion of the well penetrates a confining layer, the casing should be removed by pulling (if possible) in accordance with Section 2.1. If the casing cannot be removed by pulling, the well should be removed by overdrilling. The overdrilling method used will depend on the site-specific conditions and requirements. If pulling is attempted and fails (i.e., a portion of the riser breaks) the remaining portion of the well should be removed by using the conventional augering procedure identified in Section 2.2. In all cases, after the well construction materials have been removed, the borehole will be grouted in accordance with Section 9.0 and the upper five feet will be restored in accordance with Section 10.0.

2.5.3 Contaminated Overburden Wells

If an overburden well or the overburden portion of a bedrock well is contaminated as evidenced by historical sampling results, the first decision point in selecting a decommissioning procedure is whether the well penetrates a confining layer. If the well does not penetrate a confining layer, the selection process follows the same pathway as for uncontaminated wells that penetrate a confining layer (i.e., the casing is pulled, if possible; otherwise the well is overdrilled - see Section 2.5.2). Plastic sheeting should be placed around the well surface to contain contaminated materials displaced during removal of the well.

For overburden wells that are contaminated and which penetrate a confining layer, the next selection criteria is whether the well riser is a single stem or is telescoped inside one or more outer casings. The procedures to be followed in determining the decommissioning method are presented for both situations below.

2.5.3.1 Single Stem Riser

If the riser is a single stem, the potential for cross-contamination between confining layers must be addressed. In particular, if the lower confining unit is contaminated, there is a potential that the contamination may be transferred to the upper unit as the well construction materials are removed to the ground surface. In this event, it will be necessary to install a temporary casing having a diameter larger than the original borehole into the top of the confining layer. This may be accomplished using a hollow stem auger or by employing a spin and flush technique to advance the casing. If the confining layer is less than 5 feet thick, the casing should be installed to the top of the confining layer. Otherwise, it is installed to a depth of 2 feet below the top of the confining layer. After the temporary casing has been set, the well can be removed and grouted through pulling (if possible) or through overdrilling if pulling is not feasible. Plastic sheeting should be placed around the well surface to contain contaminated materials displaced during removal of the well. As an alternative to installation of a temporary casing, the hollow-stem auger could serve the same purpose in that it would prevent the contamination from migrating to the upper unit. The hollow-stem auger would be advanced into the confining layer until the joint between the uppermost sections was nearly flush with the ground surface, and the sections would be disconnected to expose the riser prior to pulling or overdrilling.

After the casing and screen are removed and the well is grouted, the temporary casing (if used) is removed and the casing and/or hollow stem auger can be decontaminated for reuse. The upper 5 feet of the well surface should then be restored in accordance with Section 10.0.

2.5.3.2 Telescoped Riser

If the riser is telescoped in one or more outer casings, the decommissioning approach is dependent on the integrity of the well seal. For the purpose of the monitoring well decommissioning procedures, the well seal is defined as the bentonite seal above the sand pack. Although it is not possible to visually inspect or otherwise test the well seal to assess its condition, an indication of the well seal integrity may be obtained through review of the boring logs and/or a comparison of groundwater elevations if the well is part of a cluster. Any problems noted on the boring logs pertaining to the well seal, such as bridging of bentonite pellets or running sands, or disparities between field notes (if available) and the well log would indicate the potential for a poor well seal. Alternatively, if the well is part of a cluster a comparison of groundwater elevations between the shallow and deep wells should also be performed. By observing trends at other clusters it may be possible to identify inconsistencies in groundwater elevations at the well slated for decommissioning, thereby indicating a poor well seal.

If there is no evidence that the well seal integrity is compromised, the riser should be grouted in-place in accordance with Section 2.3 or 2.4, depending on the diameter of the well casing, and the upper 5 feet of the well surface should be restored in accordance with Section 10.0. If indications are that the well seal is not competent, it will be necessary to design and implement a special procedure to remove the well construction materials, as the presence and configuration of the outer casing(s) will be specific in the individual wells and will be a key factor in the decommissioning approach. The special procedure should be designed to mitigate the potential for cross-contamination during removal of the well construction materials, and should be designed prior to initiating field work.

3.0 PREPARATION OF A SITE-SPECIFIC HEALTH AND SAFETY PLAN

Prior to initiating decommissioning activities at an NPL site, it is necessary to prepare a site-specific health and safety plan (HASP) in accordance with the requirements of 29 CFR 1910.120. Accordingly, the HASP should include:

- The names of key personnel responsible for site health and safety, including an appointed site health and safety officer.
- A safety and health risk analysis for each site task and operation.
- Employee training requirements.
- Personal protective equipment (PPE) to be used by employees for each of the site tasks and operations being conducted.
- Medical surveillance requirements.
- Frequency and types of air monitoring, personnel monitoring and environmental sampling techniques and instrumentation to be used.
- Site control measures.
- Decontamination procedures.
- Site standard operating procedures.
- A contingency plan for responses to emergencies.
- Confined space entry procedures.

An example of a health and safety plan is attached as Appendix A. This document provides a general framework for preparing a HASP. Examples of site-specific information, such as names of responsible personnel, contaminant data, and other information which must be developed to meet the OSHA requirements discussed above are included in Appendix A but will need to be modified in the site-specific HASP.

4.0 PREPARATION OF A MATERIALS HANDLING AND DISPOSAL PLAN

Materials handling and disposal procedures for each of the wells slated for decommissioning should be identified in a site-specific materials handling and disposal plan. This plan will be used as a guideline to ensure safe and efficient control of contaminated materials, and will promote conformance with the applicable regulatory requirements for storage, characterization, labelling, transportation and disposal of materials prior to off-site transport. For NPL sites, the plan should be reviewed and approved by USEPA prior to initiating construction activities.

4.1 MATERIALS HANDLING PROCEDURES

The materials anticipated to be generated during well decommissioning activities include decontamination fluids, disposable safety equipment (including personal protective equipment), drill cuttings, groundwater, well construction materials (PVC and/or stainless steel casings, well screens, sand, bentonite/grout mixtures, etc.), and any spill-contaminated materials. Proper handling of these materials is effected through a series of steps, including: identification/pre-characterization of the waste materials; segregation/containment of the wastes including storage in proper containers; characterization of the waste materials through analytical testing to determine the absence/presence or nature of the contamination, and proper labeling in accordance with 49 CFR Part 172. Each of these steps is described in the following sections.

4.1.1 Identification/Pre-characterization

Prior to initiating well decommissioning activities at a site, the site history, most importantly historical analytical data from the monitoring wells, must be reviewed as well as the monitoring well construction details: number, type (overburden, bedrock), depth, diameter, and construction materials. This knowledge will aid in estimating the nature and quantities of waste materials which potentially may be generated as a result of decommissioning activities and will also assist in pre-determining the number of roll-off boxes, 55-gallon drums, and any other containers necessary to contain the wastes generated at each respective site.

4.1.2 Segregation and Containment

During well decommissioning activities, generated waste materials must be contained and segregated according to the nature of the suspected contamination. Well materials generated from decommissioning those wells with known contamination will be segregated from materials generated from those wells with little to no contamination (based on historical results). Contaminated materials will be further segregated according to contaminant type (e.g., well materials suspected of containing volatile organic contamination will be segregated from materials suspected of containing Polychlorinated Biphenyl (PCB) contamination).

For wells exhibiting contamination, all materials brought to the surface must either be decontaminated, disposed of at an appropriate Treatment, Storage and Disposal Facility (TSDF), or properly containerized in a secure area for disposal by others. For all uncontaminated wells, the materials (except the casings) can be left at the surface near the former well unless the surrounding land use prohibits this disposal (eg., if the well is located in an area where people could be exposed to the materials left on the surface; or if recovered decommissioning materials would not be consistent with the intended use of the land). In this case, the materials must be disposed of in a 6NYCRR Part 360 landfill. For contaminated wells, PVC and/or steel casing materials may be decontaminated for disposal in a Part 360 landfill, provided that the decontamination effort is thorough and cos effective. Requirements for characterization and disposal of contaminated materials are discussed in Sections 4.1.3 through 4.1.4.

Containment methods will be based on the estimated quantity of materials anticipated to be generated at each respective site. Solid waste materials (i.e., wel construction materials, soils, drill cuttings, PPE), will typically be contained in roll-off boxe or 55-gallon drums. Since federal DOT regulations (49 CFR Part 177) generally limit the combined truck and cargo weight to 80,000 lbs, most hazardous waste transporters will limi the roll-off box capacity to 20 tons of hazardous waste per shipment. Thus, if the materials are to be transported off-site to a treatment, storage and disposal facility (TSDF) tha accepts bulk waste, and if the anticipated quantity of waste will be large (greater than 5 tons), water-tight roll-off containers may be more practical and cost-effective for temporaril containing and transporting the waste in lieu or in combination with 55-gallon drums (e.g., 55-gallon drums may still be used for personal protective equipment or other articles no directly derived from the abandoned well). The roll-off containers should be lined with disposable HDPE liners to prevent contact with the container, and will be initially labeled according to the source(s) of the contained waste materials. Likewise, if drums are used they should be initially labeled according to the source of the contaminated materials. After the contents of the roll-offs and drums have been characterized, they should be labeled in accordance with 49 CFR Part 172. Roll-off containers will be covered with polyethylene tarps during temporary storage and transportation, and all drums will be sealed.

Fluids generated during the decommissioning program will generally be contained in 55-gallon drums unless extremely large volumes are expected; in this case 5,000-gallon tankers or other suitable temporary storage may be used. All drums will be initially labeled according to the wastewater source(s) and will be assumed to contain the same contaminants as the groundwater measured by the particular monitoring well being decommissioned. All 55-gallon drums containing fluids should be sealed and temporarily stored at the decontamination pad until final off-site disposal of at an approved treatment facility.

4.1.3 Characterization

Hazardous waste characterization is necessary to determine the nature of the waste materials, to verify whether the materials are hazardous, and to determine proper disposition. Characterization of waste materials will be conducted at each of the sites to determine the appropriate disposal requirements. The decision as to the number, location and types of samples to be collected will be site specific and will depend on factors such as the quantity of waste generated and type of containers used, the nature of the waste, and the distribution of contaminant types across the site with respect to the origin of the waste materials. In general, the sample collection program will be designed to ensure that analytical data representative of all the materials to be disposed will be generated from the minimal number of samples. This may be accomplished by means such as:

- collection of composite samples for contaminants such as metals and PCBs (compositing is not typically acceptable for volatile organic compound analyses).
- collection of grab samples from select drums/containers suspected of elevated contaminant concentrations based on visual observation (e.g., soil staining, liquid sheen or non-aqueous product) or PID screening

Sample analysis will be based on site history and the requirements of the disposal facility. At a minimum, the samples should be analyzed for the parameters of concern indicated by past monitoring well analytical results, as well as the hazardous waste characteristic parameters: toxicity by TCLP; ignitability; reactivity; and corrosivity in accordance with 40 CFR Part 261.

4.1.4 Labeling

Depending on the nature of the materials, proper labeling of the storage containers (roll-offs and/or drums) must be completed according to 49 CFR Part 172.

4.1.5 Disposal

Disposal of waste materials will depend on whether the waste has been characterized as hazardous or non-hazardous. Non-hazardous waste will be disposed of on-site in accordance with NYSDEC TAGM #4032 with the prior consent of the owner and the Department, or may be landfilled at a permitted 6NYCRR Part 360 facility.

For wastes that exhibit contamination, the requirements for disposal or treatment will be dependent on the waste characteristics. To determine these requirements the following procedure should be followed upon receipt of the waste characterization results:

1) Determine if the waste is characteristically hazardous (by failure of any of the criteria for toxicity, corrosivity, reactivity, or ignitability) or if it is a listed hazardous waste per the classifications identified in 40 CFR Part 261.

2) Determine the EPA hazardous waste code(s) for the applicable waste classification(s) listed in 40 CFR Part 261.

3) Determine any treatment standards for the hazardous waste code(s) per 40 CFR Part 268. Depending on the waste classification, treatment standards may be based on final concentration in the waste/waste extract or may require a specific treatment technology (e.g., incineration).

4) If the hazardous waste contains other constituents that are not listed in the treatment standards, and if landfilling is a disposal option, it should be determined if the waste is a California List waste per the criteria in 40 CFR Part 268.32 (e.g., under these regulations, nonliquid wastes must not contain total halogenated organics at or in excess of 1,000 ppm).

5) If the hazardous waste meets all treatment standards including the California List Standards (if applicable), it may be disposed of at a permitted hazardous

waste land disposal facility. For each shipment the generator is required to provide the following manifest information:

- Hazardous Waste Code(s).
- Corresponding concentration-based or technology-based treatment standards.
- Manifest number.
- Waste analysis data.
- Certification Statement per 40 CFR 268.7(a)(2)(D)(ii).

In addition, the generator is required to maintain the records specified in 40 CFR Part 268.7(a)(7) for a minimum of 5 years.

6) If the waste fails to meet any of the treatment standards listed in 40 CFR Part 268, it must be sent to a treatment, storage, or recycling facility. If the waste's treatment standard is technology-based, it must be treated in accordance with the specified method. Land disposal is not allowable unless the waste is eligible for a National Capacity Variance (40 CFR Subpart C) and meets the California List standards. In all cases, the notification and recordkeeping requirements identified above must be fulfilled by the generator.

The hazardous waste will be transported in accordance with DOT regulations (49 CFR Parts 172-173) to either a secure hazardous waste landfill or TSDF, as appropriate. The contractor will be responsible for arranging for proper transportation and the disposal of the wastes. The Engineer will sign a hazardous waste manifest, as an agent of the Owner.

5.0 EQUIPMENT DECONTAMINATION REQUIREMENTS

Since the monitoring well decommissioning will involve multiple wells, there is a possibility of contamination from one well location to another. To avoid cross-contamination, procedures have been established for decontamination after operations at each well location is complete. The procedures for decontamination of personnel at the site will be specified in the site-specific Health and Safety Plan. Decontamination of equipment will follow established equipment cleaning protocols which are written in accordance with the Engineer's corporate policies and OSHA regulations.

The drilling and excavation equipment (i.e., drill rigs, cutting bits, and associated equipment) will be cleaned at a constructed decontamination facility. In general, the decontamination facility (i.e., decon pad or wash pad) will consist of plywood placed over a heavy synthetic liner. The pad will slope down to a sump that will collect all liquids. A detailed description and drawing of the decontamination facility that will be constructed is included in Appendix B as Item 1.

The drilling and excavation equipment will be prepared before it is brought to the decontamination facility and then cleaned at the facility. The preceding preparation includes removing gross soil/rock from the equipment to minimize losses during movement to the decon pad. At the decontamination facility, the equipment will be rinsed with low-volume water or steam, washed with phosphate-free detergent, and rinsed again with pressurized low-volume water or steam. The equipment will be inspected by the Engineer's field representative after cleaning. The detailed cleaning procedures are included in Appendix B as Item 2.

In the event that sampling equipment must be used, the decontamination guidelines included in Appendix B as Item 3 will be followed. In general, these guidelines describe cleaning with non-phosphate detergent, then performing rinsing cycles with water and acid. After the equipment is air-dried, it must be wrapped in aluminum foil to avoid accidental contamination after cleaning.

After all equipment is decontaminated, the solutions produced must be properly containerized and disposed of. All other disposable contaminated supplies/equipment such as disposable safety and sampling equipment will also need to be properly disposed of. Unless characterization of the decon fluids and disposable equipment is performed in accordance with Section 4.0, these materials will be handled in the same manner as the drill cuttings/fluids from the well locations. All materials must be temporarily stored in a secure area such as the fenced decon pad.

If sampling is necessary, the Engineer's personnel will be responsible for the decontamination of the sampling equipment. The decontamination of drilling and excavation equipment is the responsibility of the Contractor(s). The Engineer's field representative will make daily inspections to insure that decontamination procedures are being followed.

6.0 LOCATING AND SETTING-UP ON THE WELL

The following tasks shall be performed to locate the well to be decommissioned:

- Notify property owner and/or other interested parties including the governing regulatory agency prior to site mobilization whenever possible.
- Review information about the well contained in the site file. This information may include one or more of the following: the site map, well boring log, well construction diagram, field inspection log, well photograph, and proposed well decommissioning procedure.
- Verify the well location and identification by locating the identifying marker.
- Verify the depth of the well in the well construction log by sounding with a weighted tape.

After the well has been located, the decommissioning procedure should be selected in accordance with Section 2.0 based on the available boring and sampling data. The rig must be set up prior to initiating drilling to ensure proper alignment with the well (i.e., the drill string must be aligned with the monitoring well).

7.0 REMOVING THE PROTECTIVE CASING

7.1 GENERAL

Removal of the protective casing of a well must not interfere with or compromise the integrity of decommissioning activities performed at the well.

The procedure for removing the protective casing of a well depends upon the decommissioning method used. When a well is decommissioned by the overdrilling or casing pulling method, the protective casing may be removed either before or after the casing is removed. When the decommissioning procedure requires casing perforation or grouting in-place, the protective casing should be removed after grout is added to the well. The protective casing handling and disposal must be consistent with the methods used for the well materials, unless an alternate disposal method can be employed (e.g., steam cleaning followed by disposal as nonhazardous waste).

7.2 PRIOR TO SEALING THE WELL BORE

When overdrilling, the protective casing must be removed first, unless the drilling tools have an inside diameter larger than the protective casing. The variety of protective casings available preclude developing a specific removal procedure. In all cases, however, the specific procedure used must minimize the risk of:

- breaking the well casing off below ground and
- allowing foreign material to enter the well casing.

If the decommissioning method used is casing pulling, the decision of when to remove the protective casing is not critical.

An acceptable protective casing removal method involves breaking up the concrete seal surrounding the casing and jacking or hoisting the casing out of the ground. A check should be made during pulling to insure that the inner well casing is not being hoisted with the protective casing. If this occurs, the well casing should be cut off after the base of the protective casing is lifted above the land surface.

7.3 AFTER SEALING THE WELL

If the decommissioning method used allows well casing to remain in the ground, the protective casing should be removed after the well has been properly filled with grout. This will insure that the well is properly sealed regardless of problems with protective casing removal. During grouting in-place, the well casing must be removed to a depth of five feet below the land surface. The upper five feet of casing and the protective casing can be removed in one operation if a casing cutter is used. If the height of the protective casing makes working conditions at the well awkward, the casing can be cut off at a lower level. However, the inner well casing must remain aboveground and cannot be damaged in any way that prevents the well from being filled with grout.

8.0 DECOMMISSIONING OF SCREEN AND RISER

After setting up on the well and removing the protective casing (if necessary), the well screen and riser are decommissioned in accordance with the appropriate procedure and methodology as discussed in Section 2.0 (i.e., if the wells are overdrilled or pulled, the casing and riser are removed. Otherwise, they are perforated and/or grouted in-place). During the decommissioning activities the requirements of the site-specific health and safety plan, materials handling and disposal plan and equipment decontamination plan will be followed to ensure maximum protection of human health and the environment.

9.0 SELECTING, MIXING, AND PLACING GROUT

9.1 SELECTING GROUT MIXTURE

There are two types of grout mixes that may be used to seal wells: a standard mix and a special mix. Both mixes use Type 1 Portland cement and four percent bentonite by weight. However, the special mix uses a smaller volume of water and is used in situations where excessive loss of the standard grout mix is possible (e.g. highly-fractured bedrock or coarse gravels).

9.1.1 Standard Grout Mixture

For most boreholes, the following standard mixture will be used:

- One 94-pound bag Type I Portland cement
- 3.9 pounds powdered bentonite
- 7.8 gallons potable water

This mixture results in a grout with a bentonite content of four percent by weight, and will be used in all cases except in boreholes where excessive use of grout is anticipated. In these cases a special mixture will be used (see Section 9.1.2).

See Section 9.2 for grout mixing procedures.

9.1.2 Special Mixture

In cases where excessive use of grout is anticipated, such as high permeability formations and highly fractured or cavernous bedrock formations, the following special mixture will be used:

- One 94-pound bag type I Portland cement
- 3.9 pounds powdered bentonite
- 1 pound calcium chloride
- 6.0-7.8 gallons potable water (depending on desired thickness)

The special mixture results in a grout with a bentonite content of four percent by weight. It is thicker than the standard mixture because it contains ~~less~~ water. This grout is expected to set faster than the Standard Grout Mixture. The least amount of water that can be added for the mixture to be readily pumpable is six gallons per 94-pound bag of cement.

See Section 9.2 for grout mixing procedures.

9.1.3 Alternate Special Grout

In cases where the penetration of the sandpack is critical, such as bedrock wells with screens that transect multiple water-bearing zones, the following alternate mixture will be used:

- One 94 pound bag Type III Portland Cement.
- 3.9 pounds powdered bentonite.
- 7.8 gallons potable water.

Refer to Section 9.2 for grout mixing procedures. It should be noted that this grout is expected to set faster than the standard grout mixture.

9.2 GROUT MIXING PROCEDURE

To begin the grout-mixing procedure, calculate the volume of grout required to fill the borehole. If possible, the mixing basin should be large enough to hold all of the grout necessary for the borehole. Tall cylindrical and long shallow basins should not be used as it is difficult to obtain a homogeneous mixture in these types of basins.

Mix grout until a smooth, homogeneous mixture is achieved. No lumps or dry clots should be present. Grout can be mixed manually or with a mechanized mixer. One acceptable type of mixer is a vertical paddle grout mixer. Colloidal mixers should not be used as they tend to excessively decrease the thickness of the grout for the above recipes.

9.3 GROUT PLACEMENT

Grout will be placed in the borehole from the bottom to the top using a tremie pipe of not less than 1-inch diameter. Grout will then be pumped into the borehole until the grout appears at the land surface (when grouting open holes in bedrock, the grout level only needs to reach above the bedrock surface). Any groundwater displaced during grout placement will be pumped via suction lift to a 55-gallon drum for proper disposal.

At this time the rate of settling should be observed. When the grout level stabilizes, casing or augers will be removed from the hole. As each section is removed, grout will be added to keep the level between 0-feet and 5-feet below land surface. If the grout level drops below the land surface to an excessive degree, an alternate grouting method must be used. One possibility is to grout in stages; i.e., the first batch of grout is allowed to partially cure before a second batch of grout is added.

Upon completion of grouting, insure that the final grout level is approximately five feet below land surface. A ferrous metal marker will be embedded in the top of the grout to indicate the location of the former monitoring well.

10.0 BACKFILLING AND SITE RESTORATION

The uppermost five feet of the borehole at the land surface will be filled with a material appropriate to the intended use of the land. The materials will be physically similar to the natural soils. No materials will be used that limit the use of the property in any way. The surface of the borehole will be restored to the condition of the area surrounding the borehole. For example, concrete or asphalt will be patched with concrete or asphalt of the same type and thickness, grassed areas will be seeded, and topsoil will be used in other areas. All solid waste materials generated during the decommissioning process will be disposed of properly.

11.0 QUALITY ASSURANCE/QUALITY CONTROL (QA/QC) PROCEDURES

This section describes the quality control/quality assurance (QA/QC) procedures necessary for monitoring and ensuring the Contractor's adherence to the Monitoring Well Decommissioning Project procedures, plans and specifications, prepared by the Engineer.

This section will discuss the minimum inspection and documentation requirements necessary to facilitate proper well decommissioning procedures and also will:

- Review the general requirements specified in the Contract Documents.
- Define roles and responsibilities of all parties.
- Establish the key tasks to be monitored by the on-site construction inspector and the appropriate inspector forms and logs to be used for recording the Contractor's activities.
- Establish procedures for communicating change orders, field modifications and variations from the Contract Documents to the Owner.
- Establish scheduled meetings and briefings during the construction phase.

The overall goal of the project QA/QC program is to ensure that proper well decommissioning techniques and procedures are used in accordance with the requirements of the Contract Documents. The QA/QC procedures herein should be followed by QA personnel including: Construction Contractor personnel, the Contractor's subcontracted laboratory and field personnel, and the Engineer's on-site construction inspector.

11.1 RESPONSIBILITY AND AUTHORITY

The principal organizations involved in developing, designing and conducting well decommissioning activities are the Owner, Engineer, and the Construction Contractor.

11.1.1 Owner

The Owner will be responsible for reviewing the well decommissioning procedures to determine whether the documents meet their requirements, and to obtain approval of the procedures from the appropriate regulatory agencies. The Owner will have the responsibility and authority to review and accept or reject any design or procedural revisions or requests. The Owner also has the responsibility and authority to review and approve the Construction Monitoring Report and all QA documentation collected during well decommissioning activities.

11.1.2 Engineer

The Engineer will be responsible for reviewing and approving any engineering design changes, construction monitoring and quality assurance in accordance with this QA Plan. The Engineer will inform all parties involved with construction of their responsibilities, lines of communication, lines of authority, and QA/QC procedures. The Engineer's construction inspector (QA Engineer) will monitor decommissioning activities and will be assigned specific responsibilities and tasks. Most of the waste sample collection and testing will be conducted by the contractor at a frequency and manner specified in the site specific Materials Handling and Disposal Plan.

The person filling the construction inspector (QA Engineer) position will be trained and certified to operate an HNu organic vapor photoionization detector (PID), will be OSHA 40-hour Hazardous Waste Worker trained and will have a working knowledge of documents pertaining to well decommissioning activities, including this plan. The Engineer's field personnel will be instructed to contact the construction inspector (QA Engineer) in the event well decommissioning requirements are not being met, QA procedures are not being implemented, or construction problems have been encountered.

11.1.3 Construction Contractor

In addition to performing the monitoring well decommissioning in accordance with the design documents, the Contractor will be required to obtain the services of a qualified testing laboratory to perform the analytical testing of the waste materials and will also be responsible for procuring transportation and disposal/treatment services.

11.2 PROJECT MEETINGS

The Engineer's management of the monitoring well decommissioning project will include conducting periodic project meetings as described below:

11.2.1 Pre-construction Meeting

The Engineer will schedule and attend one (1) pre-construction meeting in the Buffalo area for the purpose of discussing the project approach and answering contractor questions. The Engineer will also prepare and distribute meeting minutes. The meeting will also:

- Provide each party (organization) with relevant QA documents and supporting information.
- Familiarize each organization with the QA Plan and its role relative to the well decommissioning criteria and construction documents.
- Review the responsibilities of each organization and review the lines of authority and communication for each organization.
- Discuss the established procedures for observations and tests including waste sampling.
- Discuss the established procedures for handling construction deficiencies, repairs, and/or retesting.
- Review methods for documenting and reporting inspection data.

11.2.2 Monthly Progress Meetings

Monthly project meetings will be held during the course of the work to discuss the project schedule and work performed to date, and to address and resolve any existing or anticipated problems.

A special meeting will be held when and if a major QA problem or deficiency is present or likely to occur. At a minimum, the meeting shall be attended by the Construction Contractor and the Engineer's on-site inspector (QA Engineer). The purpose of the meeting will be to define and resolve the problem(s) or deficiencies encountered. The meeting minutes will be documented by the Engineer.

11.3 KEY TASKS

The key tasks that the Engineer will conduct during the well decommissioning project are briefly summarized below.

11.3.1 Review of Contractor Submissions

Prior to well decommissioning activities, all written submissions required by the contract documents will be evaluated and forwarded to the Owner, together with written submissions regarding their suitability. The Engineer will also obtain and review all necessary shop drawings, material tests and as-built drawings submitted throughout the construction and will make recommendations for acceptance/rejection to the Owner. The contractor's progress will be continuously monitored during the construction period, and Owner will be informed of the schedule and any corrective measures planned or implemented.

Throughout the project, payment requests by the contractor will be reviewed for accuracy and completeness prior to making recommendations relative to payment. Review will involve comparing actual notes of field personnel to items contained in the payment request. Discrepancies will be discussed with the contractor and will be amended if necessary.

11.3.2 Construction Inspection

The Engineer will provide full-time inspection of the contractor during all critical well decommissioning activities at each of the sites. This will be accomplished by providing an experienced on-site inspector(s) to document the contractor's adherence to the contract specifications and monitoring the contractor's progress. The Engineer will notify the Owner in the event that the contractor fails to perform the decommissioning work as specified in the contract and recommend to the Owner the acceptance, conditional approval/disapproval or rejection of the contractor's work. The Engineer will issue instructions, field orders, interpretations and clarification of contract language to the contractor as required. In the event that a change order is necessary, the Engineer will submit the change order with a detailed cost estimate to the Owner. The Engineer will also document, evaluate and recommend a course of action for all disputes and claims with the contractor.

In addition, the Engineer will inspect, evaluate and document the monitoring well condition after the well has been removed.

11.4 DOCUMENTATION

The Engineer's on-site construction inspector will document all monitoring well decommissioning activities. Such documentation will include, at a minimum, daily reports of construction activities, photographs, and sketches as necessary. Field investigation reports will be completed by the construction inspector when major questions arise at the site. Forms to be used for this purpose are presented in Appendix C.

The Engineer will maintain complete and detailed records associated with all construction and related activities during the duration of the project. These records will be maintained at the Engineer's office(s) and will include but not be limited to the following:

- Daily work completed and important conversations.
- Contractor's daily use of personnel, material and equipment.
- Records documenting the contractor's deviation from work as specified in the contract documents, and any instructions issued regarding deviations.
- Unusual circumstances (weather conditions, labor disputes, environmental problems, health and safety hazards encountered, etc.).
- General files including correspondence and other documentation related to the project.
- Job meeting minutes with documentation on resolution of issues raised.
- Records of contractor's submittals including shop drawings, modifications/change orders, soil tests, material tests and action taken (e.g.,Owner approval/disapproval, further information needed).
- Construction photos.
- Telephone conversation

In addition, the Engineer will submit monthly Project Summary Reports to the Owner. These reports will identify the work which has been accomplished and will document the status of each monitoring well at each site where decommissioning work has occurred.

Upon substantial completion of the decommissioning activities at each site, the Engineer will prepare a detailed list of any work remaining unfinished. The Engineer will then prepare and submit a written notice to the Owner which will include a determination as to whether the completed work meets the requirements of the contract documents. Following satisfactory completion of the work, the Engineer will perform a final inspection of the site and submit a notice to the Owner that decommissioning activities were performed in accordance with the contract documents as revised by any approved change orders or modifications to the scope of work.

Documentation on the condition of the removed wells with respect to the impacts of hazardous waste, minerals and other pertinent environmental factors, or discernable through direct observation, will be presented to Owner along with any recommendations for future well installation techniques and materials.

12.0 SOURCES

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Walker, Brian. Personal Communication, Global Drilling Suppliers, Inc. 1992

EXAMPLE

APPENDIX A

HEALTH AND SAFETY PLAN

Appendix ___: Item ________ - HYDRAULIC PRESSURE TESTING OF SCREENED INTERVAL

Applicability: HAZARDOUS WASTE Revision No.: ___ Date: ________

Prepared By: BGP Date: 4/28/95 Approved By: ___ Date: ________

1.0 INTRODUCTION

This guideline presents a method for evaluating the integrity of a grout seal in the screened interval of a well being decommissioned by grouting in place.

2.0 METHODOLOGY

1. Grout the screened interval of the well using a tremie pipe, up to a level of one to two feet above the screened section.
2. Allow the grout to set for a period of not less than 24 hours and not greater than 72 hours before pressure testing of the grouted interval is begun.
3. Place a pneumatic packer at a maximum of four and one half feet above the top of the screened section of the well casing.
4. Apply an inflation pressure to the packer, not exceeding the pressure rating of the well casing material. If the interval between the top of the grout and the bottom of the packer is not saturated, use potable water to fill the interval.
5. Apply a gauge pressure of 5 psig at the well head to the interval for a period of 5 minutes to allow for temperature stabilization. After 5 minutes maintain the pressure at 5 psig for 30 minutes.
6. The grout seal shall be considered acceptable if the total loss of water to the seal does not exceed 0.5 gallons over a 30-minute period.
7. If the grout seal is determined to be unacceptable, an additional 5 feet of grout will be added to the well casing with a tremie pipe. The interval will be retested as described above.

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Appendix D

Siltation and erosion controls should be specified on plans, construction specifications and in bid documents. If controls are to be designed by the contractor, specific plans should be submitted for review prior to initiation of construction.

C. Plans for the Restoration/Replacement of Resources

If mitigation for the loss of fish and wildlife resources through restoration or replacement is required as part of the selected remedial alternative, plans should be submitted for review during the design step. Mitigation may include plans for on-site or off-site restoration or replacement of affected resources. An acceptable plan should be available prior to the initiation of remedial activity.

Step V - Monitoring Program

Scope and Objectives

In the development of a design for the implementation of the remedial action, plans for monitoring evaluation of compliance with design specifications should be included for affected fish and wildlife resources. In addition, a monitoring program, if specified in the selection of the remedial alternative, should be designed for evaluating the efficacy of the selected remedial alternative in minimizing risk to fish and widldlife resources.

A. Compliance with Design

A program for on-site inspection and evaluation to insure that the implementation of the selected alternative complies with design specifications pertinent to fish and wildlife resources should be developed and include appropriate criteria for evaluating monitoring results. Monitoring techniques may include sampling of media (soil, water, sediments etc.) and tissue sampling or bioassays to confirm the removal of contaminants, and evaluation of biological populations to insure activties are not detrimental to biota. Long term evaluations of biological communities may be necessary to insure that mitigation for lost resources is adequate. An acceptable program should be available prior to the implementation of the remedial design. If monitoring results do not meet criteria, the design and methods used for implementation of the selected remedial action should be re-evaluated.

B. Monitoring the Efficacy of the Remedial Action

If, in the selection of the remedial program, a monitoring program was indicated to insure that the remedial action is effectively minimizing the risk of site-related contaminants to fish and wildlife resources, approprite monitoring techniques should be selected for the specified components (affected resources, migration pathways, contaminants of concern). Monitoring techniques may include the sampling of various media (water, sediment, soils etc.), tissue sampling, toxicity tests, biomonitoring, and the monitoring of trends in population densities or community diversity. Long-term sampling schedules and evaluation criteria should be established. If monitoring indicates that criteria have been exceeded, the selection of the remedial alternative should be re-evaluated.

APPENDIX A

NEW YORK STATE DEPARTMENT OF ENVIRONMENTAL CONSERVATION INFORMATION SOURCES

A. SIGNIFICANT HABITATS PROGRAM AND NATURAL HERITAGE PROGRAM FILE INFORMATION

STATEWIDE REQUESTS

Include a brief narrative of the proposed project and a photocopy of the appropriate topographic quadrangle(s) with the site or sites identified when requesting information from the files. All requests should be addressed as follows:

ATTN: Information Services
Significant Habitat Unit
NYS Dept. of Environmental Conservation
700 Troy-Schenectady Road
Latham, New York 12110
(518) 783-3932

CONTACT: Kathy Schneider

REGIONAL REQUESTS

REGION 1 (Nassau, Suffolk Counties)

NYS Department of Environmental Conservation
Region 1
SUNY Campus, Building 40
Stony Brook, New York 11794
(516) 751-7900

CONTACT: Mike Scheibel

Appendix D

APPENDIX D

FISH AND WILDLIFE IMPACT ANALYSIS FOR INACTIVE HAZARDOUS WASTE SITES DATED JUNE 1991

Fish and Wildlife Impact Analysis for Inactive Hazardous Waste Sites

prepared by

New York State Department of Environmental Conservation

Division of Fish and Wildlife

June 18, 1991

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Appendix A New York State Department of Environmental Conservation Environmental Resource Information Sources

Appendix B References

Introduction

This document provides guidance for the evaluation of fish and wildlife concerns associated with the remediation of inactive hazardous waste sites and supercedes the preceding "Habitat Based Assessment" (HBA). The objectives set forth for each step within this document and in the HBA are substantially the same. However, this document indicates at what points in the investigative process certain information is required and when additional information may be necessary for a complete evaluation of site conditions and proposed remedial alternatives. This document should lead to an efficient evaluation of impacts on fish and wildlife resources associated with inactive hazardous waste sites.

The required elements for a complete impact analysis are outlined in the Table of Contents. This outline should serve as a checklist in the development of the Analysis. The Analysis is a stepwise process requiring decisions at a number of points in its development. Decisions may be made to eliminate elements of the process given the results at that point in the Analysis. Decision points in the Analysis process are indicated under Objectives and Scope for each step of the Analysis process. Decisions made to eliminate elements of the Analysis must be supported by sufficient information. If doubt exists that sufficient information exists to support a decision that would eliminate an element, staff of the Inactive Hazardous Waste Evaluation Unit in the Division of Fish and Wildlife should be consulted.

Although the Fish and Widlife Impact Analysis may be completed independently, greater efficiency may result from the integration of this Analysis with the remedial process developed by the Division of Hazardous Waste. The Objectives and Scope indicate at what point in the overall remedial process specific elements of the Analysis should be addressed to create an efficient remedial process.

Step I - Site Description

Objectives and Scope

The objectives of Step I are 1) to identify if fish and wildlife resources that may be affected by site-related contaminants are present or were present prior to contaminant introduction and 2), if resources are or were present, to provide appropriate information for designing a remedial investigation of these resources. Step I provides information in the form of maps, site descriptions, and description of the utility of resources. This information is necessary to identify potential pathways of contaminant migration that may affect fish and wildlife resources. If resources and potential pathways are evident, information obtained during Step I may be required to select locations, contaminants of concern, and types of media for sampling during the remedial investigation.

The information outlined in Step I under headings A through C is most useful prior to the work plan stage in the remedial investigation. If available early in the site investigation, a decision can be made as to whether additional ecological investigation (including field sampling) is required for the site. If required, the ecological studies can be incorporated into the overall study design of the remedial investigation thereby maximizing its efficiency and avoiding later phases in the investigation.

Applicable fish and wildlife regulatory criteria including Applicable or Relevant and Appropriate Requirements (ARARs), Standards, Criteria, and Guidance (SCGs), and To Be Considered (TBCs) should be identified for the assessment of site-related contamination and the determination of remedial objectives. Criteria will be necessary to perform an adequate analysis of impact of contaminants on fish and wildlife resources. Similarly, criteria are necessary to evaluate contaminant-specific and site-specific ecological effects associated with proposed remedial alternatives. Consequently, criteria should be identified after contaminants and resources have been identified but prior to conducting a contaminant-specific impact analysis for fish and wildlife resources in the remedial investigation or evaluating remedial alteratives in a feasibility study.

A complete description as outlined in Step I is generally necessary for sites with fish and wildlife resources potentially affected by site-related contaminants. However, if the contention that no resources are associated with the site or, if resources are present, potential for contaminant migration to resources does not exist can be supported with limited information, only the necessary supporting information should be obtained.

A. Site Maps

1. Topographic map - A topographic map showing the location of the site and documented fish and wildlife resources such as NYSDEC Significant Habitats as defined by the NYS Natural

Heritage Program (Ecological Communities of New York State, 1990), habitats supporting endangered, threatened, or rare species, species of concern, regulated wetlands, wild and scenic rivers, significant coastal zone areas, streams, lakes, and other major resources within two miles of the perimeter of the site should be provided (suggested scale: 1 inch equals 1000 feet). If major resources exist downstream of the site farther than two miles and are likely to be affected by site-related contaminants, a topographic map indicating the location of these resources should be included.

2. Covertype map - A covertype map should be drawn for the site and an area within 0.5 miles from the perimeter of the site (suggested scale: one inch equals 500 feet). The base map may be derived from aerial photos, ground-level photos, USGS topographic maps, soil maps etc. Major vegetative communities including wetlands, aquatic habitats, NYSDEC Significant Habitats, and areas of special concern should be included. The NYSDEC Natural Heritage Program descriptions and classifications of natural communities may be used to identify the covertypes; however, unique covertypes not described by the Natural Heritage Program should be identified and mapped. A qualified biologist should supervise covertype identification. If the map is drawn from secondary sources (e. g. aerial photos, descriptions from the literature etc.), limited field checking of the accuracy of the covertypes and vegetative species should be done.

The topographic and covertype maps should include an outline of the perimeter of the site. Maps should be drawn to a scale that permits features to be easily read. Copies <u>**must**</u> be legible.

B. Description of Fish and Wildlife Resources

1. Fish and Wildlife Resources and Covertypes - Identified fish and wildlife resources should be described. The description of aquatic resources should include a discussion of chemical and physical parameters such as water chemistry, temperature, DO, depth, substrate composition, discharge, flow rates, gradients, stream-bed morphology etc. Submergent aquatic vegetation should be identified and its abundance and distribution described. Wetland and stream classifications should be included. For covertypes, typical vegetative species and their abundance, distribution, and densities should be discussed. In addition to field data, NYSDEC files, EPA, US Fish and Wildlife Service, local bird clubs, colleges etc. may serve as sources of information (Appendix A).

[illegible]

2. Fauna Expected Within Each Covertype and Aquatic Habitat The fish and wildlife species expected to be associated with each covertype and aquatic habitat should be determined. Endangered, threatened, rare species, and species of of concern should be noted. In addition to field data, NYSDEC files, US Fish and Wildlife Service, National Marine Fisheries Service, local bird clubs, colleges, standard natural history references etc. may serve as sources of information (Appendix A).

3. Observations of Stress Potentially Related to Site Contaminants - A description of obviously contaminated areas such as stained soils, leachate seeps, exposed waste etc. and alterations in biota such as reduced vegetative growth and density, wildlife mortality, alterations in expected distribution of wildlife species, changes in species assemblages, or obvious abscence of expected biota that may be related to contaminant exposure should be reported. Records of past fish and wildlife contamination and/or mortality potentially associated with the site should be obtained from appropriate sources (Appendix A).

C. Value of Fish and Wildlife Resources

1. Value of Habitat to Associated Fauna - A qualitative assessment of the general ability of the area within 0.5 miles of the site to provide habitat for fish and wildlife species should be conducted. The degree to which the habitats meet the requirements for food, seasonal cover, bedding areas, breeding and roosting sites etc. should be discussed. Qualitative assessments of population densities and diversity of fish and wildlife species that habitats within 0.5 miles of the site may support should be included.

2. Value of Resources to Humans - The current and potential use of fish and wildlife resources by humans should be assessed. Resources on site, within 0.5 miles of the site, and documented resources within 2 miles and those downstream of the site that are potentially affected by contaminants should be included. Human use of fish and wildlife resources may include hunting, fishing, observation of wildlife, scientific studies, agriculture, forestry, and other recreational or economic activities. APPENDIX A includes possible sources of information.

D. Applicable Fish and Wildlife Regulatory Criteria

The identification of criteria applicable to the remediation of fish and wildlife resources should include contaminant-specific and site-specific criteria. Examples of contaminant-specific criteria applicable to fish and wildlife concerns include water quality standards/guidance values for the protection of aquatic life (6 NYCRR Part 701 and TOGS 1.1.1) and sediment criteria developed by the Division of Fish and Wildlife

(Sediment Criteria - December 1989). Site-specific criteria include but are not limited to Freshwater Wetlands Act and implementing regulations (Article 24 ECL, 6 NYCRR Part 663 and 664), Tidal Wetlands Act (Article 25 ECL, 6 NYCRR Part 661), and laws and regulations regulating streams and navigable water bodies (Article 15 ECL, 6NYCRR Part 608). Identification of site-specific criteria should include any substantive requirements for permit issuance that are cited in the regulation. The responsibilities of the Division of Fish and Wildlife include the regulation and maintenance of fish and wildlife resources for human use; consequently, criteria that reflect this role have been developed by the Division and other agencies and should be identified as applicable.

Step II - Contaminant-Specific Impact Analysis

Objectives and Scope

The objective of Step II is to determine the impacts of site-related contaminants on fish and wildlife resources. The impacts on fish and wildlife resources are dependent on the contaminants of concern, concentrations of contaminants in various media, the exposure of biota to contaminants, and toxicological effects. The impact analysis should either demonstrate that impacts on resources due to contamination are miminal or, if significant impacts exist, determine the impacts of site-related contaminants on productivity and diversity of fish and wildlife resources.

The development of the contaminant-specific impact analysis should follow a stepwise process. Step II presents three steps of increasing complexity (Pathway Analysis, Criteria-Specific Analysis, and Analysis of Toxicological Effects) that assess the impacts of site-related contaminants to fish and wildlife resources. Each step progressively relies on more specific information and fewer conservative assumptions in the analysis of impact. Whether the impact analysis progresses through additional steps will depend on the conclusions reached at each step regarding the degree of impact. If minimal impact can be demonstrated at a specific step in the analysis, additional, more information intensive steps should not be conducted.

Information for the impact analysis is dependent on Step I and the results of the characterization of contaminants and their distribution developed in the remedial investigation. Although the final impact analysis need not be reported until the last phase of the remedial investigation, information available for the impact analysis be reviewed and, if possible, steps for which information is available should be conducted during the initial phase of the remedial investigation. If these analyses do not demonstrate a minimal impact on fish and wildlife resources, the collection of additional information during subsequent phases of the remedial investigation may be required to complete the impact analysis.

A. Pathway Analysis

As an initial step in the impact analysis, a pathway analysis should be conducted. Fish and wildlife resources, contaminants of concern, sources of contaminants, and potential pathways of contaminant migration and exposure should be identified. If no significant resources or potential pathways are present, impact on resources can be considered minimal. Similarly, impact is minimal to fish and wildlife resources if results from field studies demonstrate that contaminants have not migrated to a resource along any potential pathway. Demonstration of minimal impact using a pathway analysis will eliminate the need for additional analyses.

B. Criteria-Specific Analysis

A criteria-specific analysis presumes that the presence of contaminated resources and pathways of migration for site-related contaminants have been established. This analysis requires the use of numerical criteria for contaminants of concern associated with specific media or biota. Numerical criteria may be directly available or derived according to methods established as part of the criteria (ARARs, SCGs, TBCs etc.). For example, numerical water quality criteria may be developed by applying methodology outlined in 6 NYCRR Part 701. Impact is assessed by comparing contaminant levels with numerical criteria. If contaminant levels are below criteria, impact on the resource is considered minimal and additional analyses are not required. If numerical criteria are exceeded or do not exist and cannot be developed through the application of methodolgy that is consistent with criteria, an analysis of toxicological effects is required to demonstrate the degree of impact.

C. Analysis of Toxicological Effects

An analysis of toxicological effects should be conducted if a Criteria-Specific Analysis cannot be applied due to lack of numerical criteria or if application of criteria indicate that contaminant levels exceed criteria. As in the Criteria-Specific Analysis, the toxicological analysis presumes that fish and wildlife resources have been identified and the contamination of resources and contaminant pathways have been confirmed.

An analysis of toxicological effects may be conducted using a number of analytical approaches: indicator species, population, community, and/or ecosystem analyses. The approach selected will depend on the complexity of the ecology, the relative importance assigned to specific biota, the modes of exposure to contaminants, and the expected degree of toxicity associated with the level of contamination. More than one analytical approach may be required to adequately characterize the toxicological and concomitant ecological effects. Impact is assessed by determining the degree to which contaminants affect the productivity of populations, communities, or ecosystems and the diversity of species assemblages, communities, or entire ecosystems through direct toxicological and indirect ecological effects.

1. **Indicator Species Analysis** - A toxicological analysis based on toxicological data for an indicator species is applicable if the ecology of the resource and exposure scenarios are simple. This approach is applicable if only a few dominant species are present or if a few species of a resource with a complex species composition have a high resource value such as a ecological system containing an endangered species. Exposure to contaminants is assumed to be continuous throughout the entire life cycle and does not vary among individuals or with life stages. Acute and chronic toxicity should be evaluated from the perspective of a full life cycle or most sensitive life stage of selected species. The analysis should consider the effects of contaminants on growth, reproduction, and behavior. The level of exposure should be derived from the evaluation of site-related contaminant data.

2. **Population Analysis** - A population level analysis is relevant to the evaluation of chronic toxicological effects of contaminants to entire populations or the evaluation of acute contaminant exposure limited to specific classes of organisms within a population. This analysis should include an evaluation of the degree of exposure to contaminants experienced by specific classes within the population. Exposure scenarios should be developed from site-specific data. The analysis should assess the toxicological effects of contaminant exposure on the ecological dynamics of specific populations (age structure, recruitment, survival rates etc.). Ultimately, the analysis should assess the impact on productivity within a population due to contaminant exposure.

3. **Community Analysis** - For ecological communities with highly interdependent species, an analysis of alternations in diversty due to contaminant exposure may be necessary. For example, communities that have highly specialized predators dependent on a limited array of prey species (simple food webs), communities with highly competitive species (high niche overlap), or communities whose composition and diversity are maintained by

key-stone species are likely to undergo alterations in community structure as a result of the toxicological effects of contaminants on one or more species within the community. Benthic communities in streams often exhibit a considerable alteration in species composition and community structure due to the introduction of contaminants. This analytical approach may require site-specific data describing the species composition and structure of affected ecological communities. The analysis should indicate the extent to which species composition and community structure within an ecological community are altered by exposure to contaminants.

4. **Ecosystem Analysis** - The toxicological effects and concomitant ecological effects of contaminants may be analyzed from the perspective of trophic dynamics. The analysis should include an evaluation of direct toxicological and indirect ecological effects on productivity resulting in contaminant-related alterations in trophic structure and function.

An analysis of effects of contaminant exposure on trophic structure and function should be conducted if contaminants are expected to uniformly affect physiolgical processes that are associated with energy transformation within a specific trophic level. For example, if contaminants affect photosynthetic reactions of primary producers or affect common chemical processes regulating the metabolism of decomposers, an analysis employing trophic concepts may be appropriate in characterizing the toxicological and ecological effects of the contaminant.

An analysis of materials transfer among trophic levels should be considered if trophic function is regulated by limited nutrient availability due to the presence of contaminants or if contaminants are likely to be transferred among trophic levels. For example, iron, a potential contaminant, may reduce the availability of phosphorous in a phosphorus-limited system. More commonly, an analysis of materials transfer on trophic structure and function should be applied in the evaluation of toxicological implications of contaminant transfer. Bioconcentration, bioaccumulation, biomagnification etc. are concepts that may be used to evaluate the potential effects of contaminant transfer on trophic dynamics.

Study Methods

Performing a contaminant-specific impact analysis may require specific toxicological or ecological information. A number of methods that are useful in the development of appropriate information are listed:

1. Sampling and analysis of tissues obtained from biota collected from contaminanted and uncontaminated areas.

2. Bioaccumulation calculations supported by the analysis of contaminated media and biota.

3. The modelling of environmental fate of contaminants.

4. *In situ* and laboratory toxicity tests of contaminated and uncontaminated media.

5. Histopathological studies of populations exposed to contaminants.

6. Collection and comparison of population density, diversity, or species richness data from contaminated and uncontaminated areas.

7. Contaminant-specific toxicological data obtained from literature sources for biota known to inhabit the site and surrounding area.

8. Evaluation of potential use of fish and wildlife resources by humans from information available in surveys and records.

Step III - Ecological Effects of Remedial Alternatives

Objectives and Scope

The objective of Step III is to evaluate the remedial alternatives relative to their effects on the productivity and diversity of fish and wildlife resources affected by site-related contamination. This requires the consideration of potential non-contaminant related impacts of remedial activity as well as the efficacy of remedial alternatives in correcting contaminant-specific effects. Remedial alternatives are evaluated using Contaminant-Specific Impact Analysis as a "base line". Similarly, non-contaminant related impacts are evaluated using the ecological information provided in Step I as a "base line" for comparison. Before concerns for fish and wildlife resources can be weighed relative to other concerns (human health, cost criteria etc.) in selecting the preferred alternative, further evaluation that weighs positive and negative aspects associated with both non-contaminant and contaminant-specific ecological effects of each remedial alternative and a subsequent comparision of alternatives is required.

The evaluation of ecological effects of the remedial alternatives should be conducted as part of the feasibility study. The effectiveness of remedial alternatives in achieving desired ecological effects and meeting other concerns should be evaluated concurrently. The evaluation process should clearly indicate the weight given to concerns for fish and wildlife resources versus other concerns. The discussion of the selection of the preferred alternative and selected alternative in the Proposed Remedial Action Plan (PRAP) and the Record of Decision (ROD), respectively, should indicate how and to what extent the remedial action will address concerns for fish and wildlife resources.

The need for a monitoring program should be determined. The degree of removal or destruction of contaminants under the remedial action is critical to determining the need for a monitoring program. If a monitoring program is required, components of the program including ecological resources, pathways, and contaminants of concern should be identifed in the PRAP and ROD.

A. Evaluation and Comparison of Alternatives

1. Contaminant-related effects - Contaminant-related effects include alterations in production and diversity related to the direct or indirect effects of contaminant toxicity. Direct effects include mortality, morbidity, alterations in behavior and reproduction etc. induced by exposure to contaminants. Indirect effects include alterations in species assemblages, ecological communities, and ecosystem function due to loss or reduction of biotic components caused by contaminant exposure.

The remedial alternatives should be compared initially to contaminant-specific "base line" conditions. The Contaminant-Specific Impact Assessment describing the current impacts on resources should serve as a "base line". The evaluation of each alternative should indicate whether contaminant-specific criteria (ARARs, SCGs, and TBCs) are satisfied. If criteria cannot be satisfied under one or more remedial alternatives, a comparision of alternatives should be conducted to establish the relative efficacy of alternatives in restoring and/or maintaining the productivity and diversity of affected fish and wildlife resources.

2. Non-contaminant related effects - Non-contaminant related effects include alterations in the productivity and diversty of fish and wildlife resources due to the loss or modification of habitat. Remedial actions may eliminate habitat through construction activities or affect the productivity and diversity of ecological communities through the modification of factors that affect habitat quality such as hydrological regime, soil conditions, adjacent plant communities etc.

Remedial alternatives should be compared initially to "base line" conditions to determine their potential for significant impact on productivity or diversity of resources due to habitat loss or modification. The initial evaluations of each remedial alternative should address whether site-specific criteria (ARARs, SCGs and TBCs) are met. If site-specific criteria indicate that a remedial alternative may result in detrimental effect to a resource, further delineation and description of the resource may be necessary during the feasibility study for the development of appropriate mitigation. The evaluation of each alternative should include mitigation strategies for potential loss, modification, or possible enhancement of habitat. Effects should be categorized as long or short term. If significant impact is expected from one or more alternatives, the relative potential impact of these alternatives on productivity and diversity of resources should be categorized.

B. Discussion of Ecological Factors in Selecting a Preferred Alternative

The overall risk of alternatives to the productivity and diversity of fish and wildlife resources should be compared. Comparisons should include potential positive and negative aspects of contaminant-specific and non-contaminant related effects of remedial action. The alternative that will best restore and/or maintain the productivity and diversity of affected resources should be identified as the alternative that minimizes risk to affected resources.

The role of ecological concerns relative to other concerns in the selection of a preferred alternative should be discussed. If the preferred alternative does not minimize risk to affected resources, an explanation should be provided indicating why achievement of minimization of risk to fish and wildlife resources is not possible and the extent the preferred alternative fails to meet this goal.

C. Conceptual Monitoring Program

1. Evaluation of Need for Monitoring - The remedial alternative ultimately selected as the remedial remedy should be evaluated to determine if a monitoring program is required. Sites that are remediated by containment or partial removal of contaminants to levels that do not minimize risk to resources will require a post-remedial monitoring program. Monitoring programs will not be required for remedial actions that remove or destroy contaminants to levels that minimize risk to resources.

2. **Components of Monitoring Program** - The objectives of the monitoring program are to determine 1) if remedial measures meet expectations for minimizing risk to fish and wildlife resources and 2) if remedial measures for minimizing risk to fish and wildlife resources are effective from a long-term perspective. The identification of affected resources, migration pathways, and contaminants of concern should be specified.

Step IV - Implementation of Selected Alternative in Design

Scope and Objectives

The design phase of a remedial action involves the implementation of the selected remedial alternative. This requires refinement in areas to be remediated or protected and design plans for remedial construction. Consequently, delineation, methods of protection, and plans for restoration, if necessary, need to be addressed for fish and wildlife resources. Specific information should be included on project plans or in construction specifications.

A. Delineation of Affected Fish and Wildlife Resources

Although Steps I and III generally indicate the location of fish and wildlife resources and identifies those that are contaminated and require remediation, a detailed delineation of resources affected by contamination or construction activity may be required at the design step. Because the remedial investigation may provide only general information regarding the contaminant migration, delineation of the extent of contamination may be required for the design of remedial measures for some affected resources. Often, contamination affecting flowing waters requires further delineation in the design phase. The delineation of uncontaminated resources that may be affected by construction activities may be necessary. For example, delineation of wetlands is usually required if construction activities are anticipated near the wetlands.

B. Methods of Protection for Fish and Wildlife Resources

Specific methods for the protection of affected resources should be indicated on plans and in construction specifications. For example, siltation and erosion controls and a limitation as to time of year for construction activities are often required.

Siltation and erosion controls should be specified on plans, construction specifications and in bid documents. If controls are to be designed by the contractor, specific plans should be submitted for review prior to initiation of construction.

C. Plans for the Restoration/Replacement of Resources

If mitigation for the loss of fish and wildlife resources through restoration or replacement is required as part of the selected remedial alternative, plans should be submitted for review during the design step. Mitigation may include plans for on-site or off-site restoration or replacement of affected resources. An acceptable plan should be available prior to the initiation of remedial activity.

Step V - Monitoring Program

Scope and Objectives

In the development of a design for the implementation of the remedial action, plans for monitoring evaluation of compliance with design specifications should be included for affected fish and wildlife resources. In addition, a monitoring program, if specified in the selection of the remedial alternative, should be designed for evaluating the efficacy of the selected remedial alternative in minimizing risk to fish and widldlife resources.

A. Compliance with Design

A program for on-site inspection and evaluation to insure that the implementation of the selected alternative complies with design specifications pertinent to fish and wildlife resources should be developed and include appropriate criteria for evaluating monitoring results. Monitoring techniques may include sampling of media (soil, water, sediments etc.) and tissue sampling or bioassays to confirm the removal of contaminants, and evaluation of biological populations to insure activties are not detrimental to biota. Long term evaluations of biological communities may be necessary to insure that mitigation for lost resources is adequate. An acceptable program should be available prior to the implementation of the remedial design. If monitoring results do not meet criteria, the design and methods used for implementation of the selected remedial action should be re-evaluated.

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Appendix B

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APPENDIX E

STANDARD OPERATING PROCEDURE FOR PORTABLE GAS CHROMATOGRAPH

STANDARD OPERATING PROCEDURE
FOR
PORTABLE GAS CHROMATOGRAPH (PHOTOVAC 10S PLUS)

1. Fill gas chromatograph with carrier gas (zero air).

2. Turn instrument on.

3. Run instrument blank (perform an analysis without injecting a sample).

4. Run standards (calibration). Calibration procedures will be developed on a site-specific basis. Calibrations will be based on chemicals of concern. Standard analysis must agree within ±20% of existing calibration. If within 20%, proceed to Step 5, if not, rerun standard as a calibration then proceed to Step 5.

5. Analyze samples in accordance with applicable standard operating procedure.

6. Run a standard check every 8 hours. Proceed as in No. 4.

10S+ TRAINING EXERCISES

INTRODUCTION:

These exercises are designed to operate the 10S + from the PC: most functions may be accessed via the PC or the 10S Plus. Those functions which must be entered on the PC will be **Highlighted**. The statement "*Click on*" is meant for the PC user, users with only the 10S + may simply move to the referenced item and press enter.

PART A: SYSTEM FUNCTION

Objectives

- Fill unit with carrier gas.
- Turn on instrument.
- **Load/Install Software onto PC (Optional).**
- Transfer OS, GC, VOC functions to the instrument Ram Card(s) (Optional)
- Set Time and Date.
- Review SYSTEM FUNCTION.

1. Fill unit with carrier gas up to 1500 PSI.

2. Connect 10S Plus to a PC.

3. Turn instrument ON.

4. **If Photovac 10S Plus Icon does not appear in windows Program Manager, the 10S Plus PC software must be installed onto the PC. If so insert diskette into drive, select the Run command in the File menu of the Program Manager. Type a:\setup in the command line (if diskette is in the a: drive) and select OK. A window will open *Initializing 10S+ Setup*, then another window will open select continue, next enter the directory path and select Continue. The default drive is the C:\ select continue for the sub-directory. Another window will open, *10S Plus Disk 1*, this window will display the setup progress. When the transfer is complete, the message *Setup succeeded*, will appear, select OK. Now open the 10S Plus icon from the PC Window, and double *click on* the 10S Plus GC icon to load the program onto the PC. Data will then be transmitted to the 10S Plus GC. Check the GC status, in the upper left corner of the PC display, it should read 10S Plus not detected.**

5. **If the OS, GC and VOC (if available) functions are not installed on the instrument and or the Ram Card they must be transferred and loaded. To transfer the functions: Be sure the 10S Plus is properly connected to the PC and the correct communications ports are selected and connected. Also be sure there is enough free memory on the Ram Card (s) you are sending the functions to. On the PC open the File menu select transfer 10S + Ram Disk, then select Initialize and select 10S Plus Operating System and select send. Answer Yes if the 10S Plus is displaying the message Please insert System Card. After transferring the OS you can now transfer the GC function and then the VOC function if applicable. To transfer the GC function select Initialize in the Transfer dialog box, next select 10S + GC function and select send and select begin transfer, the status will be updated and inform you when the transfer is complete.**

6 Within the Operating System of the 10S Plus press the COMMAND key, and Move the cursor using the arrow keys, to the **ANALYZER** command.

7. Open the **ANALYZER** window by pressing ENTER.

8. If the 10S Plus is connected Windows will automatically update the time and date on the 10S Plus. When using the 10S Plus alone the time and date can be set in this window.

9. Open the **COMMUNICATIONS** window, select SET UP, set the Baud Rate to 9600.

10. Open each command window in turn and review the options of the Operating System :

FILE:	Allows the user to Load Total VOC and GC Functions.
VIEW:	Allows the user to view the Alarm Log and select file types to view.
ANALYZER:	Gives analyzer status information, allows the user to select options for power, display and audio alarm, also sets the date and time and sets the instrument up to Auto On.

COMMUNICATIONS: Sets up the modem connections for communicating with the instrument remotely, selects the alarm dial information for calling up a PC from the instrument if a fault or alarm condition is encountered and hangs up remote connection.

HELP: Will display the correct OS software version. And will give the user on line help screens specific to the location of the cursor and **full help screens on the PC by topic, menu or search.**

11. **Attach printer to computer.**

12. **Ensure correct printer is selected in Windows Print setup window**

PART B: LOADING THE GC FUNCTION

Objectives
- Load the GC Function.
- Become familiar with the GC Commands.

1. Once the functions have been sent to the instrument Ram Card they must be loaded on the 10S Plus instrument , this must be done from the instrument keypad. In the OS default screen.
2. Use **EXIT** to ensure that no Command windows are open.
3. Use the **ARROW** keys to select the GC Function (GC.bin).
4. Press **ENTER**. A check will appear next to the file name.
5. Press **COMMAND**. The Command menu will appear.
6. Select **FILE**
7. Press **ENTER**. Select RUN. Press ENTER. The GC.bin will appear in the box, use the arrow keys to select OK. The GC Function will load and the window will close.
8. Press **FCN**. The GC Function Results Screen will appear.
9. Use the **FCN** key to toggle between the System Function and the GC Function.

   If available you may move over to the PC now. Status on the PC should now read Detector Off.

10. Using the **mouse** move the cursor to **FILE**.

11. Starting with the FILE menu, open each menu in turn and review the software.

FILE:	Files are opened, saved, printed and **transferred** from this window.
VIEW:	The appearance of data on the screen can be changed in this window. Options include: Disk Log, Avg/Max, Analysis Report/Graph (the chromatogram), **and Graph (a concentration vs. time graph). Up to three (3) compounds can be graphed at a time, Concentrations or Areas can be graphed against Time.** The appearance of the chromatogram may be changed using the options for: Range, Original View, Last View, Zoom In, Normalize, and Grid.
LOG:	Controls the logging of data to the GC **and PC**. The user has six different report formats that can be logged. These include: Chromatogram Results, Analysis Result Log, Alarm Log, Calibration Log, Avg/Max Report, and Fault Log. Options include logging to the following devices: 10S + RAM disk, **disk** and Printer. Up to 4 Avg/Max Periods can be set from this window.
METHOD:	Controls all GC functions. Options include: GC Method, Integration Method, Timing, Library, choice of Loop or Syringe, Reintegration and **Chromatographer software.**
ANALYZER:	Gives all Analyzer status information, records Notes and allows the user to put in an instrument location. Also Starts and Stops an analysis, sets the instrument up to Auto Run and sets Analog output settings.
COMMUNICATIONS:	**Allows the communications parameters to be setup. Options include: Setup, Phone list, Alarm dial, Hang up, and File Transfer.** (see OS for these commands on the GC)

HELP: Will give the user on line help screens specific to the location of the cursor and **full help screens on the PC by topic, menu or search.** Help key on the instrument keypad will provide similar information.

NOTE: If you are not connected to a PC, all the menus will appear the same on the instrument software as on the PC software just described above.

PART C: FLOW BALANCING AND INSTRUMENT WARM-UP

Objectives

- Turn on PID.
- Warm up Instrument.
- Retrieve a Method.
- Balance Flows.

1. Attach flow meter to DET OUT, check for flow through detector. Flow must be at least 1 mL/min.

2. Open the <u>ANALYZER</u> window. Then open the **STATUS** window. This can be done from either the PC or the instrument.

3. Turn on the PID, by *clicking on* **GC DETECTOR ON**. A beep will sound when the lamp is tuned. The column oven will begin to warm up, when the lamp comes on close the Status Window.

4. Open the <u>FILE</u> window. *Click on* **OPEN**.

5. To Recall a Method:
   PC: Select the drive and *click on* "aromatic.gc" *click on* OK. aromatic.gc" is now recalled.
   GC: Move cursor to aromatic.gc, press enter, move to open press enter.

6. Open the **<u>METHOD</u>** window.

7. Select **GC METHOD** Option.

8. Note the instrument flows. These are for notation purposes only.

9. Attach the digital flow meter to the DET OUT port on the 10S Plus.

10. Use the CARRIER CONTROL (the black controller) to set the flow to 10 mL/min (for these exercises). Turning this controller clockwise will increase the total carrier gas going into the system, therefore the flow through DET OUT will increase, as will the flow through BKFLUSH OUT.

11. Attach the flowmeter to the BKFLUSH OUT port and use the BACKFLUSH CONTROL (the silver controller) to make the flow through this port equal to the DET OUT flow. Turning this controller counter-clockwise will increase the flow through the BKFLUSH OUT port and decrease the flow through the DET OUT port.

12. Reattach the flowmeter to the DET OUT port and measure the flow. If necessary, readjust the flow to 10 mL/min.

13. Measure the flow through the BKFLUSH OUT port <u>and</u> the DET OUT port once again, readjusting the flow rate with the appropriate flow controller, if necessary, to ensure that the flows are now balanced.

14. Close the **<u>METHOD</u>** window by selecting OK.

PART D: LOOP INJECTION

Objectives
- Selecting Injection Type.
- Changing Analysis Times
- Running a Loop Injection.
- Storing a compound in the library.
- Re-integrating a run.
- Storing an analysis.

1. Open the **METHOD** window.

2. Select Loop. The window will close.

3. Open the **METHOD** window, open the **TIMING** window.

4. Change the **ANALYSIS TIME** from 470 seconds to 100 seconds. Use the mouse to move the cursor to the Analysis time box. *Click on* **OK**.

5. Open the **METHOD** window.

6. *Click on* **LIBRARY**, move the cursor to **ERASE** and *click on* **ERASE**. The cursor will appear over Benzene. *Click on* **ERASE** three times to erase all library entries. *Click on* **OK**.

7. Check that the GC Status line reads the name of the recalled file. If it is not, then ask the instructor for guidance.

8. Connect a BTX standard bag to the Sample In Port.

9. Open the **ANALYZER** window.

10. *Click on* START ANALYSIS. **Select and *click on* SAMPLE. The run will start.** When using the 10S Plus alone, press RUN/AUTO, choose Sample. The run will start.

11. After the run is complete the Benzene peak may be stored in the library. To store a compound in the library:

-*Click on* METHOD window.
-*Click on* LIBRARY.
-*Click on* STORE.
- **When adding information to boxes which already contain data that needs to be updated, use either the Mouse to highlight the box or Tab over to the box which needs to be changed (Highlighted).**
-When using the 10S Plus alone move the cursor to the information which must be added and press enter.
-Move the cursor to **Peak #** and type in the Benzene Peak number.
-Move the cursor to **Name** and type in the compound name.
-**Tab** or **Mouse** over to the Alarm 1 level and type in 0.5 PPM. (for these exercises)
-**Tab** or **Mouse** over to the Alarm 2 level and type in 1.0 PPM. (for these exercises)
-**Tab** or **Mouse** over to the Conc (PPM) and type in the concentration of the standard.
-*Click on* STORE then **OK** then *Click on* **OK** again.

12. Re-open the **METHOD** window.

13. *Click on* **RE-INTEGRATE**.

14. To store the analysis:

- Open the FILE window.
- Choose **SAVE AS....**
- Select **drive** and Type in your name for the file name, *Click on* **OK**
- To ensure that the file is now stored, Move the cursor to **FILE** and *click on* **OPEN**. *Click on* **OK**. The stored files will be listed in the window. *Click on* CANCEL

PART E: SYRINGE INJECTION

Objectives

- Running a syringe injection.
- Retrieving a library.

1. Open the <u>**METHOD**</u> window.

2. Select **SYRINGE** injection.

3. Open the <u>**METHOD**</u> window and ***Click on*** **TIMING**.

4. Change the ANALYSIS TIME from 100 seconds to 500 seconds. ***Click on*** OK.

5. Check that the file name is in the upper left corner.

6. When the instrument status is RECALLED or READY, then a run may be made. To make a syringe injection:

   -Take a 100ul sample from the Tedlar bag.
   -Using the PC, open the <u>ANALYZER</u> window.
   -Select START ANALYSIS and select SAMPLE.
   -Or using the GC press the RUN AUTO key select SAMPLE and press ENTER.
   -The timer will start. A beep will sound from 4.5-5 seconds.
   -Make the injection when the timer has stopped, at 5 seconds.

7. When the run is over, only Benzene will be identified. The correct concentration is not given for Benzene. Benzene must be erased from the library.

   -Open the <u>**METHOD**</u> window.
   -*Click on* **LIBRARY**.
   -*Click on* **ERASE.**
   -The cursor will now appear over Benzene, ***click on*** **Benzene,** ***click on*** **OK.**
   - Or if using the GC alone highlight Benzene and press the ENTER key.

8. Store the Benzene peak in the library.

9. Store Toluene and m-Xylene also.

10. Open the <u>**METHOD**</u> window.

11. Move the cursor down to **RE-INTEGRATE** the chromatogram. Benzene, Toluene, and m-Xylene will be identified and quantified.

PART F: METHOD DEVELOPMENT

Objectives

- Review Method Software Options.
- To create a new method.

1. Open the **METHOD** window.
2. Open the **GC METHOD** option.
3. Type in the new DETECTOR and BACKFLUSH FLOW rates.
4. Set the OVEN TEMPERATURE. *Click on* **OK.**
5. Open the **METHOD** window, select the appropriate injection type.
6. Open the **METHOD** window again and then, open the **TIMING** option.
7. Enter the appropriate PUMP TIME, CAL TIME and ANALYSIS TIME, if a loop injection has been chosen. If a syringe injection was chosen, then enter the INJECTION SIZE and the ANALYSIS TIME.
8. *Click on* **CONFIG.**
9. Valve timing tables will appear. The program may be changed if required.
10. Close **CONFIG** window. *Click on* **OK** to accept the changes and close the **TIMING** windows.
11. Open the **METHOD** option. *Click on* **LIBRARY** window.
12. Move cursor to ERASE and click. Move the cursor to the desired compound to be erased. *Click on* as many compounds as required to erase all library entries.
13. Close **LIBRARY** window.
14. Open the **METHOD** window and select the **INTEGRATION METHOD** option.

15. Choose Automatic or Manual Selection of integration. Automatic selection will provide the best Up Slope, Dn Slope, Min. Hght, and Filter. Choose an appropriate Integration Delay, Min. Area, Window % ***click on*** **OK**.

16. Prepare a standard mixture. Connect the bag to "Sample In" if LOOP was checked. Make a manual injection if SYRINGE was checked.

17. Store the compounds in the library.

18. Save the Method to the card or to the PC disk.

PART G: CALIBRATION PROCEDURE

Objectives

- Updating the library with CAL run.

1. Retrieve the method saved in PART F.

2. If the CALIBRANT gas will be introduced via syringe, or Tedlar bag, go to step 3, if the calibrant will be introduced via pressurized cylinder, go to step 10.

3. Open the **METHOD** window.

4. Open the **LIBRARY** option.

5. ***Click on*** **CAL CONC (PPM) enter the concentration of the compound you wish to Cal on.**

6. Repeat step 5 for each of the compounds in the calibrant mixture. *Click on* **OK.**

7. If a syringe is being used, prepare a syringe full of the calibrant gas; use the same size injection as was used for the Sample. If a Tedlar bag is being used, open the bag and attach it to the CAL inlet.

8. *Click on* ANALYZER . Select Start Analysis. *Click on* Calibration . Or if using the 10S Plus only press RUN/AUTO, select **CAL** and press enter. If using a syringe, make the injection at the beep. The Calibration is complete when the analysis is finished.

To prepare for a pressurized calibrant.

9. Open the **METHOD** window.

11. Open the **LIBRARY** option.

12. Be certain that there are no CAL concentrations entered. If there are, use the mouse to move the cursor to the CAL concentration and enter Zero ***click on*** **OK.**

13. Set up the pressurized Calibrant gas at 5 PSIG. Be certain that it is attached to the CAL inlet.

14. Open <u>**ANALYZER**</u> select Start Analysis. Select CAL. Or if using the GC Press RUN/AUTO select **CAL** and press Enter. The calibrant gas will be run, and when the chromatogram is complete, a CAL fault will appear.

15. Run a second pressurized CAL to be certain that the entire system was purged.

16. After the second run, enter the given concentrations for each compound using CAL Concentration PPM column in the LIBRARY window.

17. Run a third pressurized CAL to remove the CAL fault.

18. Avg factor/Store Avg

19.

20.

21.

PART H: CHROMATOGRAPHER

NOTE: **The CHROMATOGRAPHER application can only be run within the 10S Plus PC software, it is not available on the GC.**

Objectives

- Open Chromatographer Application.
- Become familiar with Chromatographer functions.

1. Select and *Click on* METHOD window.

2. *Click on* CHROMATOGRAPHER... . The CHROMATOGRAPHER default screen will appear.

3. Using the mouse move the cursor to FILE.

4. Starting with the FILE window, open each window and review the software.

FILE
Files are started, opened, closed, added to background, viewed, saved and printed from this window. The user can also return to the GC software or exit from the application from this window.

VIEW
The Tool bar, Status bar, and Dialog bar can all be added to or removed from the screen in this window. The appearance of the chromatogram can be changed; options include: Range, Normalize, Zoom In, Original View, Last View and Grid. The background chromatogram can be hidden from the foreground if it is behind the foreground chromatogram. An artificial offset can be placed on the background for a 3D effect.

METHOD
Controls all GC function options including: GC Method, Integration Method, Timing, Library, and Notes.

MODIFY
Gives the user various peak integration patterns and the option of user override for manually drawn baselines. All of the options in the MODIFY window are also within the Tool bar at the top of the screen.

WINDOW
Several chromatograms can be opened and arranged as windows that can be arranged so that they overlap, or as non-overlapping tiles. This allows different chromatographic results to be compared.

HELP
Gives an Index of Help Topics for the Chromatographer application. A section on How to Use Help and the current Chromatographer software version.

Objectives

- Recall stored chromatograms.
- Add chromatograms to the background.
- Skew Background
- Save chromatograms to disk.
- Print chromatographic results.

1. *Click on* FILE. *Click on* OPEN. Select an analysis report to be opened be sure that the Results, Method, Library and Notes are selected to be recalled in the recall box. *Click on* OK. The file you selected should now appear in the Chromatographer window.

2. To Add another chromatogram to the background. *Click on* FILE. *Click on* ADD TO BACKGROUND... Select the analysis file you wish to add and click on OK. Up to nine chromatograms can be added to the Background.

3. To place an artificial offset on the background chromatogram for a 3D effect, *Click on* VIEW and select SKEW BACKGROUND.

4. *Click on* FILE. *Click on* SAVE AS... Type in the File Name in the file name box. Select File Type, Drive and Directory and *click on* OK.

5. To print the new chromatogram, *Click on* FILE. *Click on* PRINT SET UP be sure the correct printer, Orientation, Paper and Print Options are selected, when complete select PRINT... If all information in the Print dialog box is correct select OK.

Objectives

- Adjust RANGE
- Zoom In on a small portion of a chromatogram.

1. To adjust the chromatogram amplitude scale, between 0 and 2 mV and 0 and 1000 mV, *Click on* VIEW, select RANGE and *click on* a scale. The range will change to the new selected range. The Original View will always go back to the Range selected here.

2. To Zoom In on a portion of the chromatogram, using the mouse line the mouse pointer up to the area you wish to Zoom In on and Click the mouse button. The mouse pointer (↗) will change to a crosshatch (+). Move the crosshatch to select the area (a box will appear around the area to be zoomed).

3. Once you have selected the area move the mouse pointer to the *Zoom In* button. *Click on* Zoom In and the area selected will be enlarged and fill the screen. You can continue to Zoom In up to 9 times.

4. To exit from the Zoomed View, select Original View, the original chromatogram will return to the screen.

5. If you would like the view to be normalized *Click on* the *Normalize* button.

Objective

- Adjust chromatogram integration setting and reintegrate.

NOTE: Be sure that a chromatogram is on the screen.

1. *Click on* MODIFY. *Click on* MODIFY BASELINE. Select and *click on Integrate as a single peak.*

2. Using the mouse move the pointer to within the slope of Peak #.... (name). Click the mouse pointer. The integration of the selected peak will change.

3. Now try to reintegrate the test chromatogram using the remaining peak I integration types listed in the Modify baseline menu or select from the choice of peak integration type buttons in the toolbar.

PART I: TOTAL VOC OPERATION WITH GC

Objectives

- Loading the VOC Function.
- Becoming familiar with VOC Commands.

NOTE:
1) Be certain that GC Function is loaded.
2) Be certain that the Total VOC Function is not loaded.

1. Load VOC Function. From the OS of the 10S Plus instrument.
2. Press **FCN** until the 10S Plus Operating Function appears on the screen.
3. Move the cursor to VOC.bin.
4. Press **ENTER**. A check will appear next to the file name.
5. Press **COMMAND**. The command menu will appear.
6. Select **FILE.**
7. Press **ENTER**. Select RUN. Press **ENTER** , VOC.bin will appear in the box use the arrow keys to select OK. The VOC function will load and the window will close.
8. Use the **FCN** key to toggle between the Operating, GC and Total VOC Functions.
9. Press **FCN** until the Total VOC Function Operating Screen appears.

   If available you may move over to the PC now. Status on the PC should read Detector Off.

10. Using the ***mouse*** move the cursor to file.
11. Move the cursor to the NOTES command.

12. Starting with NOTES open each Command menu from the right to the left in turn.

NOTES: Notes may be added to an analysis here.

AUTO: All automatic operations are set up here.

PRINT: Printouts may be obtained using these options.

METHOD: The Total VOC Method may be programmed here.

STATUS: Gives the Total VOC instrument status.

PART J: TOTAL VOC FUNCTION METHOD DEVELOPMENT AND OPERATION.

Objectives
- Devising a VOC Method.
- Calibrating the VOC.
- Using Autostart GC on Total VOC Alarm 1.

1. Press **COMMAND**.

2. Open the Status window.

3. Select VOC Detector ON and press **ENTER**.

4. Open the METHOD window.

5. Set the Alarm 1 level at 100 PPM and the Alarm 2 level at 300 PPM.

6. Set the Range to 1000 PPM.

7. Enter the appropriate SPAN GAS CONCENTRATION.

8. Now calibrate the instrument

   -Be certain that the Total VOC is sampling clean air.
   -Move the cursor to ZERO and press **ENTER**.
   -When the "Calibrating Please Wait" message disappears the unit has been zeroed.
   -Now attach the SPAN gas (Isobutylene) to the AUX 1 port.
   -Move the cursor to SPAN and press **ENTER**.
   -When the "Calibrating now Please Wait" message disappears the unit has been spanned. The Method Window will automatically close.

9. Reopen the Method Window. Move the cursor to Set Period and press ENTER. A message appears on the screen "Data will be lost! Are you sure (Y/N)".

10. Press Y.

11. Select the 0.5 hr period and press **ENTER**. After the menu closes, a message "This period has ended" appears. A new period will start.

12. The VOC Function can now be demonstrated by holding a magic marker over the AUX 1 port.

13. Press **RUN AUTO**.

14. Select NEW and press **ENTER**. Again the message "This Period has Ended" appears. A new Period will start.

15. Press **COMMAND**.

16. Open the AUTO window.

17. Select Auto Start GC on Alarm and press **ENTER**.

18. Hold a magic marker over the AUX 1 port; the GC will start if Alarm 1 is exceeded.

19 The 10S Plus may now be turned **OFF**. All loaded functions and programs will remain in resident memory. The Applications card may be removed and stored.

APPENDIX F

EQUIPMENT MANUALS

MiCROTiP™ HL-2

Hand Held Air Monitor/Photoionization Detector

CLASS 1, DIVISION 2
GROUPS A, B, C, D
HAZARDOUS LOCATIONS
Classified by Underwriters Laboratories Inc. as to electrical shock, fire and explosion hazards only.

User's Manual

Photovac International Incorporated

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Telephone 516 351-5809
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PHOTOVAC
incorporated

Photovac Inco
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Telex Answerback:

Part N

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WARNING: THIS EQUIPMENT GENERATES, USES AND CAN RADIATE RADIO FREQUENCY ENERGY AND IF NOT INSTALLED AND USED IN ACCORDANCE WITH THE INSTRUCTION MANUAL, MAY CAUSE INTERFERENCE TO RADIO COMMUNICATIONS. IT HAS BEEN TESTED AND FOUND TO COMPLY WITH THE LIMITS FOR DOC STANDARD C108.8 AND FOR A CLASS A COMPUTING DEVICE PURSUANT TO SUBPART J OF PART 15 OF FCC RULES, WHICH ARE DESIGNED TO PROVIDE REASONABLE PROTECTION AGAINST SUCH INTERFERENCE WHEN OPERATED IN A COMMERCIAL ENVIRONMENT. OPERATION OF THIS EQUIPMENT IN A RESIDENTIAL AREA IS LIKELY TO CAUSE INTERFERENCE IN WHICH CASE THE USER AT HIS OWN EXPENSE WILL BE REQUIRED TO TAKE WHATEVER MEASURES MAY BE REQUIRED TO CORRECT THE INTERFERENCE.

NOTICE

S DEVICE IS CLASSIFIED FOR USE IN CLASS I, DIVISION 2, GROUPS A,B,C,D ZARDOUS LOCATIONS.

omplies with Underwriters Laboratories Inc. UL 1604 Standard for Electrical Equip-nt for use in Hazardous Locations Class I, Division 2.

S DEVICE IS NOT INTENDED FOR CONSTANT USE WITH FLAMMABLE CONCEN-ATIONS OF GASES.

se Photovac accessories are also for use with the device in a hazardous location:

395004 headset
390006 3 meter (9.8') sample line
390022 9.5 eV lamp
390024 11.7 eV lamp

not use any other accessories with this device in a hazardous location.

bstitution of components may affect safety rating.

CAUTION

reduce the risk of fire or injury to persons, read and follow these instructions:

All calibration, maintenance and servicing of this device, including battery charging, must be performed in a safe area away from hazardous locations.

For replacement battery pack use only Photovac part number 395014.

Do not dispose of the battery pack in a fire. The cell may explode. Check with local codes for possible special disposal instructions.

Do not open or mutilate the battery cells. Released electrolyte is corrosive and may cause damage to the eyes or skin. It may be toxic if swallowed.

Exercise care in handling batteries in order not to short the battery with conducting materials such as rings, bracelets and keys. The battery or conductor may overheat and cause burns.

Do not defeat proper polarity orientation between the battery pack and battery charger.

Charge the battery pack provided with or identified for use with this product only in accordance with the instructions and limitations specified in this manual.

Chapter 1 Introduction

1.1 UNPACKING

Included with the instrument you will find the following standard accessories:

- User's Manual
- 120/230 Volt Battery Charger
- 17 cm (6.8")Sample Line
- 5 Spare Filter Cartridges
- Shoulder Strap

Remove MicroTIP™and accessories from the shipping box and e ine them for any physical damage. Inform Photovac immediately MicroTIP or the accessories are damaged.

1.2 RECHARGING THE BATTERY

Before beginning operation of MicroTIP, the battery will require charging.

Do not remove, replace or charge the battery pack in a hazardou location.

1. Ensure MicroTIP is off by pressing the front of the power switch. See Figure 1.

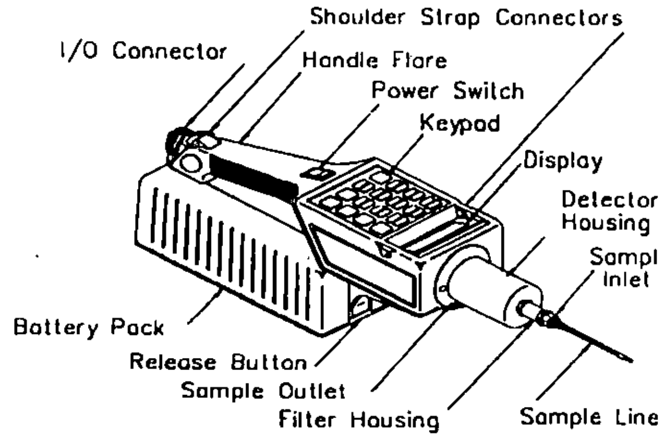


Figure 1 MicroTIP Layout

2. Set the voltage selector switch on the bottom of the battery charger to the appropriate AC line voltage.

3. Press the release button on the bottom of MicroTIP and remove the battery pack by sliding it backwards. See Figure 1.

4. Plug the charger into the battery pack and then into an AC outlet and allow the battery pack to charge for at least 8 hours.

5. After charging remove the charger, first from the wall outlet then from the battery pack, and slide the battery pack back onto MicroTIP.

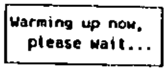
The instrument is now fully charged and ready for use.

Chapter 2 Operation

2.1 OVERVIEW

MicroTIP measures the concentration of airborne ionizable gases vapors and automatically displays and records these concentratio

MicroTIP does not distinguish between individual pollutants. The reading displayed represents the total concentration of all ionizabl chemicals present in the sample.


Warming up now,
please wait...

Turn the instrument on by pressing the back of the power switch. pump will start and the message "Warming up now, please wait" be displayed. Within three minutes the following information will a pear on the display: instrument status, current detected concentra tion, Event number, time, and date. See Figure 2.

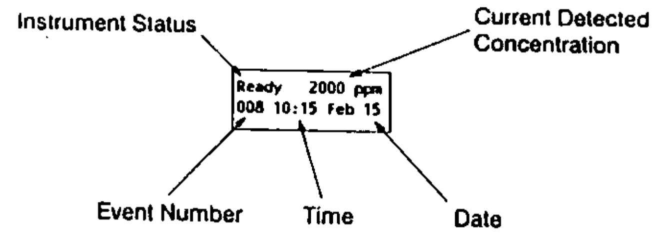


Figure 2 Normal Display

MicroTIP operates automatically. The user reads concentrations directly from MicroTIP's display. The display updates itself each ha second.

The Minimum, Maximum, and Average concentrations measured i each 15-second period are automatically recorded in MicroTIP's datalogging memory. MicroTIP's memory holds the last 12 hours concentration data measured.

Concentration data can be played back from memory on MicroTIP display or sent to a printer or computer in either a tabular or a grap cal format. Data are recorded by date, time, and by a user-entered Event number. Data are played back by the user specifying a start and a stop Event number.

The keypad is used to set up and calibrate MicroTIP, and allows th user to manipulate the concentrations measured and recorded by

MicroTIP in various ways. MicroTIP has 16 clearly labelled keys for direct numeric entry and for using MicroTIP's functions.

All information entered from the keypad and stored in MicroTIP's datalogging memory is retained when MicroTIP is switched off. The clock and calender continue to operate and do not need to be reset the next time MicroTIP is used.

2.2 TUTORIAL SESSION

To assist the user in learning the key functions, MicroTIP has a built-in tutorial session which displays a two-line description of the function of each key. Pressing MicroTIP's TUTOR key begins a tutorial session and pressing the EXIT key twice ends the session. While in the tutorial session keypresses have no effect on MicroTIP's operation.

Press the TUTOR key and begin a tutorial session. Press each key and read the display. The tutorial display for each key is given in Table 1.

BATT	Shows battery V normally 9-16V	ALARM	Shows set-point for conc alarm
DISPLAY	Displays conc as graph or numeral	MAX	Displays highest conc measured
LIGHT	Switches display light hi/lo/off	SETUP	Sets date time & options for keys
AUDIO	Selects alarm or tone or no audio	EVENT	Puts event mark into recorder
PRINT	Prints table of recorded data	TUTOR	Press a key then read explanation
GRAPH	Prints graph of recorded data	CLEAR	Erases the last number pressed
PLAY	Replays recorded data on display	EXIT	Cancels key with no more changes
CAL	Calibrates with zero & span gas	ENTER	Confirms display then continues

Table 1 Tutor Displays

Press the EXIT key twice to end the session.

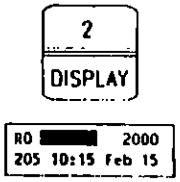
In operation, all MicroTIP's function keys work in the same way.

If there are no options to the function then the key acts immediate

If there are options, then the display will indicate these. The curre selected option is displayed on the lower line. The user is prompt to display the other options by pressing the up arrow or down arr keys. Pressing ENTER confirms that the displayed option is corre the function requires numeric input then the current value is displa on the lower line. The user can change it on the display by pressi the numeric keys. Pressing ENTER confirms that the displayed va is correct.

Some functions have multiple steps for options and/or numeric inputs. These are arranged so that the most frequently changed inputs are displayed first. Once the desired changes have been made the user can bypass the rest of the steps by pressing EXIT.

Each key function is described in more detail in the following sections. Leave MicroTIP on and try each key in turn.

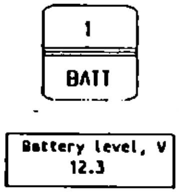


2.3 DISPLAY

If a numerical display is shown, pressing DISPLAY will change it t bar graph. If the bar graph is shown, pressing DISPLAY changes a numerical display. The bar graph range is selected with the SE key.

2.4 LIGHT

Backlighting can be one of two intensities. The brighter lighting consumes more power and is recommended for use only in very locations. Pressing the LIGHT key switches the backlight on to hi intensity, pressing it again decreases the intensity and pressing it once more turns the backlighting off.



2.5 BATT

Pressing the BATT key displays the current battery level. The batt voltage will be shown for 15 seconds and then the display reverts normal. The normal operating voltage range is 9 to 16 volts. Whe LoBat is displayed there is approximately 10 minutes of operation

The battery pack must be replaced by a fully charged pack and the discharged pack should be recharged. See Section 1.2.

BATTERY PACK
CRITICALLY LOW

If operation is continued with a low battery pack another message will appear indicating the batteries are critically low. MicroTIP will now turn off the detector lamp, the pump and the backlighting (if activated). This reduces deep discharging of the battery pack and possible memory loss.

Note: Deep discharging the battery pack will result in loss of recorded data and setup parameters. To avoid loss of data, replace the battery pack with a fully charged one and recharge the old one for at least 8 hours .

2.6 MAX

0
MAX

Press the MAX key. The maximum concentration, the Event during which it was encountered, the time and the date of the occurrence will be displayed. This is shown for 15 seconds and then the display reverts to normal.

```
Max          112 ppm
240 10:12 Feb 15
```

Pressing MAX and then CLEAR will reset the Max register. "Max Cleared" will be displayed with the current date and time. After 15 seconds the display reverts to normal.

```
Max cleared
240 10:12 Feb 15
```

Recording of real time data is not interrupted when the MAX key is pressed or when the Max register is cleared.

2.7 CLEAR

CLEAR

CLEAR erases the last numerical entry. If a number is entered in error press CLEAR to erase the entry and re-enter the correct number. CLEAR used in conjunction with the MAX key resets the Max register.

2.8 EVENT

EVENT
↑

Each press of the EVENT key advances the Event number by one unit on the display. Press EVENT to help identify a particular sample or sampling location in memory.

After Event 255, MicroTIP resets the Event counter to zero. Each time the instrument is turned on the Event number is automatically advanced by one unit. Recorded data are played and printed by specifying a start and stop Event number.

```
eady         500 ppm
55   10:28 Feb 15
```

```
Ready        500 ppm
0    10:29 Feb 15
```

Note: MicroTIP only stores the last Event seen in a 15 second period. If the user wishes to assign a specific Event num a sample, the EVENT key should be pressed only once 15 seconds. If the Event key is pressed more than once 15 second period lower Event numbers will not be store

MicroTIP records continuously for a period of 12 hours. After th time it begins to overwrite existing data one Event at a time.

For example: 6 Events of 2 hours each are recorded. Event # overwrite event #1 if it is 2 hours or less in length. If Event #7 is greater than 2 hours it will overwrite Event #2 as well. If Event # hours, then Events #3, #4, #5, #6 and #7 are now in the recor

If it is necessary to retain a copy of recorded data, the data sho printed or stored in a computer at least once every 12 hours of tion to prevent loss of information when the Events are overwritt

2.9 EXIT

EXIT

The EXIT key terminates all MicroTIP functions except DISPLAY LIGHT, and EVENT. When EXIT is pressed the display reverts t normal. Most functions exit automatically if no key is pressed f seconds.

When EXIT is pressed during printing or graphing, MicroTIP sto sending information to the printer or computer. The printer will tinue to print until its buffer is empty.

2.10 SETUP

×
SETUP
↓

The SETUP key allows MicroTIP to be set up for a specific appli tion. The current date and time are also set with the SETUP key Press SETUP and step through the options. Press ENTER to a the displayed data or enter a numerical value using the keypad then press ENTER. If no values are entered MicroTIP's display to normal.

To set up the instrument:

1. Press SETUP.

2. The first step sets the full scale range for the bar graph d play, the graph output, the audio output , and the 1 volt output. Use the up and down arrow keys to select the 2 or 2000 parts per million (ppm) range.

```
Range 0-? ppm ↓ ↑
200
```

Cal memory ? ↑↓
1

3. Next the Cal memory is selected. MicroTIP has five Cal Memories for regular operation and one for High Sensitivity operation. Only one Cal memory can be used at a time. Select Cal Memory 1 with the up and down arrow keys and press ENTER.

   No matter which Cal Memory is selected, MicroTIP's response is not specific to any one compound. The reading displayed represents the total concentration of all ionizable compounds in the sample.

Hour is ? 0-23
10

Minute is ? 0-59
45

Date is ? 01-31
2

Month is ? 1-12
2

Year is ? 0-99
89

4. Next enter the correct values for the current time. Press ENTER after each value.

5. Enter the numerical values for the day, month and year. Again press ENTER after each selection.

The instrument is now set for operation.

4
AUDIO

2.11 AUDIO

To connect the headset remove the dustcover from the I/O connector and plug in the headset.

Audio output? ↑↓
Off

Audio output? ↑↓
Audio on Alarm

Audio output? ↑↓
Continuous Audio

If a headset is being used with MicroTIP press AUDIO and use the arrow keys to select one of three options for audio output and press ENTER.

The audio output can be turned off altogether. It can be set so there is audio output during an alarm condition only. The last option is a continuous audio signal with the tone being proportional to the detected concentration.

The audio volume is controlled by a knob on the headset.

9
ALARM

2.12 ALARM

The ALARM key displays the current alarm level and allows a new alarm level to be entered.

1. Press ALARM.

Alarm at ? ppm
100.00

2. The current alarm is displayed. If this value is correct wait for the display to revert to normal in 15 seconds or press EXIT.

---

3. If a new value is to be set, enter the value, and press E

When an alarm condition is detected the instrument status ch
to "Alarm" and an audio signal is heard through the headset
"Audio on Alarm" is selected) and remains on until the alarm
tion has passed or until it is turned off with the AUDIO key.

7
PLAY

2.13 PLAY

The PLAY key plays back previously recorded data.

If either "Alarm on Alarm"or "Continous Audio"is selected th
back audio output (not the real time output) is heard through
headset. To enable playback audio output, press AUDIO and
the desired output before pressing the PLAY key.

ENTER to Play
* for Options

1. Press PLAY. Two options are available. Pressing ENT
   begins playback where it was last stopped. Press the
   (*) key to set the playback options.

Start at Event ?
10

2. Select the start Event. If the selected Event is not avail
   MicroTIP begins at the closest higher Event.

   An Event may not be available if the EVENT key was p
   more than once in 15 seconds, or if the selected Even
   been overwritten in the memory by more recent inform

3. Next select which value is to be displayed, either the M
   mum, the Average, or the Maximum, with the arrow ke
   press ENTER.

4. The data can be played back in either numerical or gra
   display by pressing the DISPLAY key.

When MicroTIP is playing back recorded data it is also measu
recording real time concentrations even though the instrumen
is "Play". If, during playback, an instrument status with a prio
higher than that of "Play" is encountered in real time operatio
be displayed, but MicroTIP will continue to play back.

Play 100 ppm
010>10:20 Feb 15

Play 100 ppm
012<10:22 Feb 15

The playback speed and direction can be selected using the a
keys. The speed can be increased or decreased and the info
can be viewed in the opposite direction as well. A forward ar
appears in the display if data are being played forward or a ba
arrow (<) if the data are being played in reverse.

Press ENTER to freeze the display at any time and use the arr
to resume playback. Press EXIT to return to the normal displa

The PLAY function provides a speed search to find the desired start and stop Event numbers for printing or graphing.

8
CAL

2.14 CAL

MicroTIP must be calibrated in order to display concentration in units equivalent to ppm. First a supply of Zero Gas, which contains no ionizable gases or vapors, is used to set MicroTIP's zero point. Then, Span Gas, containing a known concentration of an ionizable gas or vapor, is used to set the sensitivity.

Usually clean outdoor air will be suitable as Zero Gas. If there is any doubt, use a commercial source of Zero Grade Gas and a second sampling bag. A supply of Span Gas of the desired compound and concentration must be obtained for calibration. Observe proper handling techniques for all gases.

Isobutylene at 100 ppm in air is recommended as Span Gas. To calibrate the instrument use the Calibration Kit (Photovac Part No. 390033) as follows:

1. Connect the supplied regulator to the Span Gas cylinder. Hand tighten the fittings.
2. Open the valve on the gas bag by turning the valve stem fully counterclockwise.
3. Attach the gas bag adapter nut to the regulator. Hand tighten the fittings.
4. Turn the regulator knob counterclockwise about half a turn to start the flow of gas.
5. Fill the gas bag about half full and then close the regulator fully clockwise to turn off the flow of gas.
6. Disconnect the bag from the adapter and empty it. Flush the bag a few times with the Span Gas and then fill it.
7. Close the gas bag by turning the valve clockwise.

memory ? ↑↓

8. Press SETUP and select the desired Cal Memory with the arrow keys and press ENTER. Press EXIT to leave Setup.

nnect zero gas
en press ENTER

9. Press CAL and expose MicroTIP to Zero Gas. Press ENTER and MicroTIP sets its zero point.
10. MicroTIP then asks for the Span Gas concentration. Enter the known Span Gas concentration and then connect the Span Gas bag adapter to the inlet.

Span conc ? ppm
100.00

Calibrating now,
please wait...

11. Press ENTER and MicroTIP sets its sensitivity.
12. When MicroTIP's display reverts to normal, MicroTIP is calibrated and ready for use. Remove the Span Gas bag from the inlet.

MicroTIP has 5 Cal Memories and can be calibrated with 5 different span gases if desired. Only one Cal Memory can be used at a time. Each memory stores a different zero point and sensitivity. To program the Cal Memories:

1. Press SETUP and select the desired Cal Memory (1 to 5) with the arrow keys.
2. Exit from Setup and press the CAL key.
3. Follow the displayed calibration instructions. When the calibration is completed it is automatically stored in the selected Cal Memory.

Whenever the instrument is calibrated, MicroTIP updates the selected Cal Memory. The instrument should be calibrated once a day.

No matter which Cal Memory is selected, MicroTIP's response is not specific to any one compound. The reading displayed represents the total concentration of all ionizable compounds in the sample.

5
PRINT

2.15 PRINT

MicroTIP was designed to be used with an Epson FX-80® or 100% compatible printer. If another type of printer is to be used, the appropriate printer cable must be obtained. See Section 3.1 for details on the type of cable required.

The printer must be set to 8 data bits and 1 stop bit to communicate with MicroTIP. MicroTIP's baud rate and parity must be set depending on the printer requirements. Refer to the printer user's manual for more information.

MicroTIP is not UL®-classified for use in hazardous locations with printers.

To print recorded data:

1. Use the printer cable and suitable adapter (Photovac Part No.

395006) to connect the MicroTIP I/O connector to the printer.

```
ENTER to Print
  * for Options
```

2. Press the PRINT key and then the SETUP (*) key to select the desired setup options.

```
Start at Event?
     001
```

```
Stop with Event?
     024
```

3. MicroTIP will ask for the number of the start and stop Events. Enter the desired values and press ENTER.

```
Output format?↑↓
 Fit on one page
```

```
Output format?↑↓
 Output all data
```

4. MicroTIP will then ask if the selected data are to be formatted to fit on a single 8 1/2" x 11" page or if all recorded data between the selected start and stop Events are to be printed. Use the arrow keys to select the desired option and press ENTER.

```
Baud rate ?   ↑↓
   9600
```

```
Parity?       ↑↓
    None
```

5. Enter the baud rate and parity. These values are specific to the printer being used and must be set correctly. Again, refer to the printer user's manual to determine the correct baud rate and parity.

6. When the setup is correct, ensure the printer is on line and press ENTER.

If "Fit on one page" is selected, MicroTIP will divide the selected data into 54 averging intervals. One interval is printed on each line.

MicroTIP always stores one set of readings (Min, Avg and Max) each 15 seconds. In each averaging interval the printed minimum is the minimum of all the stored readings in that interval. The printed Avg is the average of all the recorded average readings and the Max is the maximum of all the recorded maximum readings.

The following information is printed:

a. The number of readings in an interval and the length of the interval are printed at the top of the page. In Figure 3 there are 16 readings in an interval and the interval is 240 seconds long.

b. The interval start time. The first interval in Figure 3 begins at 13:47.

c. The lowest Event number in the interval, only if the Event number has changed.

d. The highest priority instrument status of the interval.

e. Space for the user to add Notes to the report. N
could include identification of particular samples
sampling location based on Event numbers.

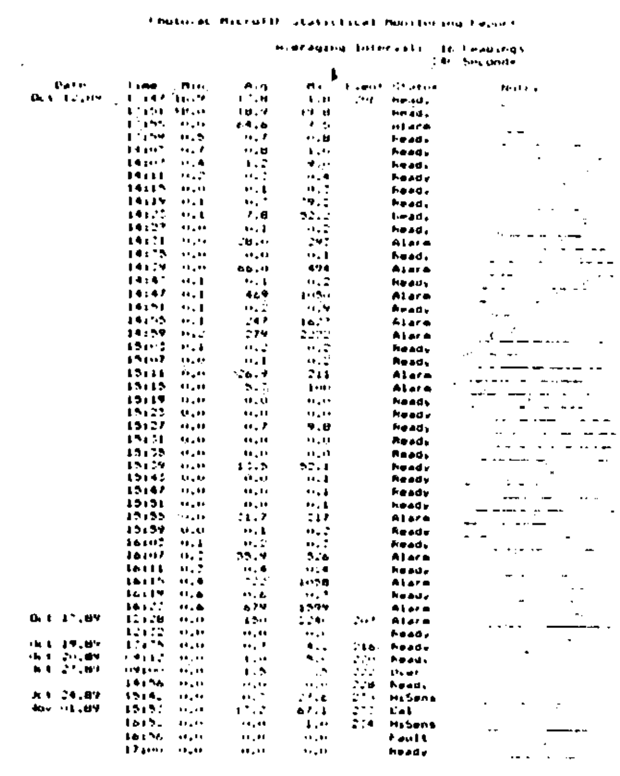


Figure 3 Printed Output

If "Output all data" is selected the header, containing the title, av
ing interval and column headings, will only be printed on the first
of the printed output. Each of the following pages will contain 66
of data.

To print all 12 hours of recorded data will require about 45 pages
take about 10 minutes. The actual printing time will depend on t
printer and print quality.

nting now
please wait...

While the information is being printed, the display shows that printing is in progress. The keypad will not accept commands until the present print job has been completed.

Pressing EXIT during printing stops the job and the display reverts to normal. The printer will continue to print until its buffer is empty.

6
GRAPH

2.16 GRAPH

Pressing the GRAPH key also prints the recorded data but in graphical format. See Figure 4.

MicroTIP is not UL classified for use in hazardous locations with printers.

To graph recorded data:

nge 0-7 ppm ↑↓
0

1. Press SETUP to set the range for the graph. Use the arrow keys to select the 20, 200 or 2000 ppm range.

2. Use the printer cable and suitable adapter (Photovac Part No. 395006) to connect the MicroTIP I/O connector to the printer.

'ER to Graph
* for Options

3. Press the GRAPH key and then the SETUP (*) key to select the desired setup options.

irt at Event?
001

p with Event?
024

4. MicroTIP will ask for the number of the start and stop Events. Enter the desired values and press ENTER.

put format? ↑↓
t on one page

put format? ↑↓
itput all data

5. MicroTIP will then ask if the selected data are to be formatted to fit on a single 8 1/2" x 11" page or if all recorded data between the selected start and stop Events are to be graphed. Use the arrow keys to select the desired option and press ENTER.

d rate ? ↑↓
9600

ity? ↑↓
None

6. Enter the baud rate and parity. These values are specific to the printer being used and must be set correctly. Again, refer to the printer user's manual to determine the correct baud rate and parity.

7. When the setup is correct, ensure the printer is on line and press ENTER.

If "Fit on one page" is selected, MicroTIP will divide the selected data into 600 averging intervals. Sixteen interval are graphed on each line.

MicroTIP always stores one set of readings (Min, Avg and Max) e
15 seconds. In each averaging interval the printed miminum is t
minimum of all the stored readings in that interval. The printed A
the average of all the recorded average readings and the Max is
maximum of all the recorded maximum readings.

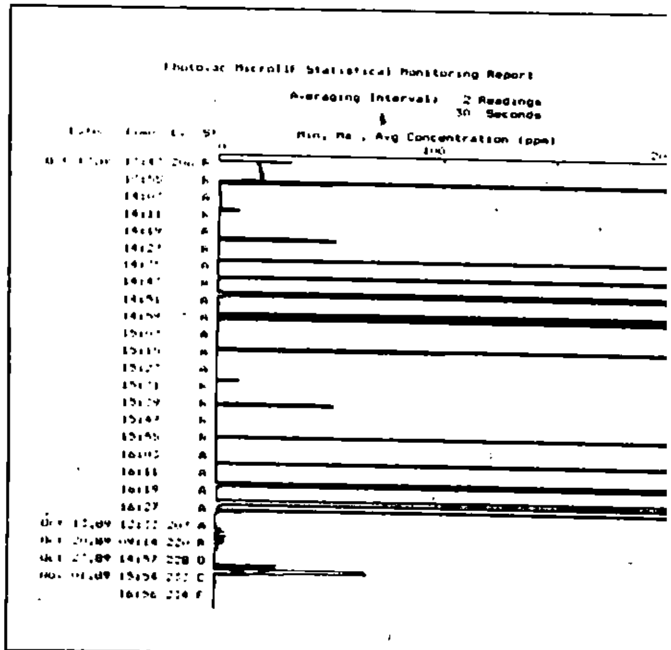


Figure 4 Graphed Output

The following information is printed:

a. The number of readings in an interval and the leng
the interval is printed at the top of the page.

In Figure 4 there are 2 readings in an interval and t
interval is 30 seconds long.

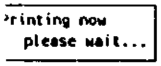
b. Time is printed once every 16 intervals or every 8
minutes for the example in Figure 4. This time will
the start time of the next 16 intervals.

c. The lowest Event number of the 16 Intervals is print
only if it has changed from the previous set of 16
intervals.

d. The highest priority instrument status code of the 16 intervals is printed.

If "Output all data" is selected the header, containing the title, averaging interval and column headings, will only be printed on the first page of the graphed output.

To graph all 12 hours of recorded data will require about 4 pages and take about 10 minutes. The actual printing time will depend on the printer and print quality.


```
rinting now
  please wait...
```

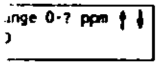
While the information is being printed, the display shows that printing is in progress. The keypad will not accept commands until the present print job has been completed.

Pressing EXIT during printing stops the job and the display reverts to normal. The printer will continue to print until its buffer is empty.

2.17 HIGH SENSITIVITY OPERATION

MicroTIP can be used as a high sensitivity leak detector. In High Sensitivity operation, MicroTIP does not read directly in ppm but displays a reading proportional to the total concentration of ionizable gases and vapors detected.

During calibration, no Span Gas is required. MicroTIP simply zeros its reading with Zero Gas and then sets itself to the maximum sensitivity.


```
nge 0-? ppm ↑↓
)
```

1. Press SETUP. Select the 0-20 ppm display range with the arrow keys and press ENTER.

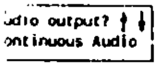

```
al memory ?  ↑↓
igh Sensitivity
```

2. Select High Sensitivity with the arrow keys and press ENTER.


```
  ██████     20
8 11:15 Feb 15
```

3. Press EXIT and then select the bar graph with the DISPLAY key.

4. Press CAL and calibrate MicroTIP with Zero Gas. High Sensitivity operation does not require Span Gas.


```
udio output? ↑↓
ontinuous Audio
```

5. Press AUDIO and select "Continuous Audio" with the arrow keys.

As MicroTIP samples air closer to the leak, the length of the shaded area on the display increases. When used with the optional headset (Photovac Part No. 395004), the frequency of the tone increases as air closer to the leak is sampled, and the user need not watch the display.

MicroTIP's 3 second response time and detection limit of 0.1 ppm isobutylene permit fast detection of small leaks.

2.18 RELATIVE RESPONSES TO GASES AND VAPORS

In situations where only a single pure compound is present in air, MicroTIP should be calibrated with a standard of that specific compound as Span Gas.

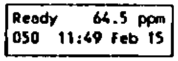
MicroTIP's five Cal Memories can be used to store calibration information for up to five different Span Gases.

MicroTIP's reading will always be influenced by any other ionizable compounds present in the air sample. Even if MicroTIP has been calibrated with a specific compound, its response is not specific and the presence of another impurity may render the numerical result invalid.

It is often impractical to carry a range of different standards into the field. Approximate results can be obtained by calibrating MicroTIP with the recommended Span Gas and then manually adjusting MicroTIP's reading by a factor based on the response of the specific compound to the response of the Span Gas.

Table 2 gives relative response data from which approximations can be made for guidance purposes. Data extrapolated from the use of relative responses must be regarded as interim and approximate only. Table 2 should be used only for concentrations up to 100 ppm of the specific compound, because relative responses change with concentration.

To use the data in Table 2, first calibrate MicroTIP with Zero Gas and 100 ppm Span Gas as described in Section 2.14. Then, note the displayed concentration while exposing MicroTIP to the sample. Look up the specific compound being sampled in Table 2, and divide the displayed concentration by the relative response shown. The resulting ratio is the approximate concentration of the specific compound.


```
Ready      64.5 ppm
050   11:49 Feb 15
```

For example: MicroTIP has been calibrated with 100 ppm isobutylene and is now being used to detect acetone. A reading of 64.5 ppm is being displayed. From Table 2, the relative response of acetone is 0.86. Using the above information the approximate concentration of acetone is calculated as follows:

Approximate Concentration = Reading/Relative Response
= 64.5 ppm/0.86
= 75 ppm

COMPOUND	RELATIVE RESPONSE
Acetone	0.86
Benzene	1.78
Butyl Acetate	0.35
Cyclohexane	0.53
Cyclohexanone	1.11
1,2-Dichlorobenzene	2.25
Ethyl Acrylate	0.30
n-Heptane	0.27
Methyl Ethyl Ketone	1.10
Methyl Isobutyl Ketone	0.87
Methyl Methacrylate	0.67
n-Octane	0.39
Perchloroethylene	1.40
Styrene	2.20
Toluene	1.91
Trichloroethylene	1.61

Table 2 Relative Responses

Note: These relative responses serve as a guide to concentrations measured by MicroTIP with a 10.6 eV lamp. Results are expected to be accurate to within +/-10ppm or +/-25% of result, whichever is greater. Accuracy of relative responses to other gases and vapors may differ from that stated.

These responses are measured relative to isobutylene Span Gas.

Chapter 3 Accessories and Other Devi

3.1 PRINTER

MicroTIP is designed to interface with an Epson FX-80 or 100% compatible printer. The printer cable (Photovac Part No. 39500 has been configured for this application only. If a printer that is 100% compatible with the Epson FX-80 is being used, a suitabl cable must be obtained or the existing one must be modified.

In order to modify the existing printer cable the pinout informati the printer connector is required. Refer to the printer user's ma for specific details. The pinouts for the MicroTIP printer cable ar shown in Figure 5 and are listed in Table 3. If modifying the cab not possible, take the electrical and pinout information to a com puter store a suitable adapter may be obtained.

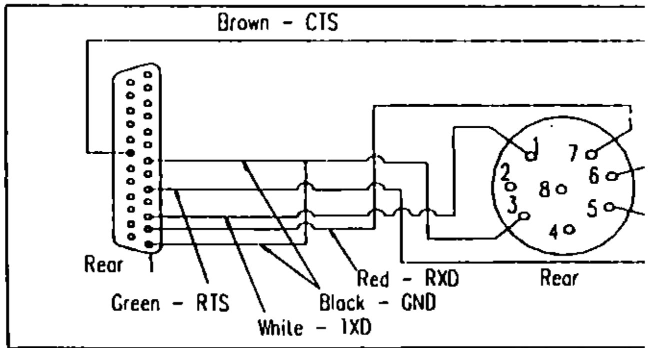


Figure 5 Printer Cable Electrical Connection

Once the proper cable has been obtained, ensure the printer is 8 data bits and 1 stop bit for communication and that the appro baud rate and parity have been selected. Use the PRINT and GRAPH keys as detailed in Sections 2-15 and 2-16.

MicroTIP is not UL-classified for use in hazardous locations wi printers.

3.2 COMPUTER

MicroTIP will send information stored in its datalogger to a com The computer must be set up to emulate a terminal. The use o Crosstalk®, a communications software package, is recomm

MicroTIP I/O Connector (8 pin) Pin #	MicroTIP Printer Cable (DB25) Pin #	Standard RS-232	Color
1	3	TXD	White
2	—	—	—
3	1 & 7	GND	Black
4	—	—	—
5	5	RTS	Green
6	20	CTS	Brown
7	2	RXD	Red
8	—	—	—

Table 3 Printer Cable Pinouts

MicroTIP is set to 8 data bits and 1 stop bit for communication. The appropriate baud rate and parity must be selected. Use the PRINT key as detailed in Section 2-15.

Use the printer cable and a suitable adapter (Photovac Part No. 395006) to connect MicroTIP to the computer's serial port. Refer to the computer user's manual as both the baud rate and parity will depend on the computer.

The baud rate can be set from 300 to 19200. The parity can be set to Odd, Even or None and again will depend on the computer. The port number will be the computer port to which MicroTIP is connected. Stop Bits must be set to 1. Correspondingly, Data Bits must be set to 8.

Set the corresponding baud rate, parity, the number of stop bits, the number of data bits and the computer port number in Crosstalk. See Table 4.

Command	Action
"SP 9600"	Sets the baud rate
"PA NONE"	Sets the parity
"PO <n>"	Sets the port number with <n> being the desired port number.
"ST 1"	Sets the number of stop bits.

Table 4 Computer Setup Commands

Options in Crosstalk, other than those listed in Table 4 will have no effect. Refer to the Crosstalk user's manual for complete instructions on running Crosstalk and issuing commands.

To initiate communications between MicroTIP and the compu

1. Type "GO L". This will initiate local communications
device on the specified port. Ensure that the correct
selected. All ports can be tested by changing the spec
port number with the "PO <n>" command, and then
issuing the "GO L" command.

2. Communications between MicroTIP and the computer
now established. Press the MicroTIP PRINT key to be
sending information to the computer display.

3. To capture (or store) the data on disk, enter the comm
"CA <filename>" where <filename> represents any
for the captured data. Capture will only record all data
ceived from this point on, so data sent to the compute
pressing the MicroTIP PRINT key will now be capture
disk. To complete the capture on disk enter the comm
"CA OFF" and the file will be closed and saved on the

Once the file has been stored, it can then be imported into a L
1-2-3® spreadsheet as a text file. See Lotus 1-2-3 user's man
specific details.

MicroTIP is not UL-classified for use in hazardous locations wi
computers.

3.3 CHART RECORDER

MicroTIP's output can be displayed as a 0 to 1 volt analog vol
a chart recorder in real time. Set the chart recorder to 1V full
and connect it to MicroTIP's I/O connector using the analog
cable (Photovac Part No. 395005). The concentration range
analog output signal is selected with the SETUP key, and can
to 20, 200, or 2000 ppm full scale.

MicroTIP is not UL-classified for use in hazardous locations wi
recorders.

3.5 SAMPLE BAG

MicroTIP is equipped with a sample outlet fitting (See Figure 1
which samples may be collected for further analysis. Connec
sample bag to the fitting with a short length of 1/8" inside dia
flexible tubing.

Note: Readings may fluctuate due to changes in detector flowrate as the sample bag is filling. The bag contents will not perfectly represent the sample. Ozone produced by MicroTIP's detector will be present, and sample composition may have been altered by passage through MicroTIP's sampling pump.

3.5 THREE METER (9.8') SAMPLE LINE

For remote sampling, connect the 3m (9.8') sample line (Photovac Part No. 390006) to MicroTIP's sample inlet in place of the 17cm (6.8") sample line supplied.

3.6 SHOULDER STRAP

Snap one end of the shoulder strap to the steel shoulder strap connector bail above MicroTIP's I/O connector. Snap the other end to one of the shoulder strap connectors beside the display. The connection point is selectable for right or left handed operation. Adjust the shoulder pad and strap length for comfort.

3.7 HEADSET

Connection and operation of the headset (Photovac Part No. 395004) is described in Section 2.11.

3.8 REPLACEMENT DETECTOR LAMPS

MicroTIP is supplied with an ultraviolet (UV) lamp which produces an energy of 10.6 electron-volts (eV). With this standard lamp installed, MicroTIP responds well to gases and vapors which ionize at 10.6 eV or less. Some of these are listed, with their relative responses, in Table 2. For special applications, MicroTIP's response can be changed by using other lamps.

With a 9.5 eV lamp, MicroTIP responds well only to gases and vapors which ionize at 9.5 eV or less. Some relative responses with a 9.5 eV lamp are listed in Table 5. To use the 9.5 eV lamp, MicroTIP must be calibrated with a compound which ionizes at below 9.5 eV. Toluene at 100 ppm in air is recommended.

Table 5 gives relative response data from which approximations can be made for guidance purposes. Data extrapolated from the use of relative responses must be regarded as interim and approximate only. Table 5 should be used only for concentrations up to 100 ppm of the specific compound, because relative responses change with concentration.

Data from Table 5 may be used in the same way as those from Table 2 if a single pure compound in air is being sampled. First calibrate MicroTIP with Zero Gas and 100 ppm Span Gas as described in Section 2.14. Use toluene in air as Span Gas. Then, note the displayed concentration while exposing MicroTIP to the sample. Look up the specific compound being sampled in Table 5 and divide the displayed concentration by the relative response shown. The resulting ratio is the approximate concentration of the specific compound.

COMPOUND	RELATIVE RESPONSE
Acetone	0.10
Benzene	0.81
Butyl Acetate	0.09
Cyclohexane	0.10
Cyclohexanone	0.55
1,2-Dichlorobenzene	0.83
Ethyl Acrylate	0.08
n-Heptane	0.07
Methyl Ethyl Ketone	0.33
Methyl Isobutyl Ketone	0.40
Methyl Methacrylate	0.12
n-Octane	0.10
Perchloroethylene	0.60
Styrene	1.22
Toluene	1.00
Trichloroethylene	0.26

Table 5 Relative Responses with 9.5 eV Lamp

Note: These relative responses serve as a guide to concentrations measured by MicroTIP with a 9.5 eV lamp. Results for compounds with relative responses of 0.30 and above are expected to be accurate to within +/-10ppm or +/-25% of result, whichever is greater. Results for compounds with relative responses below 0.30 are expected to be accurate to within +/- 40% of result. Accuracy of relative responses to other gases and vapors may differ from that stated.

These responses are measured relative to toluene Span Gas.

In a situation where a range of gases and vapors exists, MicroTIP displays a reading representing the total concentration of ionizable chemicals present. The change in relative response given by the 9.5 eV lamp is especially useful when sampling mixtures, for example, a mixture of benzene and n-octane. Both chemicals are detected with MicroTIP, and benzene is the more toxic of the two. For health protection, a relatively higher response to benzene and a lower response to n-octane are desired in order to detect lower concentrations of benzene in the presence of n-octane.

With a 10.6 eV lamp, the relative response of benzene is 1.78 and that of n-octane is 0.39 (from Table 2). MicroTIP's response to benzene is therefore 4.6 times higher than its response to n-octane. With a 9.5 eV lamp, the response to benzene is (from Table 5) 8.1 times higher than the response to n-octane.

$$\begin{aligned}\text{9.5 eV response ratio} &= \text{benzene rel. resp./n-octane rel. resp}\\ &= 0.81/0.10\\ &= 8.1\end{aligned}$$

Because of these differences in relative response, benzene vapor from the mixture contributes more to the MicroTIP reading with the 9.5 eV lamp than it does with the 10.6 eV lamp. MicroTIP with the 9.5 eV lamp is therefore better suited to detecting benzene in the presence of n-octane.

The 9.5 eV lamp is installed, maintained, and removed according to the instructions in Section 4.2.

With an 11.7 eV lamp installed, MicroTIP functions as a leak detector responding to gases and vapors which ionize at 11.7 eV or less. This lamp is intended for use in High Sensitivity operation only. It is not suitable for direct-reading use, because of limitations of the lamp window material.

The 11.7 eV lamp window material is Lithium Fluoride (LiF). Unlike other lamp windows, LiF readily absorbs water from atmospheric humidity. When contaminated by moisture, the window loses its ability to transmit UV light. Never touch the window or let liquid water near it.

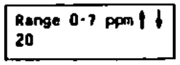
Furthermore, LiF is composed of two light elements which are easily disrupted within the crystal lattice by the same UV light generated by the lamp. Disruption of the lattice causes the crystal to turn a yellowish color, and again performance declines.

The 11.7 eV lamp is useful for detecting leaks of chemicals not ionized by the 10.6 eV lamp, for example, carbon tetrachloride. Set MicroTIP to display a bar graph. The length of the shaded area on the display represents the concentration of carbon tetrachloride (and other ionizables) in the sample. As MicroTIP samples air closer to the carbon tetrachloride source, the display indicates higher concentrations. Numerical values shown on MicroTIP's disp should not be interpreted as parts per million concentrations. They indicate only the relative presence or absence of ionizable gases a vapors.

MicroTIP's 3 second response time and detection limit of 10 ppm carbon tetrachloride permit fast determination of small leaks.

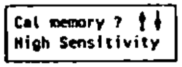
Because of the lamp window limitations, the lifetime of the 11.7 eV lamp is restricted and it must be used sparingly according to these instructions:

1. Remove the 11.7 eV lamp from the supplied dessicant bottl and install the lamp according to Section 4.2.
2. Switch on MicroTIP, wait for it to warm up, and press SETU



Range 0-? ppm ↑ ↓
20

3. Select the 0-20 ppm range with the arrow keys and press ENTER.



Cal memory ? ↑ ↓
High Sensitivity

4. Select High Sensitivity with the arrow keys and press ENTE

HD ████ 410
058 12:15 Feb 15

5. Press EXIT and then select the bar graph with the DISPLA key.

Connect zero gas
then press ENTER

6. Press CAL and calibrate MicroTIP with Zero Gas. High Sen tivity operation does not require Span Gas.
7. Every 15 minutes of operation, recalibrate MicroTIP with Ze Gas.
8. Every hour of operation, switch off MicroTIP and examine t lamp window for yellowing. If the window is yellow, then remove the lamp and regenerate the window according to procedure below.
9. Clean the lamp window with dry cerium oxide powder on a cotton swab. Do not use methanol or water.
10. Heat the lamp in a 150°C (300°F) oven for 8 hours or more.
11. Allow the lamp to cool before use.
12. Alternatively, the lamp window can be regenerated without heat by storing the lamp in a dessicator for at least 5 days.
13. After use, remove the lamp from MicroTIP and store it in th supplied dessicant bottle.

Chapter 4 Routine Maintenance

4.1 CHARGING THE BATTERY

```
  100 ppm
20  Feb 15
```

When the instrument status reads LoBat, the MicroTIP battery pack requires recharging. A fully charged battery powers MicroTIP for 6 hours. If the instrument is to be used for more than 6 hours, carry a spare battery pack. When the first one has been discharged, replace it with the spare. Upon return from field work, recharge both battery packs as outlined in Section 1.2. Two chargers are required to do this overnight.

The charger automatically charges at a high charge rate until the battery is fully charged and then maintains the full charge with a low continuous charge rate indefinitely so there is no danger of over-charging.

Note: Leaving MicroTIP for more than three days, without a charged battery pack will result in loss of recorded data and setup parameters. To avoid loss of data , charge the battery pack for at least 8 hours and replace it

Do not remove, replace, or charge the battery pack in a hazardous location.

4.2 CLEANING THE LAMP WINDOW

During the course of normal operation a film builds up on the window of the detector lamp. The rate at which the film develops depends on the type and concentration of the gases and vapors being sampled and results from the UV light interacting with them. As a guide, clean the window every 24 hours of operation. To clean the lamp window:

1. Ensure the instrument is turned off.
2. Hold the black detector housing in one hand and unscrew it from the body of MicroTIP. See Figure 1. Remove the housing, being careful not to lose the o-ring seal on top of the photoionization detector. The detector cell, lamp holder, and high frequency (HF) driver circuit board are now exposed.
3. Unplug the red and yellow wires from the HF driver circuit board.
4. Hold the lamp holder in one hand so it will not rotate and carefully unscrew the detector cell with the red and yellow wires attached. Do not touch the fine wire mesh inside the detector cell. Any dust or dirt in the detector cell can be blown out with a gentle jet of compressed air.

5. Leaving the lamp spring in place, remove the lamp from the lamp holder.
6. To remove the film, gently rub the window of the lamp with a lint free tissue moistened with methanol.
7. Allow the window to dry and then, without touching the window, replace the lamp in the lamp holder.
8. Replace the detector cell squarely on the lamp holder and ensure the o-ring seal is in position. Finger tighten only.
9. Plug the yellow wire onto the gold pin and the red wire onto the silver pin on the HF driver circuit board.
10. Check the lamp holder and ensure it is securely seated by hand.
11. Replace the detector housing and tighten by hand.
12. Once calibrated, MicroTIP is ready for operation.

4.3 REPLACING THE DETECTOR UV LAMP

```
Fault       0.0 ppm
079 14:50   Feb 15
```

```
Detector light
 intensity is low
```

If the lamp will not light then it requires replacement:

1. Remove the lamp as outlined in Section 4.2.
2. Remove the lamp spring from the lamp holder and replace it with the new lamp spring.
3. Without touching the window of the new lamp carefully place in the lamp holder.
4. Replace the detector cell and the detector housing as outline in Section 4.2.
5. Once calibrated, MicroTIP is ready for operation.

4.4 REPLACING THE FILTER CARTRIDGE

MicroTIP is equipped with a dust filter to reduce detector contamination. As the filter collects dust, MicroTIP's inlet flow rate and sensitivity decrease. Replace the filter every 240 hours of operation, or more frequently if MicroTIP is used in a dusty environment. To replace the filter:

1. Turn the instrument off.
2. Hold the filter housing near the detector housing with a 9/16" wrench.
3. Unscrew the top of the filter housing with another 9/16" wrench. Be careful not to lose the metal sealing washer.
4. Remove the spring and filter and install the new filter, open end first.
5. Replace the filter spring and the filter housing. Tighten the top nut while holding the bottom one stationary with the wrench.
6. Once calibrated, MicroTIP is ready for operation.

4.5 REPLACING THE PUMP

Replace the sample pump every 5000 hours of operation.

1. Turn the instrument off and remove the battery pack so the instrument is easy to manipulate.
2. Turn MicroTIP upside down and remove the 4 screws securing the handle to the control housing. Then remove the two screws in the handle flare.
3. Carefully lift the handle away from the control housing. The two parts remain connected by the set of wires from the I/O connector. Locate the red sample pump mounted on the power supply board. See Figure 6.
4. Disconnect the 2-pin pump connector from the power supply board.
5. Remove the two screws securing the pump bracket to the circuit board. Gently pull the pump out about 1/2" (1 cm) and remove the two pieces of clear tubing from the pump.
6. Connect the clear tubing to the new pump.
7. Slide the pump into the proper position and secure it with two screws.

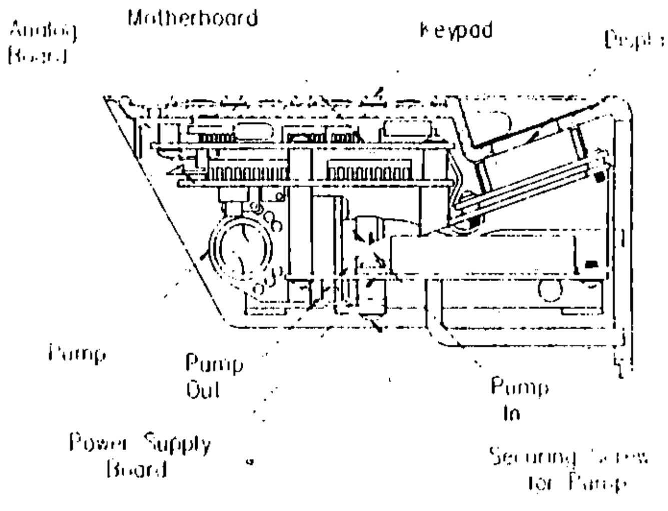


Figure 6 Control Housing Showing Pump Placement

8. Re-connect the 2-pin connector from the pump to the powe supply board.
9. Carefully position the handle over the control housing and replace the six screws from the bottom of the handle and th handle flare.
10. Snap the battery pack into place.
11. Once calibrated, MicroTIP is ready for operation.

Chapter 5 Troubleshooting

5.1 IF MICROTIP DRAWS IN LIQUID

MicroTIP accepts only gas and vapor samples. Aspirating a liquid may result in damage to the lamp and the pump. If water is drawn in, the affected parts of the instrument may be cleaned and dried.

Contact Photovac Service if another liquid is aspirated.

MicroTIP must be serviced away from hazardous locations.

1. Before taking the instrument apart allow the MicroTIP to run until no more liquid comes out of the sample outlet fitting below the detector housing. This will clean out the pump.
2. Turn the instrument off. Remove the detector cell and lamp as outlined in Section 4.2.
3. Dry the lamp with a clean lint free tissue and clean the window. See Section 4.2.
4. Clean the detector cell in distilled water, preferably in an ultrasonic cleaner.

Note: DO NOT touch the fine wire mesh in the detector cell and DO NOT use solvents as they will degrade the detector cell.

5. Dry the detector cell overnight at 50°C (125°F).
6. Dry the inside of the lamp holder.
7. Remove the filter cartridge as in Section 4:4.
8. Dry the inside of the filter housing.
9. Install a new filter cartridge and re-assemble the filter housing.
10. Once calibrated, MicroTIP is ready for operation.

5.2 INSTRUMENT STATUS AND FAULT DISPLAYS

The instrument status appears at the left of the upper line of the display and on the Print and Graph outputs. Each status has a priority assigned to it. If more than one status is in effect, then the

status with the highest priority is displayed until the condition is corrected or until the option is turned off.

When the bar graph display or the Graph output is selected, the instrument status is reduced to a one-letter code. The following table summarizes the instrument status:

Status	Code	Priority	Description
Fault	F	1	One of 3 faults is occurring. Press TUTOR for details.
Over	O	2	Detected concentration exceeds 9999 on the display.
Alarm	A	3	Detected concentration exceeds the set alarm level.
Cal	C	4	Will never be observed on the display during normal operation as various calibration prompt messages are displayed while MicroTIP is calibrating. If the instrument is turned off when it is calibrating, Cal will appear on the display when MicroTIP is turned on again indicating the last calibration was incomplete. Cal status is also shown on printed or graphed output.
LoBat	L	5	Battery pack power is low. Recharge or replace pack.
Play	P	6	The instrument is playing back previously recorded data.
HiSens	H	7	High Sensitivity operation.
Ready	R	8	Normal operation.

Table 6 Instrument Status

When the Fault status is displayed, MicroTIP's operation is compromised. Press the TUTOR key for a two-line description of the fault. Refer to Table 7 for corrective action.

MicroTIP must be serviced away from hazardous locations.

Fault Description	Probable Causes	Corrective Action
Detector light intensity is low	Defective detector UV lamp.	Replace the lamp See Section 4.3.
	Poor connection between lamp holder and HF driver circuit board.	Check the wire joining lamp holder to HF driver circuit board. See Section 4.2.
Signal from zero gas is too high	Contamination of sample line or fittings before detector.	Clean or replace line. Replace inlet filter. See Section 4.4.
	Span and Zero gases mixed up.	Ensure clean gas is used to zero MicroTIP.
	Contamination in detector.	Clean detector. See Section 5.1.
Detector field voltage is low	Contamination in detector.	Clean detector. See Section 5.1.
	Short circuit in detector.	Remove detector and ensure resistance between red and yellow wires exceeds 10 megohms.
	Internal fault in electronics.	Contact Photovac Service.

Figure 7 Fault Analysis

5.3 ESTABLISHING COMPUTER COMMUNICATIONS

If, after having followed the procedure in Section 3.2, communications cannot be established with a computer, the problem may lie with the hardware connections or the printer cable configuration.

1. Ensure MicroTIP is connected to the serial port of the computer.

   The serial port will usually be a male connector, typically 9 pins on an IBM-AT® and compatibles and 25 pins on an

Chapter 6 Technical Description

6.1 OVERVIEW

MicroTIP is a microprocessor controlled instrument for measuring the presence of ionizable chemicals in air at parts per million levels. The block diagram in Figure 7 shows the main components of MicroTIP. The microprocessor controls the components of the instrument and interprets and records the signal generated by the photoionization detector (PID). Recorded data and setup information entered into the microprocessor's memory are retained when MicroTIP is turned off.

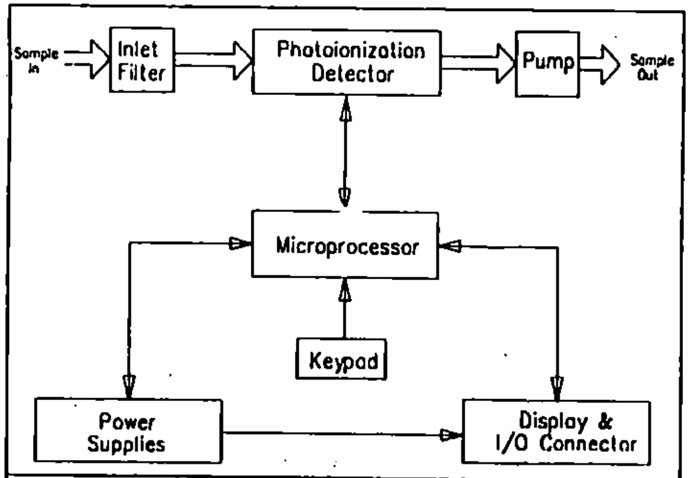


Figure 7. Block Diagram

A pump continuously pulls the air under test through MicroTIP's PID. The PID converts the concentration of ionizable chemicals in a sample into an electrical signal. The microprocessor subtracts any background from the signal and divides this signal by a sensitivity previously obtained by calibrating with a standard gas of known concentration. This concentration appears on MicroTIP's display and, depending on the values entered through MicroTIP's keypad, an alarm message may be displayed or an audio signal may be heard.

MicroTIP can detect thousands of different types of airborne gases and vapors and its response depends on the type as well as the concentration. MicroTIP does not distinguish one type of chemical from another, but displays a number indicating the total concentration of all ionizable compounds in the sample.

A standard of isobutylene at a known concentration may be used for setting the sensitivity. If MicroTIP is calibrated with isobutylene, it displays concentrations in units equivalent to ppm of isobutylene. If isobutylene were the only ionizable chemical in the sample, then MicroTIP would display its concentration directly.

MicroTIP responds more or less readily to other chemicals than it does to isobutylene. Because it has a medium sensitivity to isobutylene, this gas has been chosen as a reliable means of reporting an average concentration of total ionizables present.

For special applications, gases other than isobutylene can be used to calibrate MicroTIP.

6.2 PHOTOIONIZATION DETECTOR

MicroTIP's PID is shown in Figure 8. The PID measures the concentration of ionizable chemicals in the gas stream from the sample inlet and produces an electrical signal for the microprocessor.

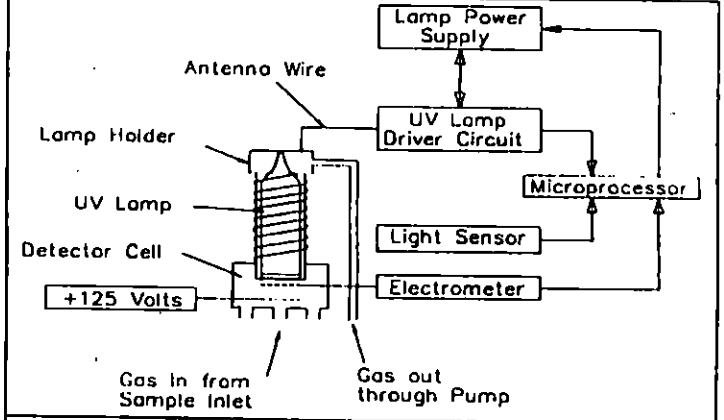


Figure 8 Photoionization Detector

A UV lamp generates photons which ionize specific molecules in the gas stream. The permanent air gases (argon, carbon dioxide, nitrogen, oxygen, water vapor etc.) require a relatively high energy for ionization, and are not ionized by the UV photons. Many of the chemicals considered pollutants, including most hydrocarbons, are ionized.

The gas stream is directed into the PID through a small port at the center of the lamp window and through a series of larger ports around the perimeter of the lamp window. This bypass arrangement permits a high sample flowrate and short response time, while minimizing contamination of the lamp window.

The ionized molecules in the detector cell are subjected to a continuous electric field between the repeller electrode and the collector electrode. The ions move in the electric field, generating a current which is proportional to the concentration of the ionized molecules in the detector cell. An electrometer circuit converts the current to a voltage which is then fed to the microprocessor.

The detector lamp is operated by an HF lamp driver circuit which delivers high frequency energy to the lamp through an antenna wrapped around the lamp holder. The lamp driver power supply is controlled by the microprocessor based on a feedback signal from a light sensor on the HF driver circuit board.

6.3 CALIBRATION AND RECORDING

Periodic calibration is required to compensate for PID output changes due to inlet filter restriction, lamp window cleanliness, sample pump wear and other factors.

During calibration, MicroTIP's PID is first exposed to Zero Gas. A small signal is generated, and this zero signal is stored by the microprocessor.

In High Sensitivity operation, the microprocessor subtracts the zero signal from the PID signal, and multiplies the difference by 1000. This number is then displayed.

When one of the 5 Cal Memories is selected, MicroTIP's PID is next exposed to Span Gas. This span signal is stored. The microprocessor subtracts the zero signal from the span signal and divides the difference by the user-entered Span Gas concentration. The resulting sensitivity is stored in the selected Cal Memory with the zero signal. In operation, the microprocessor first subtracts the zero signal from the PID signal, then divides the difference by the sensitivity. This number is then displayed.

The microprocessor accumulates all readings over a 15 second interval and determines the minimum, average and maximum readings. It stores these numbers along with the highest priority instrument status and the most recent time, date and Event

number which occurred during the 15 second interval. MicroTIP automatically records these results for 12 hours of operation.

These recorded data can now be played back on MicroTIP's display. The display is identical to the numeric or bar graph display, but the instrument status is "Play" indicating that recorded data, not real-time data, are being displayed. During playback MicroTIP continues to analyze and record new data.

Recorded data can also be printed as either a table or a graph. Data may be automatically averaged to fit on one 8 1/2" x 11" page or all recorded, data may be printed or graphed. The averaging interval and number of readings averaged are shown at the top of the page.

Note: For each averaging interval, MicroTIP prints the minimum of all the minima, the average of all the averages and the maximum of all the maxima.

Inlet flowrate:	Exceeds 500 mL/min
Operating temperature range:	0 to 40°C (32 to 105 °F)
Operating humidity range:	0 to 100% Relative Humidity (non-condensing
Operating concentration range:	0.1 to 2000 ppm isobutylene equivalent
Accuracy:	Isobutylene: (after calibration with Zero Gas an 100 ppm isobutylene Span Gas) within +/-2 ppm or +/-10% for 0 to 100 ppm within +/-15% for 100 to 1000 ppm within +/-20% for 1000 to 2000 ppm
	Acetone: (after calibration with Zero Gas and 100 ppm acetone Span Gas) within +/-4 ppm or +/-10% for 0 to 100 ppm within +/-15% for 100 to 1000 ppm within +/-20% for 1000 to 2000 ppm
	Benzene: (after calibration with Zero Gas and 100 ppm benzene Span Gas) within +/-4 ppm or +/-10% for 0 to 100 ppm within +/-20% for 100 to 1000 ppm within +/-25% for 1000 to 2000 ppm
	Toluene: (after calibration with Zero Gas and 100 ppm toluene Span Gas) within +/-2 ppm or +/-10% for 0 to 100 ppm within +/-20% for 100 to 1000 ppm within +/-25% for 1000 to 2000 ppm
	Trichloroethylene: (after calibration with Zero Gas and 100 ppm trichloroethylene Span Gas) within +/-5ppm or +/-10% for 0 to 100 ppm within +/-25% for 100 to 1000 ppm within +/-35% for 1000 to 2000 ppm
Precision:	+/-1% (100 ppm isobutylene)
Response time:	Less than 3 seconds
Detection limit:	0.1 ppm isobutylene

Chapter 8 Warranty

MicroTIP is warranted for one year from defects in materials and workmanship.

Photovac Incorporated warrants that its manufactured products (except Detector UV Lamps which carry specific warranties) will be free from defects in materials and workmanship for a period of one (1) year from the date of receipt by the Customer. This Warranty applies to proper use of the equipment by the customer and may be voided if, in the opinion of Photovac Incorporated, the product has been abused or treated in a negligent manner so as to cause damage or failure. Negligent use includes, but is not limited to, exposure of the internal parts of the equipment to water. Damage caused thereby is expressly excluded from this Warranty.

When Photovac is made aware of a problem in MicroTIP which would be eligible for remedy under Warranty, it will issue a Return Authorization Number to the Customer. No return will be accepted unless such authorization has been obtained.

If upon receipt of the equipment Photovac determines that repairs should be done under Warranty, Photovac's sole liability shall be for labor and materials necessary to put the equipment into proper order and return this to the Customer as promptly as possible. Photovac is in no way responsible for any inconvenience or loss, consequential or incidental, caused to the Customer as a result of the equipment being out of commission.

The Customer is responsible for shipping and insurance to the designated Photovac Service/Repair facility.

In USA

Photovac International
Incorporated
~~741 Park Avenue~~
25-B Jefryn Blvd west
~~Huntington~~, New York Deer Park
~~11743~~ 11729
(516)351-~~6994~~ 254-4199
[illegible]
9:00 AM

In Canada

Photovac Incorporated
105 Doncaster Avenue
Thornhill, Ontario
L3T 1L6
(416)881-8225

Outside USA and Canada: Contact the Photovac representative in your area.

Note: MicroTIP HL-200 does not carry an Intrinsic Safety Rating and must not be used in a Hazardous Location where flammable concentrations of gases or vapors are constantly present.

Index

Your opinions are important to us. We want to design documentatio[illegible]eet your needs. Please let us know what you think by completing and returning the following information.

Please rate the MicroTIP documentation on a scale of 1 to 5.

Poor	1	2	3	4	5	Excellent
Incomplete	1	2	3	4	5	Complete
Unusable	1	2	3	4	5	Usable
Unattractive	1	2	3	4	5	Attractive

How useful did you find the following parts of the manual (1 being not useful and 5 being very useful)?

Table of Contents	1	2	3	4	5
Operating Instructions	1	2	3	4	5
Troubleshooting	1	2	3	4	5
Maintenance Instructions	1	2	3	4	5
Technical Description	1	2	3	4	5
Figures and Tables	1	2	3	4	5
Index	1	2	3	4	5

Did the manual contain all the information you needed? Yes No
If not, what information did you need but could not find?

Did you find any factual errors? Yes No
If yes, please give page number and description of the error.

Which accessories are you using with MicroTIP?

_____ Calibration Kit
_____ 3 meter Sample Probe
_____ Specialty Lamp (_____eV)
_____ Field Kit
_____ Headset/Headphones
_____ Printer

Other suggestions or comments:

The following information is optional, but will allow us to address, directly, any problems you may have.

Name ______________________
Position ______________________
Specific Application ______________________
Company Name ______________________
Address ______________________
Telephone # ______________________

PHOTOVAC
incorporated

Instruction

MI
611-132
December 1985

Model OVA 128
CENTURY Organic Vapor Analyzer

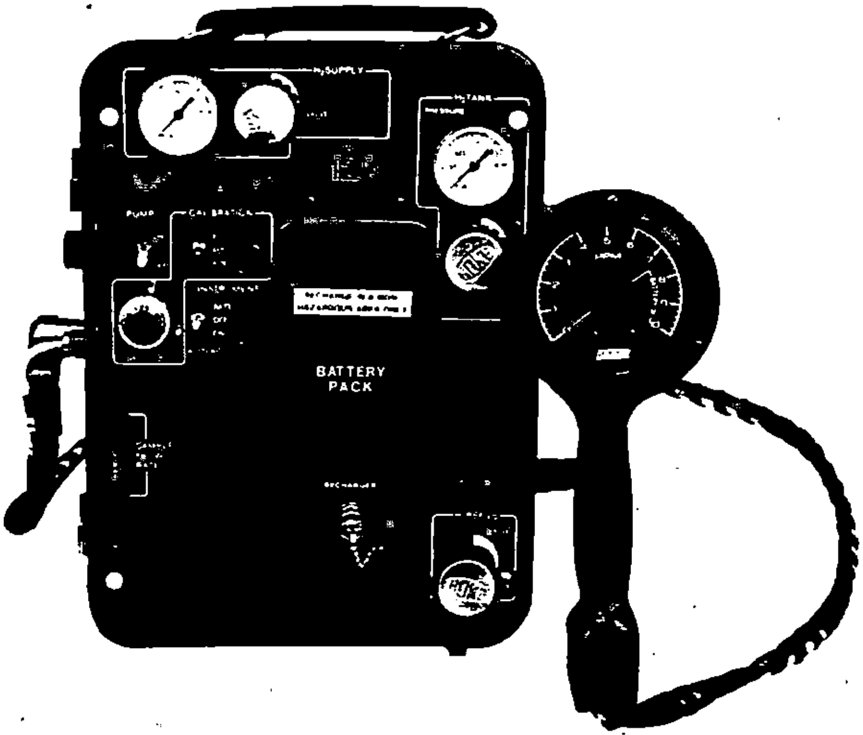


FIGURE 1
PORTABLE ORGANIC VAPOR ANALYZER

FOXBORO®

® Registered Trademark

INTRODUCTION

The Model OVA 128 CENTURY Organic Vapor Analyzer (OVA 128) is manufactured in three configurations. These are:

- Basic Flame Ionization Detector for monitoring total hydrocarbons
- Gas chromatograph supplied with two columns
- Gas chromatograph tri-column for Benzene Analysis.

A battery charger can be ordered for either 120 V ac, 60 Hz or for 220 V ac, 50 Hz. Classifications are:

- FM certified for use in Class I, Groups A, B, C, and D, Division 1 hazardous environments.
- BASEEFA certified intrinsically safe, Ex ib, for IIC, Zone 1, Temperature Class T6. BASEEFA No. 76002/B std. SFA 3007.

Accessories for the OVA 128 are:

- Strip Chart Recorder - either FM or BASEEFA certified.
- Activated Charcoal Filter Assembly - used for zeroing the analyzer in a contaminated environment. Also used with dessicant as a moisture trap.
- Sample Dilutor Assembly for 10:1, 25:1, or 50:1 sample dilution.
- Septum Adapter for direct, online injection with the GC.
- Portable Isothermal Pack (PIP) for temperature control of GC columns.

The OVA 128 is a sensitive instrument designed to measure trace quantities of organic materials in air. It is essentially a flame ionization detector such as utilized in laboratory gas chromatographs and has similar analytical capabilities. The flame ionization detector is an almost universal detector for organic compounds with the sensitivity to measure in the parts per million range (V/V) in the presence of atmospheric moisture, nitrogen oxides, carbon monoxide, and carbon dioxide.

The instrument has broad application since it has a chemically resistant air sampling system and can be readily calibrated to measure almost all organic vapors. It has a single linearly scaled readout from 0 ppm to 10 ppm with a X1, X10, and X100 range switch. This range expansion feature provides accurate readings across a wide concentration range with either 10, 100 or 1000 ppm full scale deflection. Designed for use as a portable survey instrument, it can also be readily adapted to fixed remote monitoring or mobile installations. It is ideal for the determination of many organic air pollutants and for monitoring the air in potentially contaminated areas.

The OVA 128 is certified by Factory Mutual Research Corporation (FM) for use in Class I, Groups A, B, C, & D, Division I hazardous locations. Similar foreign certifications have been obtained, including BASEEFA. This requirement is especially significant in industries where volatile flammable petroleum or chemical products are manufactured or used and for instruments which are used in portable surveying or for analyzing concentrations of gases and vapors. Such instruments must be incapable, under normal or abnormal conditions, of causing ignition of hazardous mixtures in the air. In order to maintain the certified safety, it is important that the precautions outlined in this manual be practiced and that no modifications be made to these instruments.

It is highly recommended that the entire manual be read before operating the instrument. It is essential that all portions relating to safety of operation and maintenance be thoroughly understood.

Reference Literature

MI 611-101	Operation of Tri-Column GC Option
MI 611-102	Operation of Dilutor Kit
MI 611-105	Operation of Portable Isothermal Pack

GENERAL DESCRIPTION

The OVA 128 Analyzer is designed to detect and measure hazardous organic vapors and gases found in most industries. It has broad application since it has a chemically resistant sampling system and can be calibrated to almost all organic vapors. It can provide accurate indication of gas concentration in one of three ranges: 0 to 10 ppm; 0 to 100 ppm; or 0 to 1000 ppm. While designed as a lightweight portable instrument, it can be permanently installed to monitor a fixed point.

The instrument utilizes the principle of hydrogen flame ionization for detection and measurement of organic vapors. The instrument measures organic vapor concentration by producing a response to an unknown sample, which can be related to a gas of known composition to which the instrument has previously been calibrated. During normal survey mode operation, a continuous sample is drawn into the probe and transmitted to the detector chamber by an internal pumping system.

The sample stream is metered and passed through particle filters before reaching the detector chamber. Inside the detector chamber, the sample is exposed to a hydrogen flame which ionizes the organic vapors. When most organic vapors burn, they leave positively charged carbon-containing ions. An electric field drives the ions to a collecting electrode. As the positive ions are collected, a current corresponding to the collection rate is generated. This current is measured with a linear electrometer preamplifier which has an output signal proportional to the ionization current. A signal conditioning amplifier is used to amplify the signal from the preamp and to condition it for subsequent meter or external recorder display. The display is an integral part of the Probe/Readout Assembly and has 270° scale deflection.

In general, the hydrogen flame ionization detector is more sensitive for hydrocarbons than any other class of organic compounds. The response of the OVA varies from compound to compound, but gives repeatable results with all types of hydrocarbons; i.e., saturated hydrocarbons (alkanes), unsaturated hydrocarbons (alkenes and alkynes) and aromatic hydrocarbons.

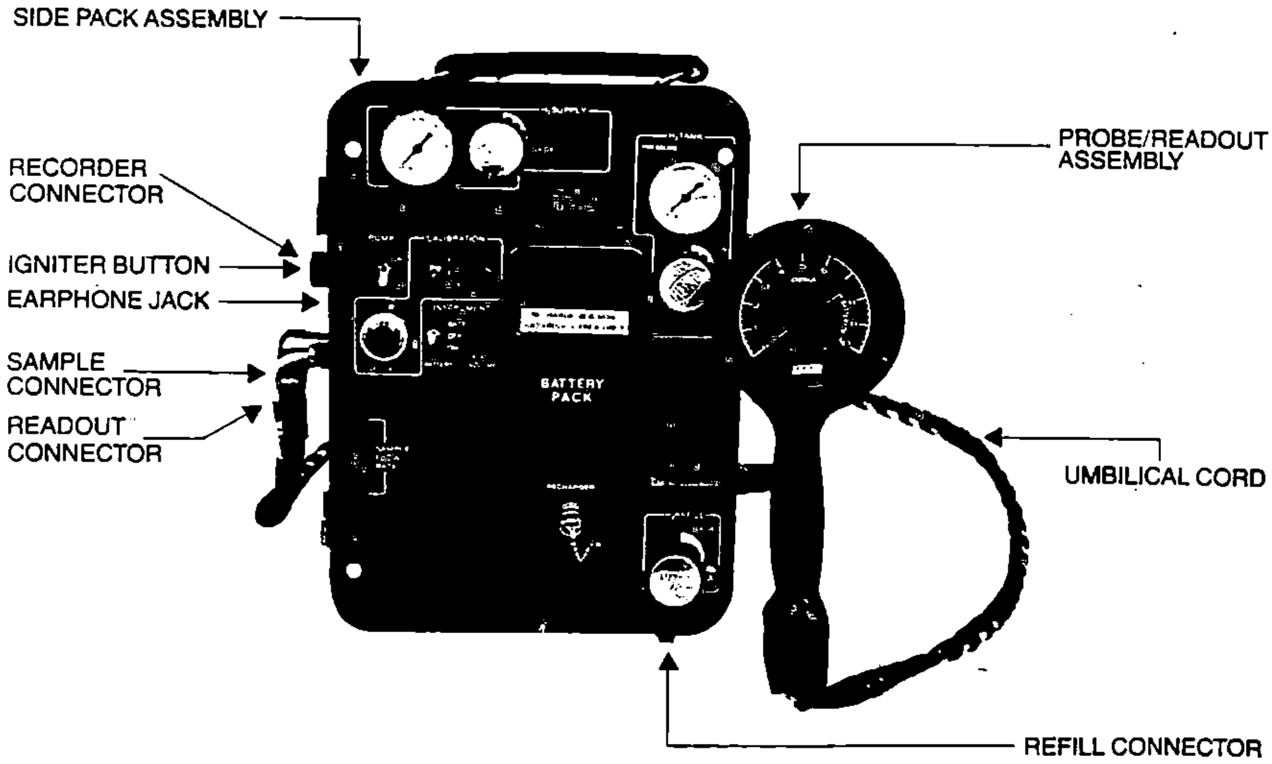


FIGURE 2
PORTABLE ORGANIC VAPOR ANALYZER
Model OVA 128

Typical response of various hydrocarbons, relative to methane is as follows:*

Compound	Relative Response (percent)
Methane	100 (reference)
Hexane	70
Propane	64
N-butane	61
N-pentane	100
Ethylene	85
Acetylene	200
Benzene	150
Toluene	120
Ethane	90

Compounds containing oxygen, such as alcohols, ethers, aldehydes, carbolic acid and esters give a lower response than that observed for hydrocarbons. This is particularly noticeable with compounds having a high ratio of oxygen to carbon such as the lower members of each series which have one, two or three carbons. With compounds containing higher numbers of carbons, the effect is diminished to such an extent that the response is similar to that of the corresponding hydrocarbons.

Nitrogen-containing compounds (i.e., amines, amides, and nitriles) respond in a manner similar to that observed for oxygenated materials. Halogenated compounds also show a lower relative response as compared with hydrocarbons. Materials containing no hydrogen, such as carbon tetrachloride, give the lowest response; the presence of hydrogen in the compounds results in higher relative responses. Thus, $CHCl_3$ gives a much higher response than does CCl_4. As in the other cases, when the carbon to halogen ratio is 5:1 or greater, the response will be similar to that observed for simple hydrocarbons.

---

*NOTE: Each OVA detector will have slightly different responses for organic vapors relative to methane. The user should determine responses for his individual instrument. The typical response of various compounds relative to methane is as follows:

KETONES	
Acetone	60
Methyl ethyl ketone	80
Methyl isobutyl ketone	100
ALCOHOLS	
Methyl alcohol	15
Ethyl	25
Isopropyl	65
HALOGEN COMPOUNDS	
Carbon tetrachloride	10
Chloroform	65
Trichloroethylene	70
Vinyl chloride	35

The OVA has negligible response to carbon monoxide and carbon dioxide which, due to their structure, do not produce appreciable ions in the detector flame. Thus, other organic materials may be analyzed in the presence of CO and CO_2.

Applications

(1) Measurement of most toxic organic vapors present in industry for compliance with Occupational Safety and Health Administration (OSHA) requirements.

(2) Evaluation and monitoring applications in the air pollution field.

(3) Source identification and measurement for fugitive emissions (leaks) as defined by EPA.

(4) Forensic science applications.

(5) Controlling and monitoring atmospheres in manufacturing and packaging operations.

(6) Leak detection related to volatile fuel handling equipment.

(7) Monitoring the background level of organic vapors at hazardous waste sites.

(8) Quality control procedures geared to leak checking, pressurized system checks, combustion efficiency checks, etc.

Major Features

The basic instrument consists of two major assemblies, the Probe/Readout Assembly and the Side Pack Assembly (See Figure 2). The recorder is optional on all models, but is normally used with all instruments which incorporate the GC Option. The output meter and alarm level adjustments are incorporated in the Probe/Readout Assembly.

The Side Pack Assembly contains the remaining operating controls and indicators, electronic circuitry, detector chamber, hydrogen fuel supply, and electrical power supply.

OPERATING PROCEDURES

Controls and Indicators

Sidepack Assembly

1) INSTR/BATT Test Switch* - Three position toggle switch controls all instrument electrical power except the pump and alarm power. It also permits display of the battery charge condition on the readout meter.

2) PUMP (ON/OFF) Switch* - Toggle switch controls power to the internal pump and audio alarms.

3) Igniter Switch - Momentary push button switch connects power to the igniter coil in the detector chamber and simultaneously disconnects power to pump.

4) CALIBRATE Switch (range selector) -Selects the desired range: X1 (0 to 10 ppm); X10 (0 to 100 ppm); X100 (0 to 1000 ppm).

5) CALIBRATE ADJUST (zero) Knob -Potentiometer used to "zero" the instrument.

6) GAS SELECT KNOB (span control) - Ten-turn dial readout potentiometer sets the gain of the instrument (commonly referred to as span control).

7) Recorder Connector - Five-pin connector used to connect the instrument to an external recorder with the following pin connections:

   Pin E - + 12 V dc
   Pin H - Ground
   Pin B - Signal 0 to 5 V dc

8) Charger Connector - BNC connector used to connect the battery pack to the battery charger.

9) HYDROGEN TANK VALVE - Valve used to supply or close off the fuel supply from the hydrogen tank.

10) HYDROGEN TANK PRESSURE Indicator - High pressure gauge measures pressure in the hydrogen fuel tank which is an indication of fuel supply.

11) HYDROGEN SUPPLY VALVE - Valve used to supply or close off hydrogen fuel to the detector chamber.

12) HYDROGEN SUPPLY PRESSURE Indicator - Low pressure gauge used to monitor hydrogen pressure at the capillary restrictor.

13) SAMPLE FLOW RATE Indicator - Indicator to monitor the sample flow rate.

14) REFILL CONNECTION - ¼ in AN fitting to connect the hydrogen refill hose to the instrument.

15) REFILL VALVE - Valve to open one end of the instrument fuel tank for refilling with hydrogen.

16) EARPHONE JACK - Used to connect the earphone; speaker is disabled when earphone is used.

17) VOLUME Knob - Potentiometer adjusts the volume of the internal speaker and earphone.

18) Readout and Sample Connectors - Used to connect the sample hose and umbilical cord from the Probe/Readout to the Side Pack.

Controls and Indicators

Probe/Readout Assembly

1) Meter - Linear scaled 270° meter displays the output signal level in ppm.

2) Alarm Level Adjust Knob - Potentiometer (located on the back of the Readout Assembly) is used to set the concentration level at which the audible alarm is actuated.

---

*Special Switch - switch handle must be pulled to change position. This prevents accidental movement.

Startup Procedure

a) Connect the Probe/Readout Assembly to the Sidepack Assembly by attaching the sample line and electronic jack to the Sidepack.

b) Select the desired sample probe (close area sampler or telescoping probe) and connect the probe handle. Before tightening the knurled nut, check that the probe accessory is firmly seated against the flat seals in the probe handle and in the tip of the telescoping probe.

c) Move the Instr/Batt Switch to the test position. The meter needle should move to a point beyond the white line, indicating that the integral battery has more than 4 hours of operating life before recharging is necessary.

d) Move the Instr/Batt Switch to the "ON" position and allow a 5 minute warm-up.

e) Turn the Pump Switch on.

f) Use the Calibrate Adjust knob to set the meter needle to the level desired for activating the audible alarm. If this alarm level is other than zero, the Calibrate Switch must be set to the appropriate range.

g) Turn the Volume Knob fully clockwise.

h) Using the Alarm Level Adjust knob, turn the knob until the audible alarm is activated.

i) Move the Calibrate Switch to X1 and adjust the meter reading to zero using the Calibrate Adjust (zero knob).

j) Open the hydrogen Tank Valve 1 or 2 turns and observe the reading on the Hydrogen Tank Pressure Indicator. (Approximately 150 psi of pressure is required for each hour of operation).

k) Open the Hydrogen Supply Valve 1 or 2 turns and observe the reading on the Hydrogen Supply Pressure Indicator. The reading should be between 8 and 12 psi.

Note: With GC instrument, a column or jumper must be installed.

l) After approximately one minute, depress the Igniter Button until the hydrogen flame lights. The meter needle will travel upscale and begin to read "Total Organic Vapors". Caution: Do not depress igniter for more than 6 seconds. If flame does not ignite, wait one minute and try again.

m) The instrument is ready for use. NOTE: If the ambient background organic vapors are "zeroed out" using the Calibrate Adjust knob, the meter needle may move off-scale in the negative direction when the OVA is moved to a location with lower background. If the OVA is to be used in the 0 to 10 ppm range, it should be "zeroed" in an area with very low background. A charcoal filter (Part No. 510095-1) can be used to generate the clean background sample.

Operating Procedures

The following procedure describes operation of the OVA in the "Survey Mode" to detect total organic vapors.

a) Set the CALIBRATE Switch to the desired range. Survey the areas of interest while observing the meter and/or listening for the audible alarm indication. For ease of operation, carry the Side Pack Assembly positioned on the side opposite the hand which holds the Probe/Readout Assembly. For broad surveys outdoors, the pickup fixture should be positioned several feet above ground level. When making quantitative readings or pinpointing, the pickup fixture should be positioned at the point of interest.

b) When organic vapors are detected, the meter pointer will move upscale and the audible alarm will sound when the setpoint is exceeded. The frequency of the alarm will increase as the detection level increases.

If the flame-out alarm is actuated, check that the pump is running, then press the igniter button. Under normal conditions, flame-out results from sampling a gas mixture that is above the lower explosve level which causes the hydrogen flame to extinguish. If this is the case, reignition is all that is required to resume monitoring. Another possible cause for flame-out is restriction of the sample flow line which would not allow sufficient air into the chamber to support combustion. The normal cause for such restriction is a clogged particle filter.

It should be noted that the chamber exhaust port is on the bottom of the case and blocking this port with the hand will cause fluctuations and/or flame-out.

Shut Down Procedure

The following procedure should be followed for shut down of the equipment:

A. Close HYDROGEN TANK VALVE
B. Close HYDROGEN SUPPLY VALVE
C. Move INSTR Switch to OFF
D. Wait 5 seconds and move PUMP Switch to OFF. INSTRUMENT IS NOW IN A SHUT DOWN CONFIGURATION.

Fuel Refilling

NOTE: Use PREPURIFIED or ZERO grade hydrogen (certified total hydrocarbons as methane <0.5 ppm recommended).

a) The instrument and the charger should be completely shut down during hydrogen tank refilling operations. Refilling should be done in a ventilated area. THERE SHOULD BE NO POTENTIAL IGNITERS OR FLAME IN THE AREA.

b) If you are making the first filling on the instrument or if the filling hose has been allowed to fill with air, the filling hose should be purged with hydrogen prior to filling the instrument tank. This purging is not required for subsequent fillings.

c) The filling hose assembly should be left attached to the hydrogen supply tank when possible. Ensure that the FILL/BLEED Valve on the instrument end of the hose is in the OFF position. Connect the hose to the refill connection on the Side Pack Assembly.

d) Open the hydrogen supply bottle valve slightly. Open the REFILL VALVE and the HYDROGEN TANK VALVE on the instrument panel and place the FILL/BLEED Valve on the filling hose assembly in the FILL position. The pressure in the instrument tank will be indicated on the HYDROGEN TANK PRESSURE Indicator.

e) After the instrument fuel tank is filled, close the REFILL VALVE on the panel, the FILL/BLEED Valve on the filling hose assembly and the hydrogen supply bottle valve.

f) The hydrogen trapped in the hose should now be bled off to atmospheric pressure. CAUTION should be used in this operation as described in Step (g) below, since the hose will contain a significant amount of hydrogen at high pressure.

g) The hose is bled by turning the FILL/BLEED Valve on the filling hose assembly to the BLEED position. After the hose is bled down to atmospheric pressure, the FILL/BLEED Valve should be turned to the FILL position to allow the hydrogen trapped in the connection fittings to go into the hose assembly. Then, again, turn the FILL/BLEED Valve to the BLEED position and exhaust the trapped hydrogen. Then turn the FILL/BLEED Valve to OFF to keep the hydrogen at one atmosphere in the hose so that at the time of the next filling there will be no air trapped in the filling line.

h) Close the HYDROGEN TANK VALVE.

i) With the HYDROGEN TANK VALVE and the HYDROGEN SUPPLY VALVE closed, a small amount of HYDROGEN at high pressure will be present in the regulators and plumbing. As a leak check, observe the HYDROGEN TANK PRESSURE Indicator while the remainder of the system is shut down and ensure that the pressure reading does not decrease rapidly (more than 350 psi/h) which would indicate a significant leak in the supply system.

Battery Charging

WARNING: Never charge in a hazardous environment.

a) Plug charger connector into mating connector on battery cover and insert ac plug into 115 V ac wall outlet.

b) Move the battery charger switch to the ON position. The lamp above the switch button should illuminate.

c) Battery charge condition is indicated by the meter on the front panel of the charger; meter will deflect to the left when charging. When fully charged, the pointer will be in line with "charged" marker above the scale.

d) Approximately one hour of charging time is required for each hour of operation. However, an overnight charge is recommended. The charger can be left on indefinitely without damaging the batteries. When finished, move the battery charger switch to OFF and disconnect from the Side Pack Assembly.

THE FOLLOWING ARE SPECIAL INSTRUCTIONS FOR RECHARGING BATTERIES WHICH HAVE BEEN COMPLETELY DISCHARGED.

It has been established that the above battery charging procedures may not be effective when the operator has allowed the battery to COMPLETELY discharge.

When this happens and the above procedures fail to charge the battery, perform the following additional steps:

e) Remove the battery from the instrument case.

f) Connect to any variable dc power supply.

g) Apply 40 volts at ½ ampere maximum.

h) Observe the power supply meter. As soon as the battery begins to draw current, gradually reduce the power maintaining ½ A maximum until the meter reads approximately 15 volts.

NOTE: The time required to reach the 15 volt reading will depend on degree of discharge.

i) Repeat steps (a), (b), (c), and (d) above to complete the charging cycle.

SUMMARY OF OPERATING PROCEDURES

Start Up

a) Check battery condition by moving the INSTR Switch to the BATT position.

b) Move INSTR Switch to ON and allow five (5) minutes to warm-up.

c) Use the Calibrate Adjust knob to set the meter needle to the level desired for activating the audible alarm. If this alarm level is other than zero, the Calibrate Switch must be set to the appropriate range.

d) Turn the Volume Knob fully clockwise.

e) Using the Alarm Level Adjust knob, turn the knob until the audible alarm is activated.

f) Set CALIBRATE Switch to X1 position, use CALIBRATE Knob and set meter to read 0.

g) Move PUMP Switch to ON position, then place instrument panel in vertical position and check SAMPLE FLOW RATE indication. The normal range is 1.5 to 2.5 units. If less, check filters.

h) Open the HYDROGEN TANK VALVE and the HYDROGEN SUPPLY VALVE. Wait one minute for hydrogen to purge the system.

i) Depress Igniter Button until burner lights. Do not depress Igniter Button for more than six seconds. (If burner does not ignite, let hydgrogen flow for one minute and again attempt ignition.)

j) Use CALIBRATE Knob to "zero" out ambient background. For maximum sensitivity below 10 ppm, set CALIBRATE Switch to X1 and readjust zero on meter. To avoid false flame-out alarm indication, set meter to 1 ppm with CALIBRATE Knob and make differential readings from there.

Shut Down

a) Close the HYDROGEN SUPPLY VALVE

b) Close the HYDROGEN TANK VALVE

c) Move the INSTR Switch and PUMP Switch to OFF

d) Instrument is now in shut down configuration

CALIBRATION

Recalibration to Various Organic Vapors

The OVA 128 is capable of responding to nearly all organic compounds. At the time of manufacture, the analyzer is calibrated to mixtures of methane in air. For precise analysis it is necessary to recalibrate with the specific compound of interest. The GAS SELECT control is used to set the electronic gain for a particular compound.

The instrument is recalibrated using a mixture of a specific vapor in air, with known concentration. After the instrument is in operation and the normal background is zeroed, draw a sample of the calibration gas into the instrument. The GAS SELECT Knob on the panel is then used to set the readout meter indication to correspond to the concentration of the calibation gas mixture.

The instrument has now been calibrated to the vapor mixture being used. After this adjustment, the setting on the "digidial" should be recorded for that particular organic vapor compound. This exercise can be performed for a large variety of compounds, thereby generating a "library" which can be used for future reference without need for additional calibration standards.

To read a particular compound, the GAS SELECT control is turned to the predetermined setting for the compound. <u>Calibration on any one range automatically calibrates the other two ranges</u>.

Using Empirical Data

Relative response data can be used to estimate the concentration of a vapor without need to recalibrate the analyzer. With the instrument calibrated to methane, obtain the concentration reading for a calibration sample of the test vapor. The response factor (R) in percent, for that vapor is:

$$R = \frac{\text{Actual Concentration}}{\text{Measured Concentration}}$$

To determine the concentration of an unknown sample of that vapor, multiply the measured concentration by R.

Calibration Standards

Commercial Standards

Commercially available standard samples offer the most convenience and are recommended for the most precise analyses. Always remember to obtain the desired vapor <u>in an air background</u>. Samples should be drawn from the cylinder into a collapsed sample bag, then drawn from the bag by the instrument to prevent a pressure or vacuum at the sample inlet.

Preparation of Standards

The following procedure is for generating calibration standards as an alternative to using commercial mixtures.

Obtain a five (5) gallon glass bottle and determine its volume by measuring the volume of water needed to fill it (use of a 1000 mL graduated cylinder is convenient). Another approach is to weigh the empty bottle, fill it with water and weigh again. The difference between the two values is the weight of water. By multiplying the weight of water in pounds by 0.455, obtain the volume of the bottle in liters. Empty the water and allow the bottle to dry. Place a one-foot piece of Teflon tubing in the flask to aid in mixing the vapors uniformly with the air. The volume of such a bottle should be about 20 liters, which is 20,000 mL. If the volume were 20,000 mL, then a 2 mL sample of a gas would be equivalent to 200 mL per 2 million mL or 100 ppm (V/V). Use of a gas tight Syringe, readable in 0.01 mL, allows the preparation of mixtures in the 1-2 ppm range, which are sufficient for the quantitative estimation of concentrations. A plastic stopper is loosely fitted to the tip of the bottle. The needle of the syringe is placed inside the jug neck and the stopper squeezed against the needle to decrease leakage during sample introduction. Inject the sample into the bottle and withdraw the needle without removing the stopper. Tighten the stopper and shake the bottle for a few minutes with sufficient vigor that the plastic tubing in the bottle moves around to ensure good mixture of the vapors with the air.

Calculations

$$\text{Injection} = \frac{\text{Volume Concentration X Molecular Weight X System Volume}}{\text{Density X Molar Volume at STP*}}$$

$$= \frac{(C)\ (MW)\ (V)}{(D)\ (V)}$$

Using the Ideal Gas Law, PV=RT, the molar volume of any gas at STP ($25\,^{\circ}C$ and 1 atm) is:

$$V = \frac{RT}{P} = \frac{\text{Universal Gas Constant x Temperature}}{\text{Pressure}}$$

$$= \frac{\left[\left(0.08206\ \frac{\text{litre atm}}{\text{mol K}}\right)\right](298.15\ K)}{1\ \text{atm}}$$

$$= (24.47\ L)\ (\text{mol}^{-1})$$

Therefore, the injection volume necessary to prepare 1 liter of a 100 ppm sample of hexane would be:

$$\text{Injection Volume} = \frac{(100\ \text{ppm})\ [(86.18\ g)\ (\text{mol}^{-1})]\ (1\ \text{liter})}{[(0.659\ g)\ (\text{mL}^{-1})]\ [(24.47\ L)\ (\text{mol}^{-1})]\ [(1000\ \text{mL})\ (l^{-1})]}$$

$$= 0.534\ \mu L$$

* STP - Standard Temperature and Pressure

Primary Calibration for Methane

Internal electronic adjustments are provided to calibrate and align the circuits. After initial factory calibration, it should not be necessary to repeat the calibration unless the analyzer undergoes repairs which affect calibration. If the OVA 128 will be extensively used for analysis of a sample other than methane, recalibration of the electronics (after resetting the GAS SELECT CONTROL) may result in better accuracy. See Recalibration to Various Organic Vapors above.

Primary calibration of this instrument is accomplished at the factory using methane-in-air, sample gases.

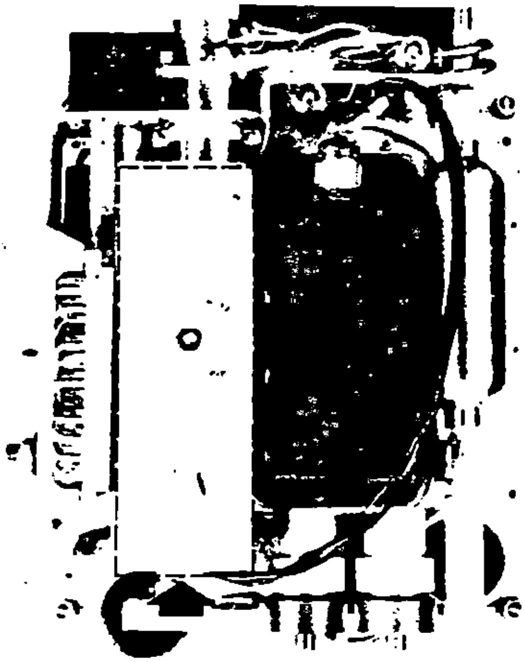


FIGURE 4
LOCATION OF ELECTRONIC ADJUSTMENTS

Calibration Using Known Samples for Each Range *(Refer to Figure 4)*

The accuracy stated under Specifications is obtained when the instrument is calibrated with known concentrations for each range. Prepare separate samples of methane-in-air in these concentration ranges: 7 to 10 ppm, 90 to 100 ppm, and 900 to 1000 ppm. Calibrate the instrument as follows:

a) Place the instrument in normal operation and allow a minimum of 15 minutes for warm-up and stabilization.

b) Set the GAS SELECT control to 300.

c) Set the CALIBRATE Switch to X1.

d) Set the CALIBRATE ADJUST (Zero) Knob so that the meter reads zero.

e) Check that the meter reads zero on the X10 and X100 ranges.

f) Set the CALIBRATE Switch to X1 and introduce the sample with known concentration in the 7 to 10 ppm range.

g) Adjust R31 so that the meter reading corresponds to the sample concentration.

h) Set the CALIBRATE Switch to X10 and introduce the sample with known concentration in the 90 to 100 ppm range.

i) Adjust R32 so that the meter reading corresponds to the sample concentration.

j) Set the CALIBRATE Switch to X100 and introduce the sample with known concentration in the 900 to 1000 ppm range.

k) Adjust R33 so that the meter reading corresponds to the sample concentration.

l) The instrument is now calibrated for methane and ready for service.

Calibration Using a Single Sample Calibration *(Refer to Figure 4)*

Calibration may be accomplished using a single known sample of methane in air in the range of 90 to 100 ppm. This may not provide the accuracy stated under specifications but is adequate for field survey work.

a) Place instrument in normal operation with CALIBRATE Switch set to X10 and GAS SELECT control set to 300.

b) Use the CALIBRATE ADJUST (zero Knob to adjust the meter reading to zero.

c) Introduce a methane sample of a known concentration (between 90 and 100 ppm not to exceed 100 ppm) and adjust trimpot R-32 so the meter reading corresponds to the known sample.

d) This sets the instrument gain for methane with the panel mounted gain adjustment (GAS SELECT) set at a reference number of 300.

e) Turn off HYDROGEN SUPPLY VALVE to put out flame.

f) Leave CALIBRATE Switch on X10 position and use CALIBRATE ADJUST (zero) Knob to adjust meter reading to 4 ppm.

g) Place CALIBRATE Switch in X1 position and using trimpot R-31 adjust meter reading to 4 ppm.

h) Move CALIBRATE Switch to X10 position again. Use CALIBRATE ADJUST (zero) Knob to adjust meter to a reading of 40 ppm.

i) Move CALIBRATE Switch to X100 position and use trimpot R-33 to adjust meter reading to 40 ppm.

j) Move CALIBRATE ADJUST (zero) Knob to adjust meter reading to zero.

k) Unit is now balanced from range to range, calibrated to methane, and ready to be placed in normal service.

SAFETY PRECAUTIONS

The OVA 128 has been tested and certified by Factory Mutual Research Corporation (FM) as safe for use in Class I, Division 1, Groups A, B, C and D hazardous atmospheres. Similar foreign certifications have been obtained, including BASEEFA. Special restrictions must be strictly adhered to, to ensure the certification is not invalidated by actions of operating or service personnel.

All flame ionization hydrocarbon detectors are potentially hazardous since they use hydrogen or hydrogen mixtures in the detector cell. Mixtures of hydrogen and air are flammable over a wide range of concentrations whether an inert gas such as nitrogen is present or not. Therefore, the recommended precautions and procedures should be followed for maximum safety. Safety considerations were a major factor in the design of the Organic Vapor Analyzer (OVA).

All connections are of the permanent type as opposed to quick disconnect. To protect against external ignition of flammable gas mixtures, the flame detection chamber has porous metal flame arrestors on the sample input and the exhaust ports as well as on the hydrogen inlet connector. The standard battery pack and other circuits are internally current limited to an intrinsically safe level.

No Modifications Permissible

It is imperative that operation and service procedures described in this manual be carefully followed in order to maintain the intrinsic safety which is built into the OVA. NO MODIFICATION TO THIS INSTRUMENT IS PERMISSIBLE. Therefore, component replacement must be accomplished with approved parts.

Electrical Protection

The 12 V battery power supply circuit is current limited to an instrinsically safe level. Fuses are not utilized and all current limiting resistors and other components which are critical to the safety certification are encapsulated to prevent inadvertent replacement with components of the wrong value or specification. Under no circumstances should the encapsulation be removed.

Fuel Supply System

The OVA fuel tank has a volume of approximately 75 cm which, when filled to the maximum rated pressure of 2300 psig, holds approximately 5/8 ft of gas. The fuel used in the OVA should be PREPURIFIED or ZERO grade hydrogen (certified total hydrocarbons as methane <.5 ppm recommended.)

Hydrogen gas gains heat when expanding and, therefore, should not be rapidly released from a high pressure tank to a low pressure environment. Flow restrictors are incorporated in the hydrogen refill fitting and hydrogen is restricted on the output side of the tank by the low flow rate control system. In addition, a special flow restrictor is incorporated in the FILL/BLEED valve of the hydrogen filling hose assembly. These precautions limit the flow rate of the hydrogen to prevent ignition due to self-heat from expansion.

Precautions should be taken during hydrogen filling or hydrogen emptying operations to ensure that there are no sources of ignition in the immediate area. Since the instrument tank at 2300 psig holds only 5/8 ft of hydrogen, the total quantity, if released to the atmosphere, would be quickly diluted to a non-flammable level. There is, however, the possibility of generating flammable mixtures in the immediate vicinity of the instrument during filling or emptying operations if normal care is not exercised.

Detector Chamber

The input and output ports of the flame ionization chamber have sintered metal flame arrestors. The chamber is ruggedly constructed of Teflon such that even if highly explosive mixtures of hydrogen and air are inadvertently created in the chamber and ignited, the chamber would NOT rupture.

MAINTENANCE

This section describes the routine maintenance schedule and provides procedures for trouble-shooting an instrument malfunction.

CAUTION: Maintenance personnel should be thoroughly familiar with instrument operation before performing maintenance. It is essential that all portions of this manual relating to safety of operation, servicing and maintenance, be thoroughly understood. There should be no potential igniters or flame in the area when filling, emptying or purging the hydrogen system and the instrument should be turned off.

Extreme care should be exercised to ensure that required parts replacement is accomplished with the parts specified by Foxboro. NO MODIFICATIONS ARE PERMITTED. DISASSEMBLE INSTRUMENT ONLY IN A NON-HAZARDOUS ATMOSPHERE.

Routine Maintenance *(Refer to Figure 5)*

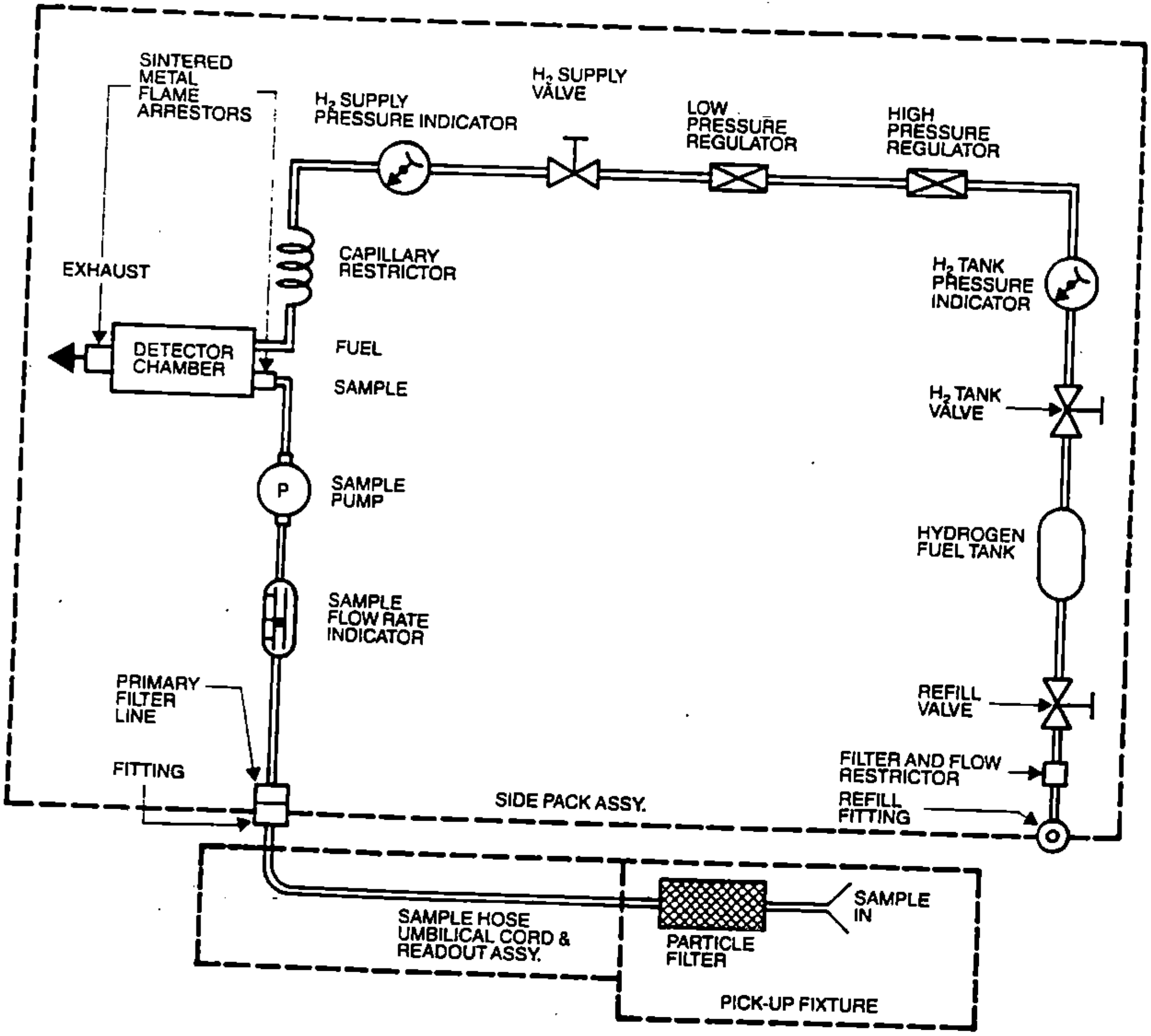


FIGURE 5
BLOCK DIAGRAM - GAS HANDLING SYSTEM

Primary Filter

This filter is located behind the sample inlet connector (Fitting Assembly) on the Side Pack Assembly and is removed for cleaning by using a 7/16 inch thin wall socket to unscrew the Fitting Assembly. The filter cup, "O" ring and loading spring will then come out. The porous stainless filter cup can be cleaned by blowing out or washing in solvent. If a solvent is used, care should be taken to ensure that all solvent is removed by blowing out or heating the filter. Reassemble in reverse order ensuring that the "O" ring seal on the Fitting Assembly is intact.

Secondary Filter

A particle filter is located in each pick-up fixture. One of these filters must be in the sample line whenever the instrument is in use. The OVA 128 uses a porous metal filter which can be replaced or cleaned.

Mixer/Burner Assembly Filter

A porous metal particle filter is incorporated in the Mixer/Burner Assembly which screws into the Preamp Assembly. This filter is used as the sample mixer and inlet flame arrestor in the chamber. The filter should not become contaminated under normal conditions but can be cleaned or the assembly replaced if necessary.

Access to this filter or output surface does not require removing the instrument from the case. For access, remove the safety cover using a hex key wrench (supplied) then unscrew the exhaust port. The Filter Assembly can now be seen on the side of the chamber (Preamp Assembly) and can be cleaned with a small wire brush.

Exhaust Flame Arrestor

A porous metal flame arrestor is located in the exhaust port of the detector chamber (Preamp Assembly). It acts as a particle filter on the chamber output and restricts foreign matter from entering the chamber. This filter may be cleaned by removing the exhaust port. For access, see Mixer/Burner section above. Note that the filter is captive to the exhaust port. Clean the filter with a solvent or detergent and ensure that it is dry and completely baked out at 120°F before reinstalling.

Sampling Fixtures

Sampling fixtures should be periodically cleaned with an air hose and/or detergent water to eliminate foreign particle matter.

If a solvent is used, the fixture should be subsequently cleaned with detergent and baked out at 120°F to eliminate residual hydrocarbons from the solvent.

Hydrogen Tank Supply & Refill Valves

After some time, the Teflon washers under each valve packing nut can "cold flow" (move with pressure) and allow hydrogen to leak. Leakage can be determined by using Leak-Tec, Snoop or a soap solution around the valve stems. This leakage can usually be stopped by tightening the compression nut (adapter) as outlined below.

a) Unscrew the packing nut with a 7/16 inch wrench
b) Unscrew the valve
c) Replace the compression rings

This compression is against soft material and only a small amount of force is necessary to sufficiently compress the Teflon washers. If, after tightening, leakage still occurs, it would be advisable to replace the two Teflon washers, as follows:

a) Drain hydrogen system slowly and to the extent necessary to work on the leaking valve(s). Observe safety precautions. There should be no potential igniters in the area.

b) Remove all three (3) knob screws and knobs.

c) Remove the compression nut on the valve that is not sealing properly. Remove the stem by unscrewing it from the valve body. Observe the sandwich of metal and Teflon washers and note their order.

d) Visually check the Kel-F^{TM} seat on the stem for cracks or foreign material. Wipe clean, if necessary, with a lint free cloth (no solvents or oils) and replace if damaged.

e) Remove the washers and replace the Teflon washers (the factory procedure is a light wipe of HYDROCARBON FREE silicone grease).

f) Replace the stem assembly in the valve body and tighten lightly.

g) Push the washers down into the compression area in the same order as noted upon removal. Replace the compression nut and tighten snuggly.

h) Close the low pressure valve and fill the tank assembly. Check valves for leaks. Tighten again, if necessary, and reassemble the unit.

Air Sampling System Maintenance

A potential problem associated with the OVA instrument is that leaks can develop in the air sample pumping system. These leaks can result in dilution or loss of sample, causing low reading of vapor concentration and slow response.

The OVA is equipped with a flow gauge that provides a method to check for air leaks. Assemble the pickup probe selected for use to the readout assembly and then position the sidepack vertically so the flow gauge may be observed. Cover the end of the pickup probe with your finger and observe that the ball in the flow gauge goes to the bottom, indicating no air flow (if ball has slight chatter while on bottom, this is acceptable). Cover the center of the chamber exhaust port with your thumb and again observe the ball going to the bottom. Another simple check is to expose the pickup probe to cigarette smoke or a light vapor (butane) and observe that the meter responds in approximately 2.0 seconds. It should be noted that slow meter response may also indicate a restriction in the air sampling system.

Failure of the ball to go to the bottom when the inlet is blocked indicates a leak in the system between the probe and the pump inlet or the inlet check valve. To isolate the problem, remove parts, one at a time, and again block off the air inlet. Remove the pickup probe(s) and cover the air inlet at the Readout Assembly. If the ball goes to the bottom, check that the "readout to probe" seal washer is in place and replace the probes, holding them back against this seal while tightening the nut. Recheck, and if leakage is still present, it is probably in the probe (pickup fixture), which should be repaired or replaced.

If leakage is indicated as being past the readout handle when the connection to the sidepack is tight, disconnect the sample line at the fitting on the sidepack and cover this inlet with your finger. If the flow gauge ball goes to the bottom, the problem should be a leak in the umbilical cord/Readout Assembly, which should be investigated and repaired. There is also the possibility of a leaking check valve in the pump which would not show up on this test. If the leakage is not found in the umbilical cord, it is most likely in the pump check valve. The pump should be replaced.

If the ball does not go to the bottom, the leak will be either in the flow gauge or it's connecting tubing. Visually check that the tubing is connected and if so, the flow gauge should be repaired or replaced. Check the "O" ring installation in the sample inlet connector (Fitting Assembly).

As an alternate approach, leaks on the inlet side of the pump can be detected by using alcohol on a "Q" Tip and lightly swabbing the connections one at a time or by directing organic vapor or smoke at the potential leakage points and observing the meter response or audible alarm.

Leaks (beyond the pump) are easier to locate, as any of the commercially available leak detection solutions can be used. Cover the exhaust port, which will place the exhaust system under pressure, and check each connection, one at a time. Replace the Teflon tubing or retape the threaded connections with Teflon joint tape. Check the igniter and Mixer/Burner Assembly where they screw into the detector, the high voltage terminal screw on the side of the Mixer/Burner and exhaust port itself. If after these checks, the flow gauge ball still will not go to the bottom with the exhaust blocked, the problem is likely a leaking exhaust check valve in the pump, which should be repaired or replaced.

Contaminating Control

On occasion, the background reading may be relatively high under normal ambient conditions. Ambient background readings will vary somewhat depending on the geographical location where the instrument is being used. However, the background reading normally should be in the range of 3 to 5 ppm as methane. The acceptable background reading consists of 1 to 1½ ppm of methane which is present in the normal air environment. In addition to the measurement of a normal methane background, there will normally be 2 to 4 ppm of equivalent methane background caused by acceptable levels of contamination in the hydrogen fuel and/or hydrogen fuel handling system resulting in a total equivalent methane reading of 3 to 5 ppm in clean air.

If the background reading goes above 5 ppm to 6 or 7 ppm, this is normally still acceptable since any measurement is additive to that background reading, i.e., 2 ppm on top of 5 or 2 ppm on top of 7 provides the same differential reading, however, the lower background is obviously desirable.

The background reading is zeroed out or nulled - even though in reality the background still exists. The background reading is measured by zeroing the meter with the flame out and noting the meter indication after the flame is on.

The cause for a high background reading is usually associated with contamination in the hydrogen fuel system. This will, of course, cause a background reading since this is the function of the basic detector "to measure contamination entering the detector chamber". In addition, contamination present in the hydrogen will many times leave a small unobservable deposit on the burner face which can continue to generate a background reading when the detector is in operation and the burner assembly is heated.

Another possible cause of contamination is the Mixer/Burner Assembly when the contamination is trapped in the porous bronze sample filter. This is not a common problem and usually only happens when an unusually high level of contaminant is drawn into the assembly. Another possible cause of high background reading is contamination in the air sample line to the detector. This is uncommon but can be the source of the problem.

NOTE: An OVA that has the Chromatograph Option can have high background caused by saturation or contamination of the activated charcoal filter, which is in the line during chromatograph analysis, or of the column which is in the hydrogen line at all times.

Analysis and Correction

Prior to analyzing the problem, the OVA should be checked for proper electronic operation. It should be ensured that the instrument is calibrated to methane as referenced.

If, after checking that the OVA is properly calibrated, the background is still higher than normal for ambient conditions, the following procedure should be followed to isolate the cause of the problem:

a) Let the OVA run for a period of time (15 to 30 minutes) and see if the background level decreases as a function of time. The background could go down as a result of clearing line contamination which is removable simply by the normal flow of air through the sample line.

b) Take a reading in a known, relatively clean air environment. Normally, outside air environment is clean enough to assess by comparison whether the background reading is internal to the instrument or is present in the location where the instrument is being used.

c) If the OVA has the Gas Chromatograph Option, depress the sample inject valve, so that the activated charcoal is in the line, and observe whether the background reading goes down and stays steady after elution of the air peak. The reading should always go down or stay the same but never increase when the sample valve is depressed, since the charcoal filter will remove trace elements of organic vapors in the air sample heavier than C_2. If another activated charcoal filter is available, this may be attached to the end of the probe to scrub the air so that a clean air sample is supplied to the detector. The external activated charcoal filter can be used on any instrument, with or without chromatograph, for providing a clean air sample to assess background level.

d) If the background cannot be reduced by any of the previous steps, remove the safety cover and the exhaust port of the detector chamber (on the bottom of the case) and clean the cavity and the electrode using the small wire brush supplied with the analyzer. This will remove any small quantities of contamination which could be the source of the background vapor. After cleaning, replace the exhaust port and safety cover and reignite the OVA. If detector contamination was the cause, the problem should be immediately resolved and the ambient back ground will drop to an acceptable level.

e) If the high background is still present, the various parts of the sample flow line such as pickup probes, umbilical cord to the instrument, etc., should be investigated by the process of elimination to see if the contamination can be isolated.

Serious contamination in the air sample line is very uncommon, however, if very large doses of low vapor pressure compounds are sampled, there is a possibility of residual contamination. This would eventually clear itself out but may take a considerable period of time. A typical cause for high background from the sample line is a contaminated Mixer/Burner Assembly. If heavy contamination of the Mixer/Burner is indicated, replace the Mixer/Burner Assembly.

f) In the event of contamination in the pump or other internal parts of the sample flow lines which cannot be removed, the sample flow components have to be disassembled and cleaned. This is normally a factory operation, however, components such as the pump can be replaced in the field along with any contaminated tubing.

g) High background readings on OVA's which include the Gas Chromatograph Option can be caused by other sources of contamination. If the charcoal filter mounted on the instrument panel is saturated, contaminated air would be supplied to the detector and raise the ambient level background. To check for this, refill the cartridge with fresh charcoal, Foxboro P/N CSC004. This would determine if the charcoal was the source of the background reading. It is also possible that a high background reading could be due to contamination in the column. This could be caused by compounds slowly eluting from a column which has become contaminated. The easiest way to check for column contamination is to replace the column with a clean column or a short empty piece of column tubing and see if the high background reading drops.

h) If the above steps do not correct the high background, the cause will normally be contamination in the hydrogen fuel system.

Contamination in the hydrogen fuel system is usually the direct result of contaminated hydrogen gas or contamination introduced during the filling operation. Filling hose contamination can be caused by storing the hose in a contaminated area.

To remove contamination, the fuel system should be purged with hydrogen. Effective purging is accomplished by disconnecting the capillary tube fitting to the manifold block which has the low pressure gauge (Hydrogen Supply Pressure Gauge and Hydrogen Supply Valve). This disconnects the capillary tubing from the hydrogen line so that hydrogen may be purged at a reasonable rate from the tank assembly through the regulators, gauges and valves. After disconnecting the capillary, the hydrogen tank can be filled in the normal manner. The tank valve and hydrogen supply valve can then be opened which will bleed the hydrogen from the tank through the hydrogen fuel system, purging contamination which is in vapor form. There is the possibility that contamination has been introduced into the hydrogen fuel system which is not readily purged by the hydrogen gas, but this is unlikely. After purging with clean hydrogen two or three times, the capillary tube should be reconnected and the background again checked. Five or ten minutes should be allowed before assessing the background reading, since contaminated hydrogen can be trapped in the capillary tube.

If another clean instrument is available, the fuel system from the clean instrument can be connected to the contaminated instrument to verify whether the problem is associated with the hydrogen fuel supply system. The interconnection should be made to the capillary tube of the contaminated instrument.

Troubleshooting

Table 1 presents a summary of field troubleshooting procedures. If necessary, the instrument can be easily removed from the case by unlocking the four (4) ¼ turn fasteners on the panel face and removing the refill cap. The battery pack is removed by taking out the four (4) screws on the panel and disconnecting the power connector.

Factory Maintenance

To ensure continuous trouble-free operation, a periodic factory maintenance, overhaul, and recalibration is recommended. The recommended schedule is every six to nine months. This maintenance program includes replacement of plastic seals and parts as required, pump overhaul, motor check, sample line cleaning, hydrogen leak check, recalibration, and detailed examination of the unit for any other required maintenance and repair.

Recommended Spare Parts

Item	Description	Part Number	Recommended Quantity
1	Igniter	510461-1	2
2	Pump Assembly	510223-6	1
3	Cup, Filter (3/8 inch OD, ss)	510318-1 (5/pkg.)	1
4	Mixer/Burner Assembly	510513-1	1
5	Wafer, Teflon, H_2 Valve	510160-1 (10/pkg.)	1
6	Washer, Brass, H_2 Valve	510160-2 (10/pkg.)	1
7	Exhaust Port Assembly	510530-1	1
8	Battery Pack Assembly	510542-1	1
9	Sample Line Assembly	510316-1	1
10	Particle Filters	510116-1	1

TABLE 1

PROBLEM	TROUBLE SHOOTING PROCEDURE	REMEDY
1) Low sample flow rate on flow indicator. Nominally 2 units on flow gauge. (See also 6 below)	a) Check primary filter in sidepack and particle filters in the pickup assembly.	Replace or clean filter if clogged.
	b) Determine assembly containing restriction by process of elimination, i.e., remove probe, remove Readout Assembly, remove primary filter, etc.	Investigate the assembly containing this restriction to determine cause of blockage. Clean or replace as required.
	c) If the restriction is in the Side Pack Assembly, further isolate by disconnecting the sample flow tubing at various points, i.e., pump output chamber, etc. NOTE: The inherent restrictions due to length of sample line, flame arrestors, etc., must be taken into account when troubleshooting.	If in the detector chamber, remove and clean or replace porous metal flame arrestors. If pump is found to be the problem, remove and clean or replace.
2) Hydrogen flame will not light. (See also 6 below)	a) Check sample flow rate (see 1 above)	If sample flow rate is low, follow procedure 1 above.
	b) Check igniter by removing the chamber exhaust port and observing the glow when the IGNITE BUTTON is depressed.	If igniter does not light up, replace the plug. If igniter still does not light, check the battery and wiring.
	c) Check for rated Hydrogen Supply Pressure. (Listed on calibration plate on pump bracket).	If low, remove battery pack and adjust to proper level by turning the allen wrench adjustment on the low pressure regulator cap.
	d) Check hydrogen flow rate by observing the psi decrease in pressure on the Hydrogen Tank Pressure gauge. The correct flow rate will cause about 130 psi decrease in pressure per hour. (Approximately 12 cm^3/min at detector).	The most likely cause for hydrogen flow restriction would be a blocked or partially blocked capillary tube. If flow rate is marginally low, attempt to compensate by increasing the Hydrogen Supply Pressure by one-half or one psi. If flow rate cannot be compensated for, replace capillary tubing.
	e) Check all hydrogen plumbing joints for leaks using soap bubble solution. Also, shut off all valves and note pressure decay on hydrogen tank gauge. It should be less than 350 psi per hour.	Repair leaking joint.

TABLE 1

PROBLEM	TROUBLE SHOOTING PROCEDURE	REMEDY
	f) Check to see if hydrogen supply system is frozen up by taking unit into a warm area.	If there is moisture in the hydrogen supply system and the unit must be operated in subfreezing temperatures, purge the hydrogen system with dry nitrogen and ensure the hydrogen gas used is dry.
	g) Remove exhaust port and check for contamination.	If the chamber is dirty, clean with ethyl alcohol and dry by running pump for approximately 15 minutes. If hydrogen fuel jet is misaligned, ensure the porous metal flame arrestor is properly seated.
	h) Check spacing between collecting electrode and burner tip. Spacing should be 0.1 to 0.15 inches.	Adjust by screwing Mixer/Burner Assembly in or out. This spacing problem should only occur after assembling a Mixer/Burner Assembly to a Preamp Assembly.
3) Hydrogen flame lights but will not stay lighted.	a) Follow procedures 2(a), (c), (d), (e), (g) and (h) above. Also refer to 5 below.	
4) Flame-out alarm will not go on when hydrogen flame is out.	a) Check instrument calibration setting and GAS SELECT control setting.	Readjust as required to proper setting. Note that the flame-out alarm is actuated when the meter reading goes below zero.
	b) Remove exhaust port and check for leakage current path in chamber (probably moisture or dirt in chamber).	Clean contamination and/or moisture from the chamber using a swab and alcohol, dry chamber by running pump for approximately 15 minutes.
	c) If above procedures do not resolve the problem, the probable cause is a malfunction in the preamp or power board assemblies.	Return preamp chamber or power board assembly to the factory for repair.
	d) Check that volume control knob is turned up.	Adjust for desired volume.

TABLE 1

PROBLEM	TROUBLE SHOOTING PROCEDURE	REMEDY
5) False flame-out alarm.	a) Flame-out alarm is actuated when signal goes below electronic zero (with flame on). This can be due to inaccurate initial setting, drift, or a decrease in ambient concentration. Verify if this is the problem by zeroing meter with flame out and reigniting.	When using the X1 range adjust meter to 1 ppm, rather than zero, be sure instrument has been zeroed to "lowest expected ambient background level".
6) Slow response, i.e., time to obtain response after sample is applied to input is too long.	a) Check to ensure that probe is firmly seated on the rubber seal in the readout assembly.	Reseat by holding the probe firmly against the rubber seat and then lock in position with the knurled locking nut.
	b) Check sample flow rate per procedure 1 above.	See 1 above.
7) Slow recovery time, i.e., too long a time for the reading to get back to ambient after exposure to a high concentration or organic vapor.	a) This problem is normally caused by contamination in the sample input line. This requires pumping for a long period to get the system clean of vapors. Charcoal in the lines would be the worst type of contamination. Isolate through the process of elimination. (See 1(b)).	Clean or replace contaminated sample line or assembly as required.
	b) Check flame chamber for contamination.	Clean as required.
8) Ambient background reading in clean environment is too high.	a) A false ambient background reading can be caused by hydrocarbons in the hydrogen fuel supply system. Place finger over sample probe tube restricting sample flow and if meter indication does not go down signficantly the contamination is probably in the hydrogen fuel.	Use a higher grade of hydrocarbon free hydrogen. Check for contaminated fittings on filling hose assembly.
	b) A false ambient background reading can also be caused by a residue of sample building up on the face of the sample inlet filter. If the test in 8(a) above produces a large drop in reading, this is usually the cause.	Remove the exhaust port (it is not necessary to remove instrument from case). Use the small wire brush from the tool kit or a knife blade and lightly scrub surface of sample inlet filler.

TABLE 1

PROBLEM	TROUBLE SHOOTING PROCEDURE	REMEDY
	c) A false ambient background reading can also be caused by hydrocarbon contamination in the sample input system. The most likely cause would be a contaminant absorbed or condensed in the sample line. NOTE: It should be emphasized that running the instrument tends to keep down the buildup of background vapors. Therefore, run the unit whenever possible and store it with the carrying case open in clean air.	Clean and/or replace the sample input lines. Normally the false reading will clear up with sufficient running.
9) Pump will not run.	a) Check that there is no short circuit in wiring.	If no short circuit, pump motor is defective.
10) No power to electronics but pump runs.	a) Short circuit in electronics.	There is a short in the electronics assembly. Return OVA to factory or authorized repair facility.
11) No power to pump or electronics	a) Place battery on charger and see if power is then available. Recharge in a non-hazardous area only.	If power is available, battery pack is dead or open. Recharge battery pack. If still defective, replace battery pack.

GAS CHROMATOGRAPH (GC) OPTION

The Model OVA 128 CENTURY Organic Vapor Analyzer provides efficient and accurate indication of total organic compound concentrations on a continuous sampling basis. However, in areas where mixtures of organic vapors are present, it often becomes necessary to determine the relative concentration of the components and/or to make quantitative analysis of specific compounds.

To provide this capability, a gas chromatograph (GC) option is available. See Figure 6 for the location of the major components and controls associated with the GC option. When the GC option is used, the capability of the OVA includes both qualitative and on-the-spot quantitative analysis of specific components present in the ambient environment. The Recorder, which is used with the GC option, is described separately.

This section is applicable only to an OVA with the optional gas chromatograph system.

Modes of Operation

The OVA with GC option has two modes of operation. The first mode is the measurement of total organic vapors in the same manner as described for the basic OVA instrument. This mode is referred to as the "Survey Mode". The OVA is in the "Survey Mode" of operation whenever the Sample Inject Valve is in the "out" position.

The second mode of operation is called the "GC Mode". The OVA is in this mode of operation any time a sample has been injected into the GC system and the sample is being transported through the GC column. This section provides a brief description of how a gas chromatograph (GC) operates and specifically, how the model OVA 128 performs the required operations. A comprehensive discussion of gas chromatography theory, column selection, and data analysis is beyond the scope of this manual.

The OVA with GC option can be utilized for many types of analysis in the outdoor or indoor ambient environment or for specific laboratory type analysis. The OVA was not designed to compete with the research or process gas chromatograph but to compliment these instruments or eliminate their need in field applications.

This manual is intended to provide the operator with information to operate and maintain the OVA. Foxboro publishes Application/Technical Notes to assist the operators in applying the instrument to field monitoring situations.

All flame ionization detector (FID) gas chromatographs require certain elements for their operation. These elements include three flow regulated gas supplies as follows: 1) A carrier gas to transport the sample through the column; 2) Hydrogen gas for operation of the FID; 3) A clean air supply to support combustion to the FID. In addition, a method for injecting a known volume of sample air (aliquot) to be analyzed is required.

In standard gas chromatographs these three (3) flow regulated gases are individually supplied from pressurized cylinders equipped with regulators and flow control apparatus. The Model 128 GC system differs in that the hydrogen fuel for the FID is also used as the carrier gas. The clean air supply is simply the normal air sample pumped to the FID. During the GC analysis, this air is scrubbed in a charcoal filter to provide the clean air supply. The end result is that no additional gas supplies are required to add the GC option to the basic OVA instrument.

A valving arrangement is incorporated to provide a method for transferring a fixed volume of air into the GC system for analysis. The sample air injected into the GC column is the same sample being analyzed by the OVA for total organic vapor concentration. Therefore, the instrument provides the unique capability to observe the total organic vapor concentration of the sample prior to injecting it into the GC system. This operating feature is invaluable in field work where the environment is continually changing and where valuable GC analysis time must be expended only on the sample of concern.

OVA Columns

Columns are available in 4, 8, 12, 24, 36 and 48 inch lengths as standard offerings with any of the column packings listed below. Longer lengths are available in 12-inch increments on a non-standard basis. To order a column simply use the general part number for a column which is 510454 followed by a dash (-), the Foxboro packing material designation, a second dash and the desired length in inches. A sample column designation is 510454-G-24. This would represent a 24 inch column with 10% OV 101 on chromosorb W, HP 60/80 mesh. If a specific application arises which calls for a column material not listed below, please contact Foxboro. We will be happy to check on its availability.

Foxboro Designation	Material
A	20% Dioctyl Phthalate on Chromosorb-P, AW 60/80 Mesh
C	Chromosorb 101, 60/80 Mesh
D	20% Ucon 50 HB 280 on Chromosorb-P, AW 60/80 Mesh
E	20% Carbowax 400 on Chromosorb-P, AW 60/80 Mesh
F	5/1.75% Diethylhexyl Sebecate/Bentone 34 on Chomosorb W, AW 60/80 Mesh
G	10% OV-101 on Chromosorb W, HP 60/80 Mesh
T	10% 1,2,3-Tris (2-cyanoethoxy) Propane on Chromosorb P, AW 60/80 Mesh
B	3% Diisodecyl Phthalate on Chromosorb W, AW 60/80 Mesh
PT	Poropak T, 60/80 Mesh
Q	Poropak Q, 60/80 Mesh
H	20% Carbowax 20M on Chromosorb P, AW 60/80 Mesh
J	n-Octane on Porasil C, 80/100 Mesh
N	Porapak N, 60/80 Mesh

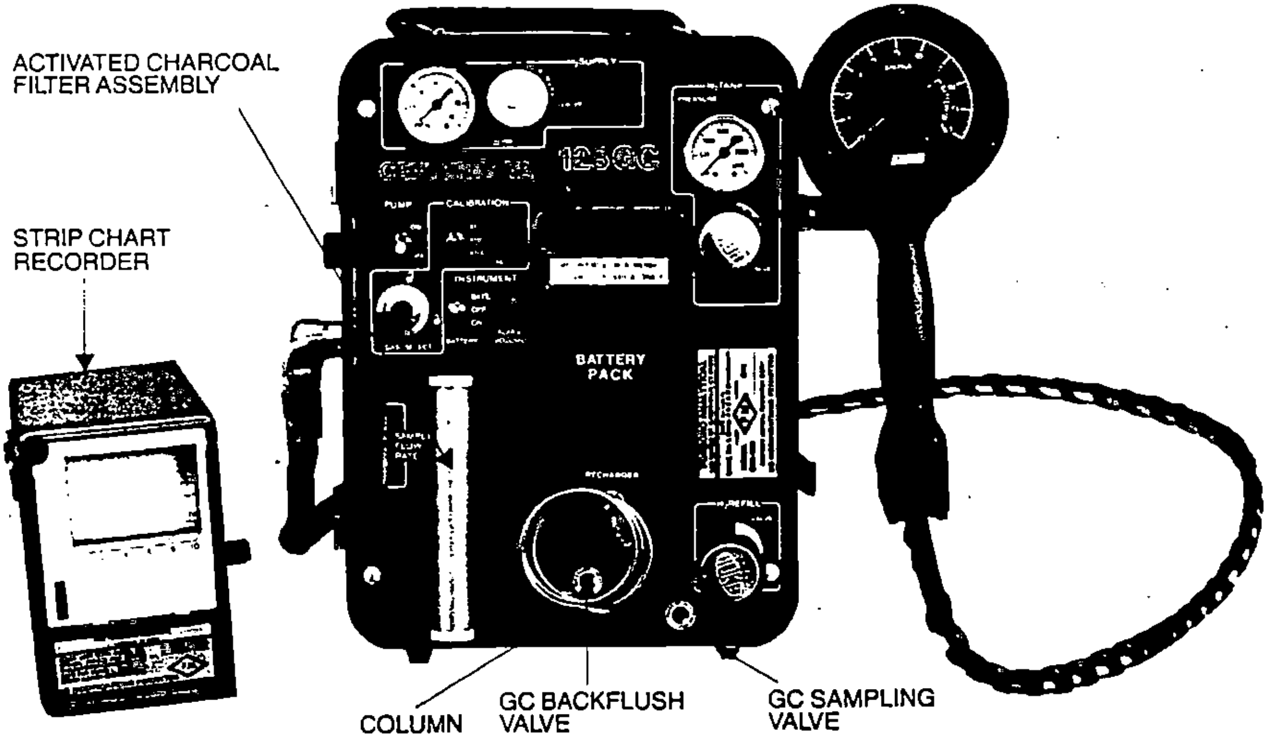


FIGURE 6
ADDITIONAL CONTROLS & COMPONENTS - GC OPTION

Sample Flow

Figure 7 is a flow diagram illustrating the flow paths of the hydrogen fuel, sample air supply, and GC injected sample aliquot.

Two push-pull valves are used in the GC system; the Sample Inject Valve and the Backflush Valve.

Block D illustrates the flow paths with the Sample Inject Valve in the "out" position. With this valve in the "out" position, the OVA functions in its normal manner as a total organic vapor analyzer.

Block C illustrates the flow paths after the Sample Inject Valve is moved to the "in" position to initiate the GC Mode.

The hydrogen flow path is now through the sample loop which enables hydrogen to sweep the air sample from the loop and carry it through the GC column.

Also note that the sample air going to the FID chamber is now routed through the activated charcoal filter where essentially all organic vapor contamination is removed from the air. The activated charcoal filter will effectively absorb most organic vapors with the exception of methane and ethane. The functions of the Sample Inject Valve are, therefore, to transfer a fixed volume sample of the air being monitored into the hydrogen stream and to reroute the sample air supply through a filter (scrubber).

The Backflush Valve has no prepositioning requirement to function. It can be in either the "in" or "out" position at the time a sample is injected into the GC system for analysis. The Backflush Valve simply reverses the direction of the hydrogen flow through the GC column.

Regardless of the operating mode, hydrogen always flows through the column to the FID detector and the sample air supply always flows to the FID detector to provide oxygen for the hydrogen flame.

The recommended hydrogen flow rate is 12 cm /min for proper FID operation and as a standard flow rate for generating GC reference/calibration data. This hydrogen flow rate is adjusted by varying the Hydrogen Supply Pressure, which is the hydrogen pressure at the input of the flow control capillary tube of the OVA. The pressure is changed by adjusting the set screw in the bonnet of the low pressure regulator, accessible by removing the battery pack from the instrument panel. To monitor the hydrogen flow rate, connect a bubble flowmeter to an end of the GC column which has been disconnected from the panel fitting and move the Backflush Valve so that hydrogen is flowing out of the column. Primary hydrogen flow control is accomplished by the capillary tube of the OVA. However, the flow restriction of a GC column will also affect the hydrogen rate and the effect will vary with column length, type of packing and packing methods. The nominal Hydrogen Supply Pressure is around 10 psig and the pressure drop across a typical 24 inch long column packed with 60/80 mesh material is approximately 1 to 1.5 psig. Normally, when the hydrogen flow rate is set at 12 cm^3/min with a standard 24 inch long column, no adjustment needs to be made when using columns from four (4) inches to four (4) feet long. Longer columns may require hydrogen flow adjustment for proper operation. Adjustment would be required if and when precisely controlled analysis was being conducted or when the hydrogen flow was too low to keep the flame burning.

The sample air flow rate is not adjustable and is nominally 1.0 liter/minute. This flow rate should remain relatively constant. A sample flow gauge is provided on the OVA panel to monitor the sample flow rate. (Note: Panel gauge is not calibrated in L/min). When the Sample Inject Valve is in the "in" position, there may be a slight increase or decrease in sample air flow rate (0 to 15%). This change will normally not affect operation of the instrument as long as the flow rate is consistent from analysis to analysis. Basically, if the flow rate is consistent between calibration and end usage, there will be suitable precision in the measurements.

GC Analysis

1) SAMPLE INJECTION

When the Sample Injection Valve is depressed, the air in the sample loop is injected into the hydrogen stream which transports the sample through the column for separation of its components and to the flame chamber for analysis. This small volume of injected sample is qualitatively analyzed based on the retention time of the individual components of that sample while passing through the column. Quantitative analysis can then be accomplished by peak height or peak area analysis methods.

2) THE COLUMN

The column consists of tubing packed with a material which physically interacts with organic vapors and retards the passage of the vapors through the column. Since the packing material has a different attraction for each organic substance, each component in a mixture of gases will be slowed down to a different extent.

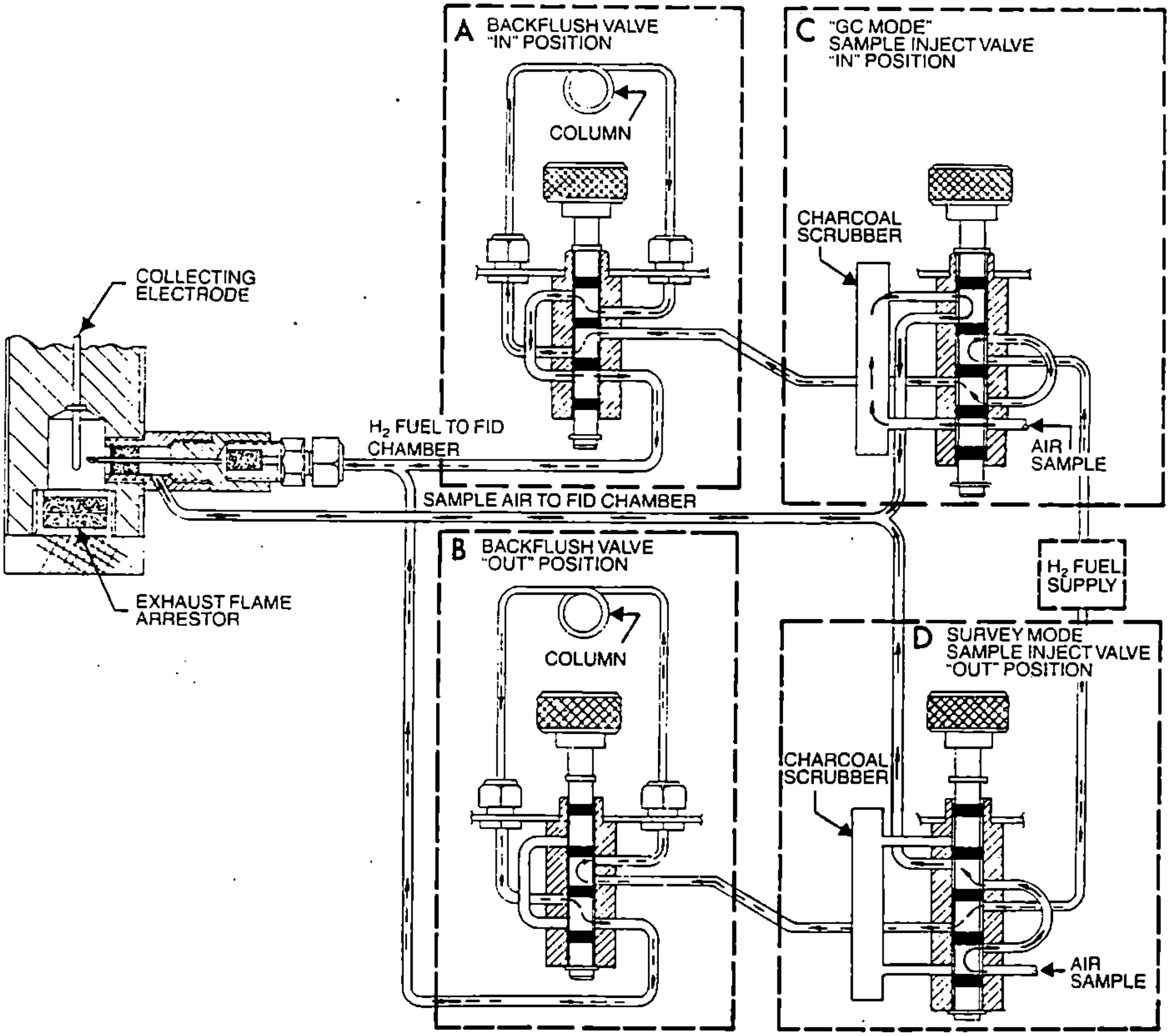


FIGURE 7
FLOW DIAGRAM - GC OPTION

The net effect is that each component elutes from the column at a different time. The components are then fed to the detector which gives a response to the meter or to an external strip chart recorder.

A portable isothermal pack (PIP) can be used for temperature control and/or isothermal analysis. This is described further under PIP kit option.

3) QUALITATIVE ANALYSIS

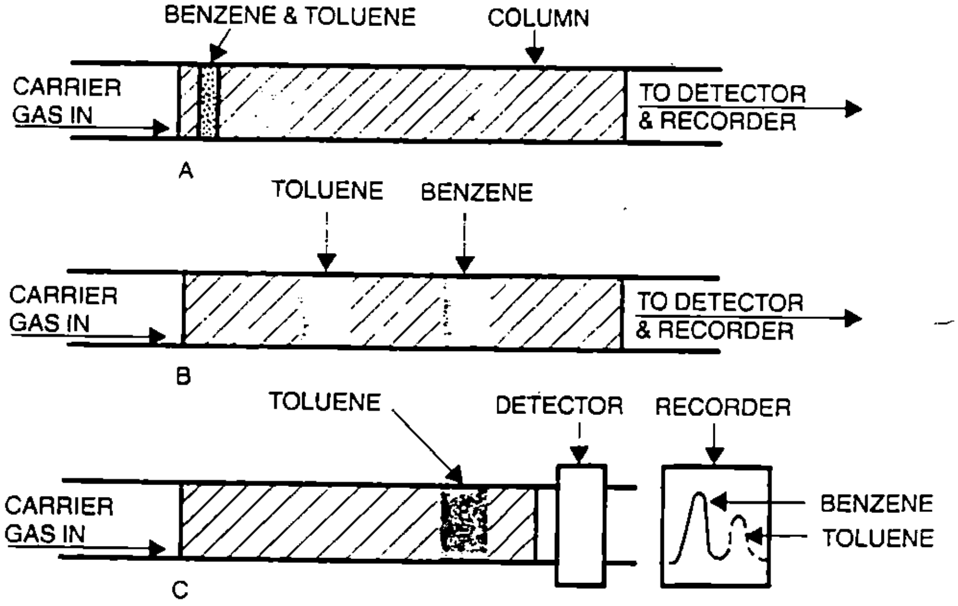
As each organic substance has a unique interaction with the column packing material, the time that the substance is retained on the column is also unique and thus characteristic of that particular substance. The "retention time" (RT) is primarily dependent on the type of packing material, the length of the column, the flow rate of the gas carrying the mixture through the column and the temperature range of the system.

When these variables are controlled, the retention times can be used to identify each of the components in a mixture. Because of these variables, it is usually necessary to establish retention times for each instrument by making a test with the pure substances of interest or to refer to established time data charts prepared in advance for that specific instrument. In those cases where retention times of the components are too close together for a good analysis, an adjustment in one or more of the operating variables will effect a sufficient difference in retention times to enable meaningful analysis.

4) QUANTITATIVE ANALYSIS

The detector response to any organic component is proportional to the quantity of material passing through the detector at a given time. For an eluted component, a plot of concentration vs. time forms a bell-shaped curve.

When using a strip chart recorder, the curve drawn on the paper is triangularly shaped and the area under the peak is related to the amount of substance being analyzed.



PICTORIAL SEPARATION OF BENZENE AND TOLUENE – "A" AT BEGINNING OF SEPARATION; "B" DURING SEPARATION; "C" BENZENE HAS ALREADY PASSED THE DETECTOR AND IS RECORDED. TOLUENE (DOTTED LINES) WILL APPEAR ON RECORDER AS IT PASSES THE DETECTOR.

FIGURE 8
TYPICAL COLUMN SEPARATION SEQUENCE

5) BACKFLUSH

The column Backflush Valve is provided to reverse the flow of the carrier gas (hydrogen) through the column. It is necessary that the column be backflushed after each individual analysis except under certain special conditions. The primary purpose of the backflush function is to clear the column of heavy compounds (with long retention times) which would contaminate the column and cause interferences to future GC analysis. The Backflush Valve has no prepositioning requirement; it is reversed from either position it was in during GC analysis. The Backflush Valve should be actuated immediately after the peak of the last compound of interest elutes. Figure 8 illustrates the function of the Backflush Valve.

In the GC system, the backflush is "to the detector". This is possible because the carrier gas and detector fuel are the same, i.e., hydrogen. It provides a convenient means of quantifying the total compounds in the backflush by simply recording the peak that elutes during the backflush operation. For field instruments, this quantitative backflush information is valuable since it provides a direct means of observing the condition of the column and seeing when the column is clean and the detector response has returned to baseline. The time required for the backflush is usually 1.2 to 1.5 times the GC analysis time.

6) SURVEY TO GC MODE

There is an inherent advantage to integrating the GC system to the basic total Organic Vapor Analyser (OVA). The OVA provides a direct reading of total organic vapors in the air being sampled, which gives the operator information about the sample being injected into the GC system. This information can be used to predict and verify the peaks that result during the GC analysis, including the backflush peak.

This feature eliminates expending valuable GC analysis time where there is no contamination of concern (comparable to taking noise measurements in quiet corners). It also enables the operator to select the most appropriate location to conduct an analysis, normally the area of highest concentration.

GC MODE OPERATING PROCEDURES

The gas chromatographic analysis mode (GC Mode) of operation can be initiated at any time during a survey by simply depressing the Sample Inject Valve. After completion of the analysis and backflush operations, the Sample Inject Valve is pulled out and the survey continued or another sample injected. Note that when the Sample Inject Valve is in the survey mode (out position) the OVA operates in the same manner as an OVA which does not incorporate the GC option.

Controls/Indicators

Refer to Figure 6.

1) Sample Inject Valve - This two (2) position valve (shown schematically in Figure 7) is used to select either Survey Mode (valve out) or GC Mode (valve in).

2) Backflush Valve - This two (2) position valve (shown schematically in Figure 7) is used to reverse the flow of hydrogen through the column to:

   a) Backflush the column for cleaning.

   b) Quantitatively measure total compounds after a selected point. Example: Separation of methane from non-methane hydrocarbons to read total non-methane hvdrocarbon level.

3) Column - Separates components of a gas mixture so that each component of the mixture elutes from the column at a different time.

4) Activated Charcoal Filter Assembly - This assembly functions only in the GC Mode (Sample Inject Valve "in") as shown schematically in Figure 7). It removes organic compounds (except methane and ethane) by absorption from the sample air supply.

Turn on Procedure

Place the Sample Inject Valve in the "out" position and put the OVA instrument in operation per "Operating Procedures" for the survey mode. NOTE: Leave the hydrogen fuel and pump "on" for three (3) to four (4) minutes before attempting ignition to allow time for hydrogen purging of the column.

Survey Mode

When using the OVA in the Survey Mode, ensure that the Sample Inject Valve remains in the full "out" position and that the Backflush Valve is either full "in" or full "out". Note that when changing from the GC Mode to the Survey Mode, the OVA output reading will continue to change until all compounds have been eluted from the GC column. Therefore, under normal field conditions, the GC column should be backflushed for clearing, which takes approximately 1.2 to 1.5 times the forward analysis time. The backflush peak may be observed returning to baseline, after which the Sample Inject Valve may be moved to the Survey Mode (out) position.

When the compound(s) being analyzed are known to be the only compound(s) present in the air sample, backflushing may be omitted.

GC Mode Operation

In normal GC analysis, a strip chart recorder is used to record the output concentration from the OVA as a function of time. This record, called a chromatogram, is utilized for interpretation of the GC data.

a) OPERATION

   1) Turn on recorder and push Sample Inject Valve "in" with a fast, positive motion. This starts the GC analysis which is automatic up to the point of backflushing. NOTE: Rapid and positive motion should be used when moving either the Sample Inject or Backflush Valves. On occasion, the flame in the FID detector may go out, which would be indicated by a sharp and continued drop of the concentration level. If this occurs, reignite the flame and continue the analysis. NOTE: A negative "air" peak typically occurs shortly after sample injection and should not be confused with flame-out.

   2) The negative air peak and various positive compound peaks indicated on the OVA readout meter and the strip chart recorder represent the chromatogram.

3) After the predetermined time for the analysis has elapsed (normally immediately after the peak of the last compound of concern), rapidly move the Backflush Valve to its alternate position (in or out). Leave the instrument in this condition until the backflush peak returns to baseline, then pull the Sample Inject Valve to the "out" position. If no backflush peak appears, pull the Sample Inject Valve out after being in the backflush condition for a period at least twice as long as the analysis time. The OVA is now in the Survey Mode and ready for survey or injection of another sample into the GC system.

b) INTERPRETATION OF RESULTS

The OVA 128 with GC option is intended for applications where there are a limited number of compounds of interest and the compounds are normally known. Under these conditions, the operator must know the retention time and peak height characteristics of the compounds under specific operating conditions. To calibrate the OVA in the GC Mode, determine, by test, the retention time and peak area (using peak height analysis) for the compounds of concern. These tests should be conducted on the column to be utilized and over the concentration and temperature range of concern. When representative characteristic data is available, such as in the Application/Technical Notes, a spot calibration check is normally all that is required.

It should be noted that under normal field conditions, the vapor concentrations vary continually as a function of time, location, and conditions. Field measurements for industrial hygene work are normally associated with a threshhold level around a pre-established concentration. Surveys for locating fugitive emission sources present a continually varying situation. Under these conditions, it is desirable to have a simple method of interpreting the GC data for on-the-spot analysis and decision making. High precision is normally not a requirement for these type analyses since the environment is continually changing. The methods presented in this section are designed to provide a means for typical field analysis. When the OVA is used under laboratory conditions, standard laboratory methodology may be used for greater precision.

Technical Discussion

The chromatogram is a chart recorder trace of the organic vapor concentration from the Organic Vapor Analyzer (OVA) as a function of time. A typical chromatogram is illustrated in Figure 9 and is a series of triangular shaped peaks originating from and returning to a fixed baseline. Qualitative interpretation of a chromatogram involves identifying a peak by analyzing the time it took for the peak to appear after initial injection [referred to as retention time (RT)] and comparing this RT to reference data. Quantitative interpretation involves analyzing the area under the peak and relating this area to calibration data of peak area versus concentration for that specific compound under the conditions present during the GC analysis.

It can be seen that interpretation of a chromatogram requires the use of calibration reference data. GC reference data is always generated empirically, i.e., through tests. Foxboro Application/Technical Notes may be used as a reference for selecting columns and interpreting chromatograms. However, simple tests must be conducted to obtain the required reference data.

a) QUALITATIVE ANALYSIS

Under a given set of operating conditions the retention time is characteristic of that particular substance and can be used to identify specific compounds. It will be necessary to calibrate retention times by making tests with the pure compounds of interest.

The retention time (RT) is defined as that period of time from injection until the time of maximum detector response for each substance. Retention time is measured from the time of sample injection to the time the apex of the triangle shaped curve is obtained on the strip chart recorder. (See Figure 9). The strip chart recorder operates on a clock mechanism such that the distance along the baseline is proportional to time. While retention times are characteristic for each compound, it is possible that two materials could have the same retention times. Thus, if there is any question as to the identity of the vapor, it may be necessary to verify identification by retention times on different columns.

Use of a longer column will increase the retention times of those components it is capable of separating. The time between peaks will also be increased. This is especially useful if a component comes through too fast or if desired peaks are so close that they overlap.

b) COLUMN SELECTION

Two columns are supplied with the instrument. These are general purpose columns which are useful in a wide variety of applications. If they do not achieve separations for a particular application, it may be necessary to select other packing materials or longer columns. Foxboro will assist in this selection or prepare a custom column if necessary.

If columns are made by the user or purchased from other sources, ensure that the packing density does not create too large a pressure drop. A large pressure drop can result in flame-out problems.

c) TEMPERATURE EFFECT ON RETENTION TIME

An increase in temperature will decrease column retention time (RT) and vice versa. Normally retention time (RT), as a function of temperature, changes linearly over the range of 0 to 40°C. For complex qualitative analysis, a calibration plot of RT versus temperature will be required. In typical usage, such as inside a factory, the effect of temperature can be compensated for during chromatogram interpretation. A single component tracer compound can be sampled at any time to provide a "key" for other compound identification.

d) CARRIER GAS FLOW RATE AFFECT ON RETENTION TIME

An increase in carrier gas flow rate will decrease retention time. For reproducible data, the carrier gas (hydrogen) flow rate must be recorded in association with a chromatogram. Primary control of the hydrogen flow rate is accomplished in the OVA by regulating the hydrogen pressure across a capillary tube. The hydrogen flow rate is also affected by the restriction of the GC column but most columns have a limited effect. The hydrogen flow rate is factory set at 12 cm /minute with a typical 24 inch column.

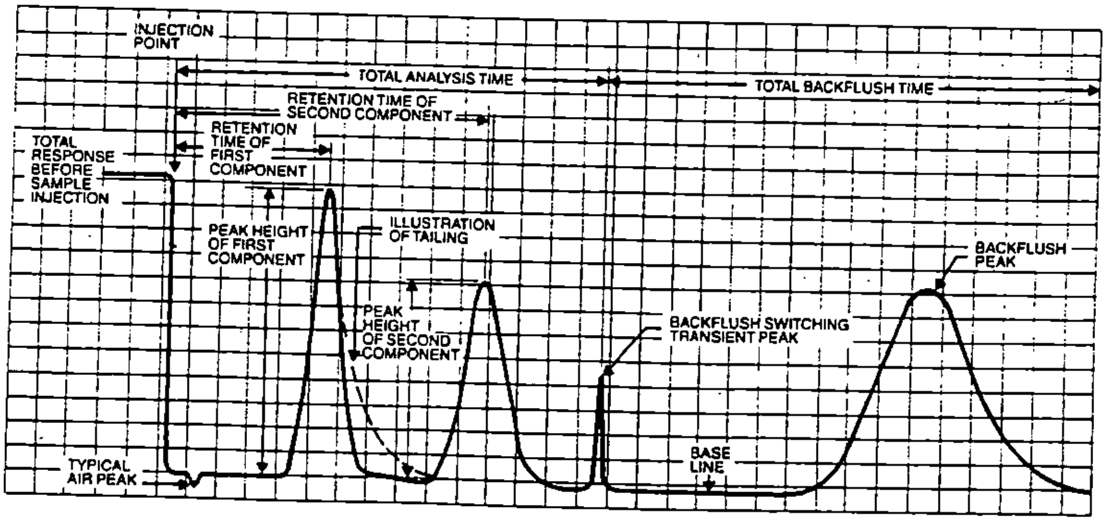


FIGURE 9
TYPICAL CHROMATOGRAM

e) QUANTITATIVE ANALYSIS

In general, the more triangularly symmetrical the peak, the better the peak height analysis capability. However, many GC peaks have "tailing" as illustrated in Figure 9. Peak height calibration is an acceptable method for quantitative analysis as long as the area under the tail is small compared with the total peak area. If severe tailing occurs, empirical calibration data generated through tests may be required to plot the peak height versus the concentration curve.

Only peak height analysis will be discussed in this manual. The method involves injecting a known concentration of the compound and recording the peak height under the test conditions. Peak height characteristics can be established for various columns and various temperatures. Normally, both retention time and peak height characteristics will be measured.

When peak area measurements are desired, the areas may be measured using an integrator on the OVA output signal. Other manual methods may also be used, such as counting squares, weighing curves or simple triangulation. When the GC peaks have good symmentry, triangulation (area equals 1/2 base x height) is a convenient method.

Calibration Data

When conducting tests to obtain GC calibration data, the following information should be recorded.

a) Column - description and serial number as applicable.
b) Temperature - column temperature, normally room ambient.
c) Chart speed - distance/unit time.
d) Carrier flow rate - hydrogen flow rate through the column (cm /min).
e) Sample concentration - ppm for each compound,
f) Sample volume - OVA by serial number or typically 0.25 cm for standard value.
g) Recorder scaling - ppm per unit deflection.
h) Range - range of OVA being used, i.e. X1, X10, X100.
i) OVA serial number.

To obtain a calibration point, inject a known concentration sample into the GC system and record the resulting chromatogram peak. The retention time for the peak may be scaled from the record or timed with a stop watch. The peak height may be scaled from the record or the OVA readout meter may be observed during the elution of the peak. Figure 10B presents the format of a chart which may be used to record calibration data. Experience has indicated that the peak height response of a compound is linear within the concentration range of 0 to 160 ppm. Therefore, a single calibration point, preferable around the concentration of concern, is normally all that is required to plot peak height response in ppm as a function of compound concentration. Data for other compounds on the same column may also be plotted along with their associated retention times, percent relative response in the total organic Survey Mode, TLV, etc. It is recommended that copies of the actual chromatograms be kept with the charts for observing the peak shapes, peak interferences, etc. It should be noted that a chromatogram can be utilized like a fingerprint for compound identification or peak height and shape comparison. Transparent overlays are an aid in chromatogram analysis.

When temperature variations are anticipated, data should be taken at several points and recorded on the chart as a new curve or as a relative change as a function of temperature as illustrated in Figure 10B.

Preparing and using the calibration chart is very straightforward. As an example, once the elution sequence of a group of compounds is determined, a mixture of 100 ppm of each can be prepared and run on the GC for chart data. The retention time of each compound and the peak height of each can be read directly from the chromatogram and the data put on the chart. If temperature data is to be taken, additional chromatograms may be run with the same sample and the RT and peak height as a function of temperature.

When complex mixtures such as gasoline are analyzed, it may be desirable to keep the record of the backflush peak for future reference and peak area comparison. It is also recommended that the total organic vapor concentration reading on the OVA be recorded for each calibration sample used. This reading is used for arriving at relative response numbers and as a check on sample preparation precision.

Routine Maintenance

a) COLUMN

Any column can be contaminated with compounds having long retention times. This will result in high background readings. This condition can be checked by installing a new column or a blank column (tubing only). If this reduces the background reading, the contaminated column should be baked at 100°C (212°F) for three (3) to four (4) hours in a drying oven while passing nitrogen through the column. Higher temperatures may permanently damage the column packing.

When installing any column, avoid touching the ends, as this may cause contamination. Also, ensure that the fittings are tight to avoid hydrogen leakage.

IMPORTANT: The following simple test may be run to determine whether the GC column is contaminated. While in a clean ambient air background, place the Sample Inject Valve in the "in" (GC Mode) position. Observe the background reading on the meter or recorder. After one (1) to two (2) minutes, change the position of the Backflush Valve and again observe the background reading. If the background reading went down and then started to increase in one to two minutes, the column is probably contaminated and needs to be cleaned. Note that if hydrogen flows into one end of the column for a period of time, the contamination is pushed into the column.

Then when the hydrogen flow is reversed, the exhaust end of the column will be clean until the contamination is again pushed through. Remember that to clean a column the purge gas must be run through the column in one direction until all contamination is removed. NOTE: Contaminated columns can be avoided by backflushing the column after every analysis.

b) CHARCOAL FILTER ASSEMBLY

After repeated use, the Charcoal Filter Assembly will become saturated. Periodically, the operator should check the effectiveness of the activated charcoal.

This can easily be done by operating the unit with the Sample Injection Valve "in" and passing the probe near a concentrated sample of the compound being analyzed. The readout should remain nearly steady (should not rise more than 0 to 2 parts per million (ppm)). If rise is more than 2 ppm, replace the old charcoal with new activated charcoal. Care should be taken to completely fill the tube to prevent a path for sample to bypass the charcoal. The life of the charcoal depends on the time (length) of exposure and the concentration level during that exposure. When changing charcoal, be sure that any fine charcoal dust is removed from the assembly.

Another test of the charcoal filter is to note the background reading with the Sample Inject Valve "out" and then note the reading with the valve "in". The level should never be higher when the valve is in the "in" position and the charcoal filter is in the air line. If the reading with the valve in the "in" position is higher, the charcoal filter is probably contaminated and acting like a contamination emitter.

TroubleShooting

Table 2 presents recommended field trouble shooting procedures which are associated with the GC system. These procedures are in addition to those found in the basic OVA section of the manual.

TABLE 2

PROBLEM	TROUBLE SHOOTING PROCEDURE	REMEDY
1) Low sample flow rate on flow indicator.	a) Check Teflon tubing on valve assemblv for kinks, etc.	Straighten or replace teflon tubing.
	b) Check flow rate with valve in down position.	Check for over restriction of charcoal filter.
2) Hydrogen flame will not light.	a) Check column connections on top of unit to make sure thev are tight.	Tighten fittings.
	b) Check column for sharp bends or kinks. (Hydrogen flows through this column at all times and a sharp bend will compact packing too tightlv for proper hvdrogen flow).	Replace column.
	c) Check charcoal filter fittings to make sure they are tight.	Tighten fittings.
	d) Check hvdrogen flow rate from the column.	Adjust hydrogen pressure to obtain 12 cm^3/min flow rate.
	e) Check that the Inject and Backflush Valves are both completely in or out. A partially activated valve will block the hydrogen and air flow paths.	Ensure both valves are either completely in or out.
	f) If a new column was installed prior to problem identification, check for proper hvdrogen flow rate through the column (should be approximatelv 12 cm /min).	Increase hydrogen pressure to obtain proper hydrogen flow rate or if column is excessively restrictive, replace or repack the column.
3) Ambient background reading in clean environment is too high.	a) Check for contamination in charcoal filter assemblv. This can be detected if ambient reading increases when going in to the chromatographic mode.	Replace activated charcoal in charcoal filter assembly.
	b) Check for contamination in column.	Replace or clean column.
	c) Check for contamination in column valve assembly.	Remove valve stems and wipe with clean lint-free cloth. Heat valve assembly during operation to vaporize and remove contaminants.
4) Flame-out when operating either valve.	a) Ensure valves are being operated with a quick, positive motion.	Operate valve with a positive motion.

TABLE 2

PROBLEM	TROUBLE SHOOTING PROCEDURE	REMEDY
	b) Either hydrogen or air may be leaking around one or more of the valve quad rings. Assess by tests and "O" ring inspection.	Remove stems and lightly coat with silicone grease, only on contact surface of the "O" ring. Wipe off excess (do not remove quad rings).
	c) Damaged or worn quad rings causing leak.	Replace quad rings and grease as above.
5) Excessive peak tailing	a) Change or clean GC; see if problem disappears.	Ensure columns are clean prior to use. If one of the same type of column tails are worse than others, repack the column or discard.
	b) Inspect GC valves for excessive silicone grease or contamination.	Excessive lubricant or foreign matter in the valve assembly can cause excessive tailing. Clean valve assemblies and lightly relubricate as required. Lubricant should be put only on the outside contact surface of the "O" ring. Do not get grease into the "O" ring grooves.

Recommended Spares

The following spare parts and supplies are recommended to support the GC system and recorder. These are an addition to the spare parts list for the basic OVA described in the "OVA MAINTENANCE" section.

ITEM DESCRIPTION	PART NO.
1) Quad Rings	510496-1 (10/pkg.)
2) Tubing, .148 in ID .020 wall	12942
3) Tubing, Teflon .120 in ID .030 wall	12941
4) Activated Charcoal	CSC-004
5) "O" Ring for Charcoal Scrubber	U0118CE
6) Chart Paper (linear)	CSC-008 (6/rls/pkg)

ACCESSORIES

Recorder Accessory

A portable Strip Chart Recorder is available for use with the OVA (reference Figure 11). The recorder is powered from the OVA battery pack and the output can be scaled to match the OVA readout meter, thereby providing a permanent record for subsequent analysis or reference. P/N 510445-4 is FM certified intrinsically safe. P/N 510445-6 is BASEEFA certified.

The recorder can be used with the OVA to provide a long term monitoring profile of total hydrocarbon or can be used with the Gas Chromatograph Option to provide a chromatogram.

Features

The recorder prints dry (no ink) on pressure sensitive chart paper. The recorder is equipped with two gain ranges and an electronic zero adjustment. The HIGH gain position is normally used to provide a means of scale expansion.

Controls and Connections

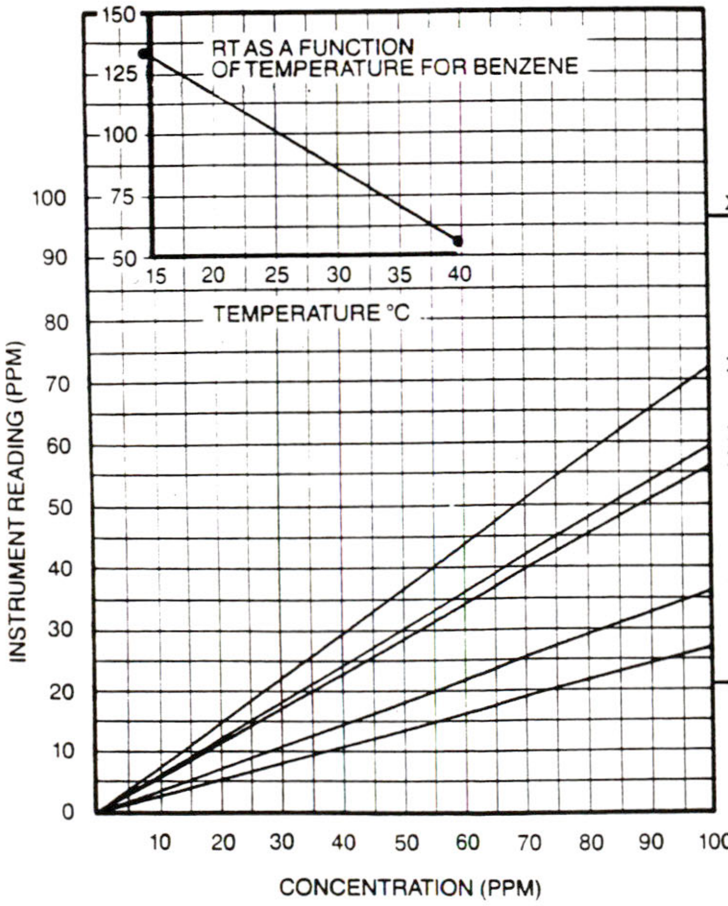
Described below are the functions of recorder controls and connectors.

1) HIGH-LOW Switch - This switch, located on the right hand side of the recorder, provides 2 ranges. The LOW range is set for the same full scale reading as the OVA readout meter. The HIGH range can be set to give an increased sensitivity to the recorder without effecting the OVA calibration.

2) ZERO ADJUST Knob - This potentiometer, located on the right hand side of the recorder, permits "nulling" of the background reading on the recorder without affecting the calibration of the OVA displayed on the OVA readout. In the full clockwise position, the recorder will display the same reading as the OVA meter. Counterclockwise rotation will reduce the reading on the recorder.

3) POWER CONNECTOR - This 126 series, 5 pin connector provides power and signal to the recorder, as follows:

PIN	FUNCTION
B	Input Signal
E	pos. 12VDC input
H	Ground

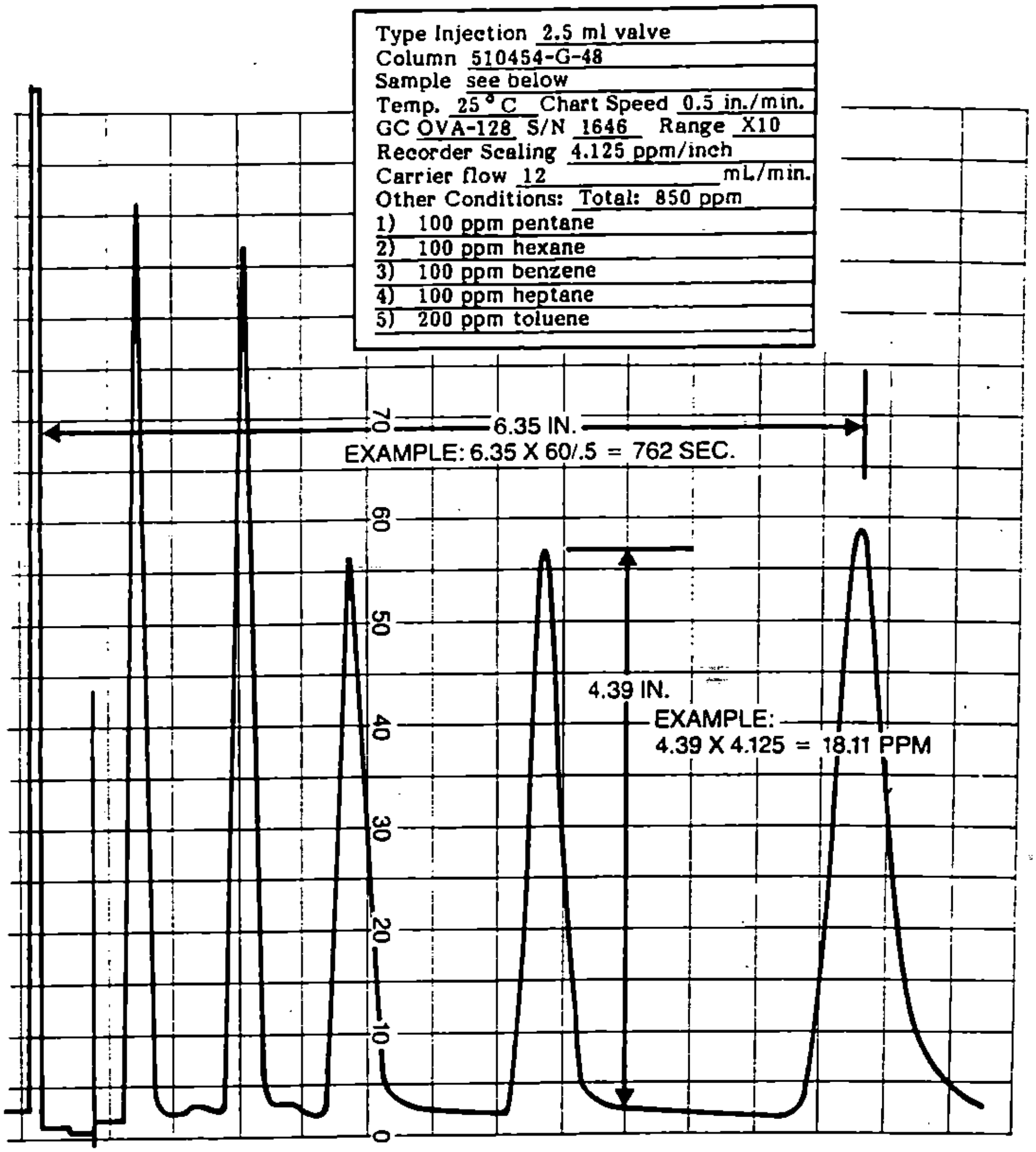


Column G-48 Chromatograph OVA-128
Temperature 25°C Carrier Flow 12 mL/sec.
Injection: Volume 2.5 mL Type valve

X	Compound	R.R (%)	RT (sec)	TLV (ppm)
X4	Heptane	80	471	400
X2	Pentane	100	90	1000
X2	Hexane	90	198	100
X2	Benzene	150	291	1
X3	Toluene	120	762	200

NOTE:
1. Concentration used for test is 100 ppm or as indicated.
2. X = Concentration multiplier

FIGURE 10A
CALIBRATION CHART



Type Injection 2.5 ml valve
Column 510454-G-48
Sample see below
Temp. 25 °C Chart Speed 0.5 in./min.
GC OVA-128 S/N 1646 Range X10
Recorder Scaling 4.125 ppm/inch
Carrier flow 12 mL/min.
Other Conditions: Total: 850 ppm
1) 100 ppm pentane
2) 100 ppm hexane
3) 100 ppm benzene
4) 100 ppm heptane
5) 200 ppm toluene

6.35 IN.
EXAMPLE: 6.35 X 60/.5 = 762 SEC.

4.39 IN.
EXAMPLE:
4.39 X 4.125 = 18.11 PPM

FIGURE 10B
CHROMATOGRAM

Calibration

Electronic and mechanical adjustments, other than the operational adjustments on the side panel, are provided to calibrate and align the recorder. (See Figure 11).

MECHANICAL ZERO ADJUSTMENT

A) Snap out the front panel nameplate using a small blade screwdriver in the left hand slot) for access to mechanical zero adjust screw, place HIGH-LOW Switch in OFF position.

B) Unscrew knurled fastener at top of front panel to open recorder. Pull down plastic chassis latch on right side to release sticker bar tension on paper and adjust mechanical zero as required. Replace nameplate, chassis latch and resecure front panel.

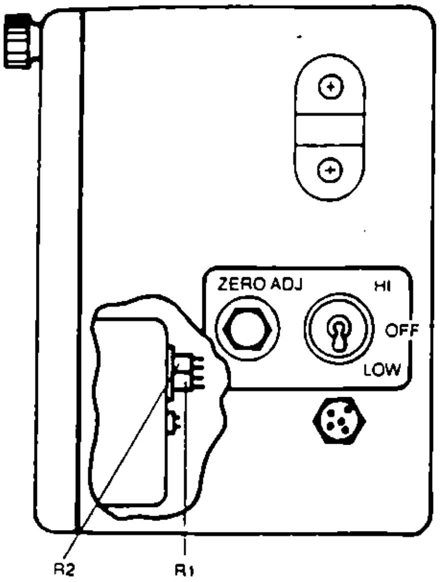


FIGURE 11
RECORDER CONTROLS AND ADJUSTMENTS

GAIN ADJUSTMENT

Separate adjustments are provided for the HIGH and LOW ranges on the recorder. (Refer to Figure 11 for location).

a) Connect recorder to OVA and adjust OVA for full scale reading on readout (about 5 VDC).
b) Loosen knurled fastener on upper left of the front panel and pull front panel down.
c) Place HIGH-LOW Switch in LOW and adjust R1 until recorder prints full scale.
d) Place HIGH-LOW Switch in HIGH and adjust OVA to read the desired full scale with front panel CALIBRATE ADJUST Knob, typically half scale on the readout. Adjust R2 until recorder reads full scale. NOTE: Full scale adjustment of the recorder for 1/2 scale on the OVA gives a gain increase of two (2) in the height of the peak on the chromatograms. This is the factory set point for the HIGH gain range; however, other points can be set as desired with a gain of three being the maximum obtainable without amplifier loading.

Maintenance and Routine Operations

Refer to the manufacturer's (Gulton) manual on the recorder which is enclosed with each recorder when shipped.

Changing Chart Speeds

The recorder is equipped with a 16 RPM motor which gives a writing speed of four (4) strikes per second. The chart advance speed is determined by the gear train assembly number used. The inches per hour for each gear train is given in the table on page 9 of the Gulton recorder manual. Refer to the bottom line of the chart adjacent to drive motor 16 and note for example that a number 1 gear train has a chart speed of 8"/hour.

a) To change the paper speed, open the recorder, remove gear box spring (on left side), move gear box in direction of arrow on its case and lift out from top. Do not force out from bottom. Insert new gear, bottom first, slide into position against arrow direction. Replace gear box spring.

Activated Charcoal Filter Accessory

The Activated Charcoal Filter Assembly is an accessory which can be installed on the OVA Readout Assembly or attached at the end of the telescoping probe. The filter is typically filled with activated charcoal which acts as an absorbent and effectively filters out organic vapors other than methane or ethane.

A screw cap on the probe end is removed for refilling the filter with activated charcoal or other filtering media.

Applications of the filter include:

1) Obtaining a clean air sample for zero baseline check and adjustment.

2) Running "blank" chromatograms to assess instrument contamination.

3) Rapid screening of methane and non-methane organic vapors.

4) Selective screening for natural gas surveys.

5) As a moisture filter when filled with a desiccant such as silica gel.

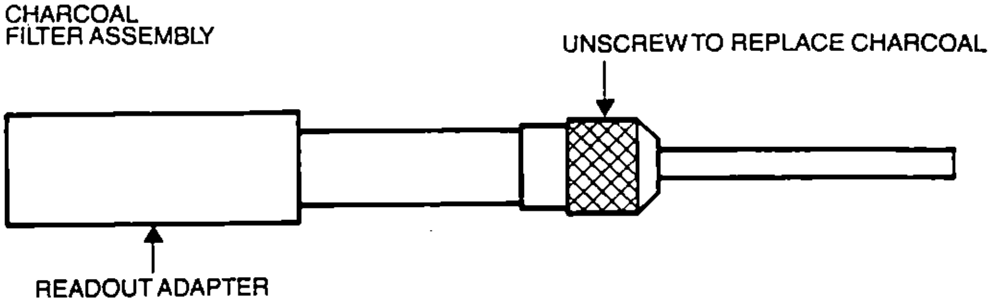


FIGURE 12
ACTIVATED CHARCOAL FILTER ASSEMBLY

A press fit adapter on the back of the filter assembly is removed when installing the unit on the telescoping probe. When replacing the cap end after refilling, one wrap of ¼ inch teflon tape should be used to seal the threads.

The life of the filter will depend on the time in use and the concentrations of the compounds being filtered. Under typical industrial air monitoring conditions, the filter will last for many days of continuous sampling. See Figure 12.

Sample Dilutor Accessory

An adjustable sample dilutor assembly, P/N 511745-1 is an accessory. The dilutor is supplied with a 10:1 dilution orifice as standard. Orifices for 25:1, P/N 511770-2, and 50:1, P/N 511770-3, dilution are also available.

In operation, the dilutor is attached to the end of the telescoping probe or connected by external tubing to the input fitting of the OVA side pack. Dilution of the air being monitored is accomplished by stream splitting through the use of a needle valve on the sample input. An activated charcoal scrubber is inserted in the main air supply line to the OVA and scrubs the air of organic vapors. It also creates a slight vacuum at its output side of the scrubber and the vacuum at this point draws the sample air through the needle valve where it mixes with the main air supply going to the OVA detector.

The dilution valve provides a means of sampling vapor levels above the lower explosive level (LEL) and in oxygen deficient atmospheres. These conditions can occur in normal leak or source survey as the operator gets close to the leak or vapor source or in monitoring various manufacturing or material handling processes. Approximately 14% oxygen is required to sustain operation of the FID in the OVA.

Setting Dilution Rate

Prepare a sample in a bag at a high level, typically 1,000 to 5,000 ppm. Any suitable gas can be used, such as butane from a cigarette lighter; however, a compound similar to those to be measured provides greater accuracy. The actual concentration of the gas does not have to be known, since the dilution rate is simply a relative level.

Obtain an OVA reading on the vapor sample with the dilution valve removed. Then install the valve, loosen the jam nut and turn the needle valve until the meter reading corresponds to the original reading divided by the dilution factor desired. Retighten the jam nut.

It should be noted that when the dilution valve is used for natural gas leak survey and pinpointing, the charcoal filter will not remove the methane from the dilution air supply. Care should be taken so that natural gas is not allowed to enter the main air inlet. (See Figure 13.)

OVA Septum Adapter Accessory

A Septum Adapter, P/N 510645-1, is available for direct on-line sample injection to the GC column inlet. The Septum Adapter mounts directly on the OVA front panel and sample injections from .025 to 2.5 cm may be made using a gas tight syringe.

This provides a range of sensitivity of approximately 10% to 1000% of the OVA standard valve, which has a sample loop volume of approximately 0.25 cm . Syringe injection can cause flame-out, however, the OVA may be reignited after the injection is made. The air in the sample must elute from the column before reignition. The time for the air peak to elute is a function of the column length and the volume of the sample injected. For example, a 1 cm sample into a 12" column will require approximately 5 seconds; and, a 2.5 cm sample into a 48" column will require approximately 20 seconds.

The Septum Adapter also provides a means whereby samples from oxygen deficient atmospheres or process streams can be injected directly into the chromatograph. Headspace analysis may also be accomplished using the Septum Adapter and a syringe.

OVA Portable Isothermal Pack (PIP) Accessory

A column can separate an exceptionally wide variety of components if the separations are made at different temperature ranges. In addition, peak heights and retention times can vary with column temperature. The PIP option was developed to control column temperature, without affecting the analyzer's intrinsic safety specifications and without compromising the analyzer's portability.

When the Septum Adapter is installed on the OVA, the normal GC sample valve may still be used alternatively with the syringe injection. In addition to variable sample size and sensitivity, syringe injections will normally provide greater symmetry and reduce tailing of chromatogram peaks as compared with the standard valve injection.

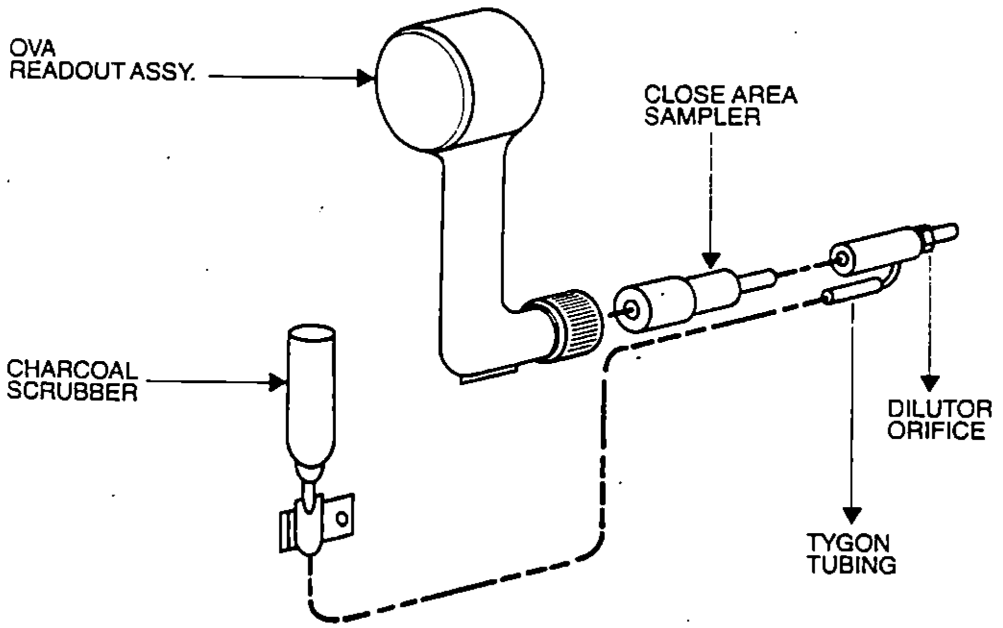


FIGURE 13
OVA SAMPLE DILUTOR

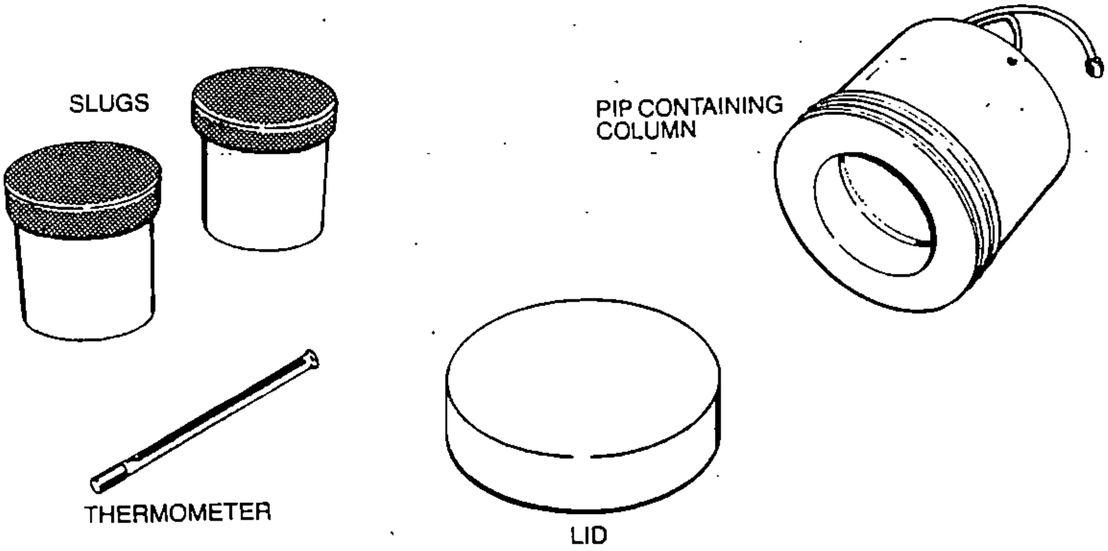


FIGURE 14
PORTABLE ISOTHERMAL PACK

PIP Components & Spare Parts

511800-1	PIP Kit
511805-1	PIP Assembly (specify column length and packing material)
511810-1	40°C Slug (phase-change material)
511830-1	Seeder for 40°C Slug
511815-1	Aluminum Slug
511820-1	Empty Bottles (package of six)
511825-1	Insulating Cover
511826-1	Thermometer

PIP columns can be prepared with any standard column packing material. A temperature control slug is inserted into the PIP slug cavity which has exterior foam insulation. For field operation in extreme ambient temperatures, an additional sheepskin jacket can be installed. The period of temperature control depends upon the temperature difference between ambient and the slug. For a 0°C ice pack and ambient temperature of 27°C, a control period of approximately 10 hours is typical. Additional information on the PIP system will be found in Foxboro document TI611-105.

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Chromosorb is a trademark of Johns-Manville.

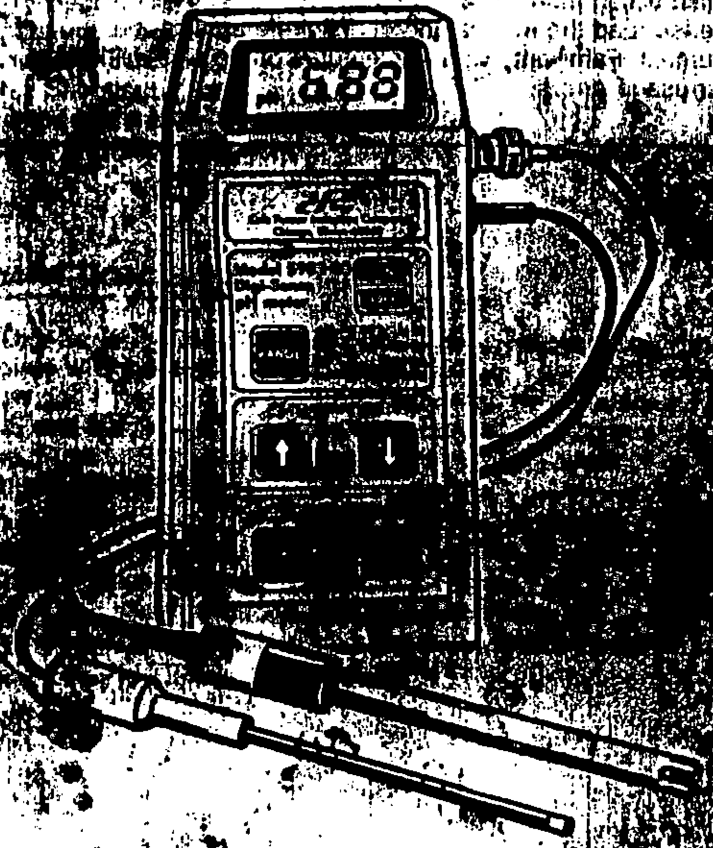
Carbowax is a trademark of Union Carbide Corporation.

Poropak is a trademark of Waters Associates.

Porasil is a trademark of Waters Associates.

Operating Instructions

Cole-Parmer Model 5985-80
Digi-Sense® pH Meter



Cole-Parmer Instrument Company
7425 N. Oak Park Avenue, Chicago, Illinois 60648
1-312-647-7600 or toll-free 1-800-323-4340 (except Alaska)
Telex [illegible]-9405 [illegible] 647-9660

CAUTION

Use the Digi-Sense® 5985-80 pH meter only as directed in this instruction manual.

Please read the manual thoroughly before using the instrument. Familiarity with the features of the unit will lead to fewer usage errors.

Table of Contents

General Description

The Digi-Sense® 5985-80 is a portable, microprocessor-based pH meter complete with pH electrode and an ATC probe. The pH, °C and mV ranges and other controls are easily accessed by a membrane keyboard, and the unit automatically compensates for temperature variations. Pushbutton calibration is stored in memory, even after the unit has been shut off.

Each meter includes a silver-chloride type pH electrode with BNC connector and sealed epoxy body. An ATC probe with miniature phone jack, stainless steel sheath (5mm OD x 109mm L) and plastic handle is also included. Both electrode and ATC probe have a one-meter cable. Carrying case is soft black vinyl; a battery is also included.

Digi-Sense®—Reg TM Cole-Parmer Instrument Company

Specifications

Ranges:	pH: 0.00 to 14.00 °C: 0 to 100° mV: ±399 and ±1999
Resolution:	pH: 0.01 °C: 0.1 mV: 0.1
Accuracy:	pH: ±0.01 (±0.05%), ±1 digit FS °C: ±0.5° FS mV: ±399: ±0.20 (±0.05%), ±1 digit ±1999: ±1.0 mV (0.05%), ±1 digit FS
Calibration:	Automatic with automatic recognition of the buffer
Temp compensation:	Automatic, from 0 to +100°C Manual, from 0 to +100°C
Battery:	9-volt (included); battery life is approximately 40 hours
Memory life:	Approximately 1 year
Display:	3½ digit with mode (pH, °C or mV) and low battery indication
Dimensions:	7.20" x 3.20" x 1.88" (180x80x47mm)
Weight:	1 lb, 5 oz (600 g); with pH electrode, ATC probe and carrying case

Accessories (optional):

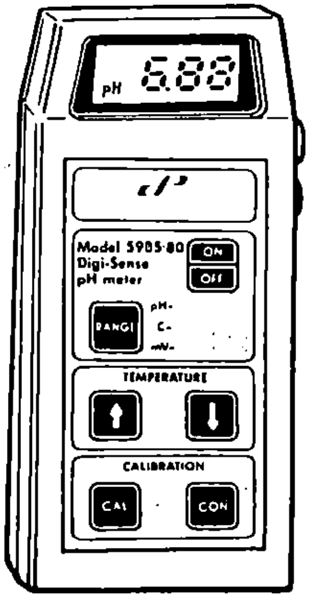
5992-20 Replacement electrode and 5985-81 ATC probe, plus KCl solution and a variety of pH buffers and testers. Call Cole-Parmer, toll-free 1-800-323-4340, for technical assistance and ordering information.

Before Operation

For a fast response, the tip of the electrode should always be kept moist. A rubber cap is supplied to cover the tip end of the electrode. Before use, remove this cap. If the cap has been left off and the tip is dry, dip it in KCl solution for 30 minutes or tap water for two hours. When the electrode is not in use, replace this cap filled with KCl solution. If this solution is not available, use tap water.

NOTE: Do not use distilled water, under any circumstances.

Figure [illegible] —Front Panel



Front Panel Controls

The function keys on the front panel are as follows:

ON-OFF:	Switches the unit on and off
RANGE:	Selects pH, °C or mV ranges
TEMPERATURE:	Use these keys only when ATC probe is disconnected
CALIBRATION:	The key CAL is used to start the pH calibration procedure and the key CON to confirm the calibration data

pH Calibration

A new instrument must be calibrated before use in pH measurements. Recalibration will be necessary after the battery is replaced, of if the pH electrode or the ATC probe are replaced.

The instrument can be calibrated with a pH 7.00 and either pH 4.01 or pH 10.00 standard technical buffers. If you are measuring between 0 and 7 pH (acid to neutral), use pH 7.00 and pH 4.01 buffers. If your measurements will be between 7 and 14 pH (neutral to base), use pH 7.00 and pH 10.00 buffers.

NOTE: When using the ATC probe, the meter automatically compensates for changes in buffer temperature. (Ideally, buffer solutions should be kept at 25°C for calibration.) When not using the ATC probe, make sure that the temperature is set manually to reflect the temperature of the solution.

To calibrate for pH:

1. Place the electrode in a pH 7.00 buffer solution. Wait approximately 30 seconds for the sensor to stabilize, and then press CAL.
2. If the electrode recognizes the pH 7.00 solution, the exact value will appear on the display in accordance with the pH and temperature charts (i.e., pH 7.01 at 25°C). If not, the symbol "E4" will be displayed—see "Error Code Guide".
3. Wait 30 seconds and then push CON again to accept the buffer value.

   NOTE: The first calibration is now finished. "E5" will appear on the display at this point, indicating that the instrument has entered the slope calibration mode. If slope calibration is to be performed, follow the instructions below. Otherwise, switch the instrument off.
4. Take the electrode out of the pH 7.00 solution, rinse it with distilled water and dip it into the pH 4.01 or pH 10.01 solution. "E5" will disappear when the electrode is placed in the second buffer, and the value of the chosen buffer will then appear on the display. Wait 30 seconds and press CON. The instrument is now calibrated, and will remain calibrated even after the unit is shut off.

Operation

1. Connect the pH electrode and the ATC probe (if ATC probe is to be used).
2. Push the ON/OFF switch to turn the unit on.
3. Then push RANGE until the display indicates the desired mode.

   For pH measurement: When the instrument is switched on, it is already in the pH mode. Dip the pH electrode and the temperature sensor in the solution. The pH value displayed will stabilize after a few seconds.

   For temperature measurement: Press the key RANGE until the symbol "°C" appears on the display. Dip the temperature sensor in the solution and wait until the reading stabilizes (about 30 seconds).

   For mV measurement: Press the key RANGE until the symbol "mV" appears on the display. For values within ±400 mV, tenths of mV are also displayed; for values outside 400 mV, only the mV is displayed. Note that the change in scale is automatic.
4. When measurements have been taken, press the ON/OFF switch again to turn the unit off.

NOTE: When using the instrument, press the keys firmly and hold for half second. Otherwise the microprocessor will reduce the meter's response time to conserve the battery.

Temperature Compensation

When the temperature probe is plugged in, temperature compensation is automatic and goes from 0 to 100°C.

Without the ATC probe, temperature compensation will be set manually using the arrow keys to increase and decrease the value. ("Up" arrow increases the temperature; "down" arrow decreases temperature.) When the temperature probe is not plugged in, the display will show a blinking °C symbol while the meter is in the °C mode.

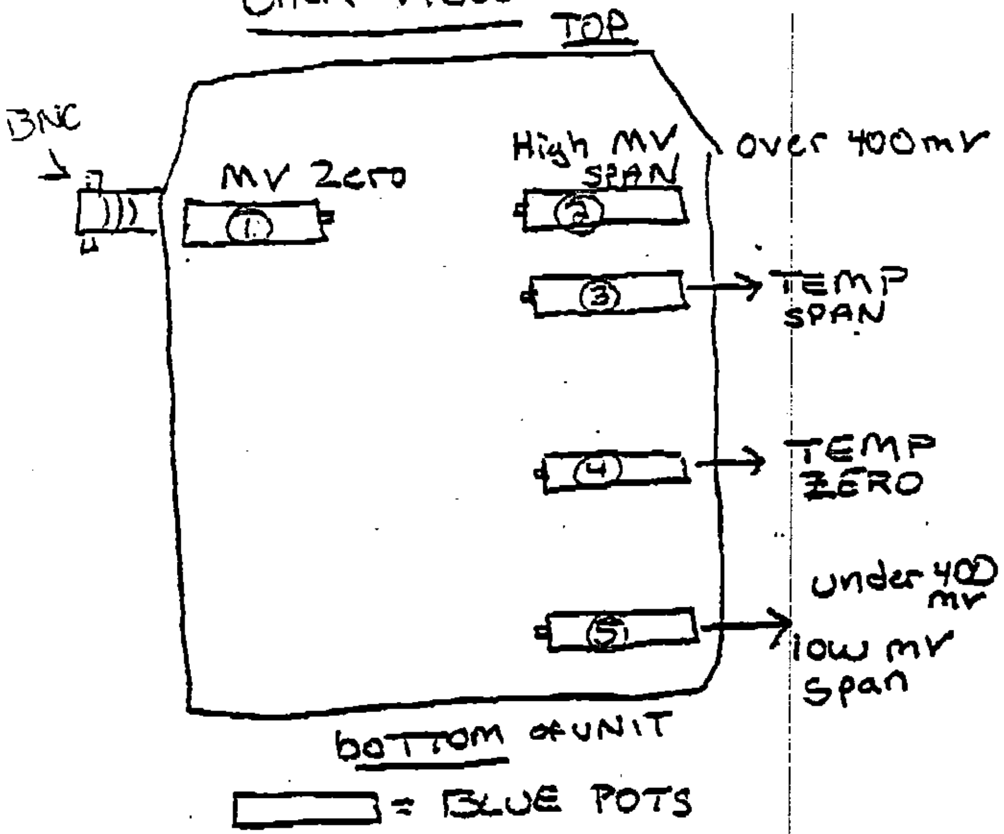
From the desk of . . . BRIAN BARNETT

FYI

UPDATE ON 05985-80

HERE IS THE SCHEMATIC FOR THE POTS AND THE CORRECT LABELING FOR THE NEW CIRCUIT BOARD

BACK VIEW



ANY QUESTIONS PLEASE SEE ME.

Cole-Parmer Instrument Company
7425 North Oak Park Avenue, Chicago, Illinois 60648
708/647-7600

Error Code Guide

Five error codes are programmed into the pH meter to indicate error messages. These are as follows:

Display reads...	Problem is...	Possible solutions
"E 1"	Out of range on pH scale	a. Make sure that the sample falls within the pH range. b. Check meter for proper calibration. c. Check pH electrode. If empty, refill it; if defective, replace it.
"E 2"	Out of range in °C	a. Make sure temperature is within the unit's range.
"E 3"	Out of range in mV	a. Make sure signal is within the ±1999 range.
"E 4"	Offset calibration with wrong buffer	a. Make sure you are using the pH 7.00 buffer. b. Use fresh buffer solution.
"E 5"	Slope calibration with wrong buffer	a. Make sure you are using a pH 4.01 or 10.00 buffer. b. Use fresh buffer solution.

Battery Replacement

The instrument uses a 9-volt battery with a life of approximately 40 hours. From the moment that the far left hand side decimal point is lit, the battery has 3 more hours of life.

To change the battery, unscrew the back cover of the unit. Replace the old battery and close the case.

NOTE: The meter will need to be recalibrated after changing the battery.

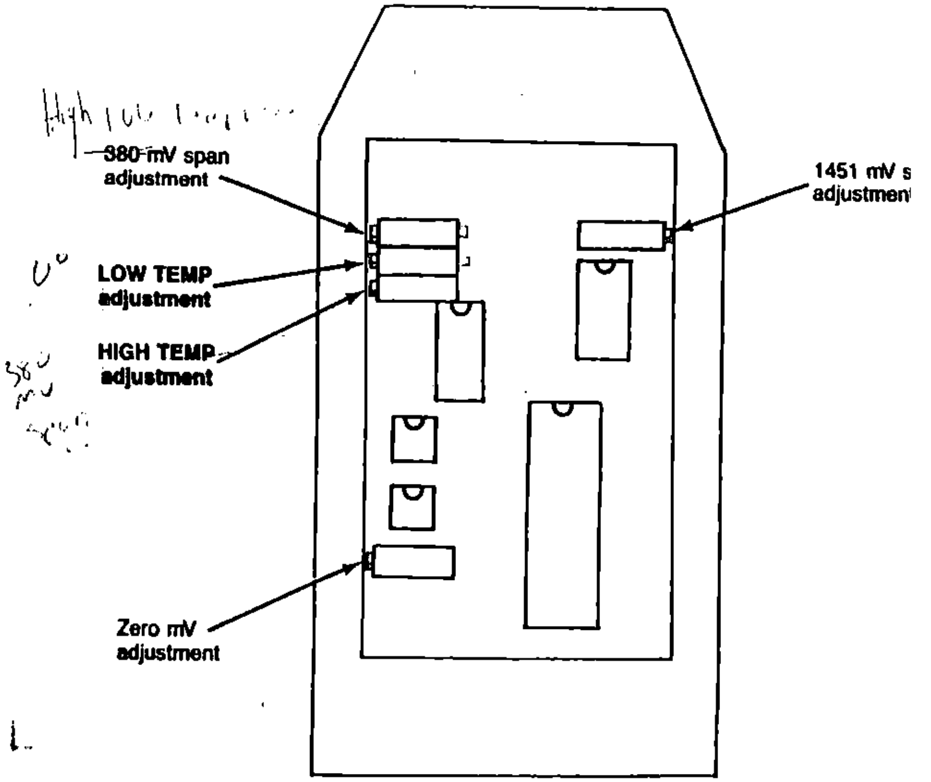
Temperature Sensor Calibration

The pH meter has been calibrated with the ATC probe supplied and is ready for measurements. If, for any reason this temperature sensor is replaced, follow this procedure for recalibration:

1. Remove the back cover and locate the two temperature trimpots (adjustments). Refer to the Figure 2 as needed.
2. Dip the ATC probe for at least 3 minutes into an ice bath (about 0°C, ±1°C), and then adjust the LOW TEMP trimpot until the display reads the same temperature measured by a second thermometer.
3. Dip the ATC probe for a few minutes in 50°C water and adjust the HIGH TEMP trimpot as explained above.

NOTE: When calibrating ATC probe, the pH electrode should not be connected to the unit or immersed with the ATC probe.

Figure 2—Internal Calibration Adjustments



Cleaning the Electrodes

The sensitive part of the electrode should always be kept clean. Rinse the electrode with distilled water after use. Before storage, rinse with tap or distilled water, shake dry and replace the protective cap filled with KCl solution. Make sure the cap contains enough KCl solution to cover the end.

When measurements are taken in fatty or oily substances (i.e., proteins), or to avoid a change in offset calibration, periodically clean the electrode with alcohol and a cotton wad. If the reference junction becomes clogged with these types of products, it may be cleaned by soaking in methanol (approximately one hour).

NOTE: Rinse the electrode thoroughly with distilled water after each cleaning.

Trouble-Shooting Guide

Symptom is...	Problem is...	Possible solutions
Unit does not function with ATC probe	Defective ATC probe	Replace probe and recalibrate pH electrode and ATC probe.
Unit will not calibrate or gives erroneous readings	Defective pH electrode	Replace electrode and recalibrate pH electrode and ATC probe.
Unit gives slow response or erroneous readings	Dry elecrode or clogged junction	Attempt cleaning procedure. If unsuccessful, replace pH electrode and recalibrate electrode and ATC probe.
Unit will not accept second buffer	Defective pH electrode	Attempt cleaning procedure. If unsuccessful, replace pH electrode and recalibrate electrode and ATC probe.
Readings drift on LED display	Defective pH electrode (minute crack in glass)	Replace pH electrode; recalibrate pH electrode and ATC probe.
Left decimal indication on LED display	Low battery	Replace battery and recalibrate pH electrode and ATC probe.

Temperature/pH Value Chart

Temperature		Buffer Values (pH)		
°C	(°F)	4.01	7.00	10.00
0	32	4.01	7.12	10.33
5	41	4.01	7.09	10.25
10	50	4.00	7.06	10.18
15	59	4.00	7.04	10.11
20	68	4.00	7.02	10.05
25	77	4.01	7.00	10.00
30	86	4.01	6.99	9.95
35	95	4.02	6.98	9.92
40	104	4.03	6.98	9.88
45	113	4.04	6.97	9.85
50	122	4.06	6.97	9.82
55	131	4.07	6.98	9.80
60	140	4.09	6.98	9.77
70	158	4.12	6.99	9.73
80	176	4.16	7.00	9.69
90	194	4.20	7.02	9.66

Warranty

The Cole-Parmer Instrument Company warrants this product to be free from defects in material and workmanship for a period of six months from date of purchase. If repair or adjustment is necessary and has not been the result of abuse or misuse within the six month period, please return—freight prepaid—and correction of the defect will be made without charge.

Out-of-warranty products will be repaired on a charge basis.

Return of items

Authorization must be obtained from our Customer Service Department before returning items for any reason. When applying for authorization, please include data regarding the reason the items are to be returned.

For your protection, items must be carefully packed to prevent damage in shipment and insured against possible damage or loss. Cole-Parmer will not be responsible for damage resulting from careless or insufficient packing. A 15% restocking charge will be made on all unauthorized returns.

NOTE: The Cole-Parmer Instrument Company reserves the right to make improvements in design, construction and appearance of our products without notice.

niles 60714

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Neotronics N.A. Inc.,
P.O. Box 370
2144 Hilton Drive S W
Gainesville, Georgia [illegible]
Tel: 404-[illegible]-0600

Pat. Nos. [illegible]; 1,497,087; [illegible]/75 (JAP); P[illegible].4 (D); 1,477,334; [illegible] (US); 149,[illegible] (US); 1,548,572; 1,600,618.
Pat. Applied for: [illegible]; 82.28130

080-0017-02
LIST NO. EX 4C

Distributor

404 535 0600

Neotronics

1982

BASEEFA CERTIFICATE No. EX 84254X

EXOTOX™

PORTABLE MULTI — GAS MONITOR

Model 40
USER MANUAL

EXOTOX Models Available

EXOTOX Model 20 Series

Go/No-Go instruments for the detection of the following combinations of gases with alarm lights and audible alarms:

Model Code	Gases Monitored
20-OFH	Oxygen, Flammable Gases and Hydrogen Sulfide (H_2S)
20-OFC	Oxygen, Flammable Gases and Carbon Monoxide (CO)
20-OF	Oxygen and Flammable Gases
20-F	Flammable Gases only

EXOTOX Model 40 Series

With Liquid Crystal Display digital readout and alarm light and audible alarms:

Model Code	Gases Measured
40-OFH	Oxygen, Flammable Gases and Hydrogen Sulfide (H_2S)
40-OFC	Oxygen, Flammable Gases and Carbon Monoxide (CO)
40-OF	Oxygen and Flammable Gases
40-F	Flammable Gas only

NOTE: For users of EXOTOX Models 20-OF/20-F/40-OF/40-F. Please ignore sections referring to Oxygen and or Toxic Gas Monitoring as these sections are only relevant to triple Gas Instruments (OFH & OFC).

EXOTOX Model 60 Series

Four Gas instrument with Liquid Crystal Display digital readout, Toxic Gas LED's, alarm LED's and audible alarms:

Model Code	Gases Measured
60-OFCH	Oxygen, Flammable Gases, Carbon Monoxide (CO) and Hydrogen Sulfide (H_2S)

WARNING

ALWAYS SWITCH THE EXOTOX OFF BEFORE REMOVING THE BATTERY PACK.

CONTENTS LIST

Page Number

LIST OF ILLUSTRATIONS

CHAPTER 1 THE MONITOR

1.1 How to use the EXOTOX Model 40

Switch on EXOTOX by firmly pressing button marked Power and On Off ('A' in Figure 1). The EXOTOX will automatically run through a self test sequence, flashing all LCD segments with audible and visual alarms operating for 6 seconds.

Following this automatic test, the EXOTOX will go into the Oxygen measurement mode and display digitally, real time percentage Oxygen. Consecutive operation of the 'Select' button ('B' on Figure 1) will display Toxic gas in parts per million (ppm), Time Weighted Average (TWA) to date, 'Hr (hours)' time elapsed from switch on in decimal hours, and Explosive gas in percentage LEL-LFL (Lower Explosive Limit Lower Flammable Limit). This sequence must always be completed before instrument is used to ensure correct calibration. The displays should read approximately 20.9% Oxygen, 0 ppm Toxic gas, 0 ppm TWA 0.0 Hr and 0% LEL (LFL) Explosive gas, in fresh air. Should the display read significantly different from these recommended figures, check calibration as explained in Chapter 5.

1.2 The Operating Signal

Normally operating instruments will continue to emit a short audible bleep every ten seconds and flash the visual alarm lamp as indication that it is functioning correctly. IF THE EXOTOX DOES NOT PRODUCE THE REGULAR OPERATING SIGNAL, THE INSTRUMENT SHOULD NOT BE USED

1.3 Gas Testing Modes

Flammable gas, Oxygen and Toxic gas levels are monitored continuously on the Model 40 OFH and 40 OFC, and can be digitally displayed by pressing the 'Select' button in sequence.

The user can select 5 different readouts

1 Real Time Oxygen 0-35%

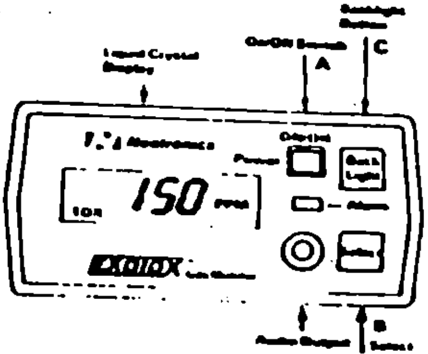


Figure 1 Display Panel

2. Real Time Toxic Gas 0-999 ppm CO, 0-500 ppm H₂S
3. Time Weighted Average (TWA) of Toxic Gas in ppm (see Technical Specifications for details).
4. Decimal hour (i.e. 0.1/Hr = 6 minutes) representing how long the EXOTOX has been on, and therefore the base for the TWA. 'Hr.' and decimal hours flash alternately
5. Real Time percentage LEL (LFL) of explosive gas 0-99%

1.4 The LCD Backlight

The EXOTOX has a backlight facility to illuminate the LCD in low ambient light levels. To operate, press the 'Back Light' button ('C' on Figure 1) while reading the display. The back light only operates while button is depressed.

1.5 Flammable Gas Sensor Fault Alarm

Should a fault condition occur with the Explosive Gas Sensor, the EXOTOX will give a continuous audible alarm, read 'Flt' on the LCD with a side legend of 'EXP' and illuminate the red 'Alarm' light. Under these circumstances the instrument must not be used.

1.6 Low Battery Alarms

The EXOTOX 40 has a two stage low battery indication. The first warning that the batteries are getting low is given by the flashing side legend 'BAT' on the digital display. When this occurs, there is approximately 1 hour of battery life left. The second warning is an audible continuous tone and a blank digital display except for the side legend 'BAT' which indicates final battery discharge. Fit a fully charged, stand by battery pack or charge instrument complete with battery pack in accordance with Chapter 2.

Completely discharged batteries will have insufficient power to operate display. To confirm this is the problem, charge instrument for a few minutes and then re-check.

N.B. For standard alarm settings of gases being monitored, see 'Technical Specifications' at back of manual.

1.7 Overrange Indication

If the EXOTOX is exposed to levels of gas exceeding the instruments readout, (see section 1.3 for ranges) the following will be displayed

Flammable Gas	'EEE'.
Oxygen	'OFL'.
Toxic	'OFL'

The display will return to normal when the high gas concentration is removed but the EXOTOX will need to be reset (switch off and on again) to cancel audible alarm.

CHAPTER 2 THE BATTERY

2.1 Removing the Battery Pack

All EXOTOX monitors are supplied complete with a battery pack connected to the instrument. The battery pack may be charged when still connected to the monitor, or may be charged independently. To disconnect the battery pack from the monitor the following procedure must be used (see Figure 2).

2.1.1 Unscrew self retaining lock screw 'A' on monitor using allen key provided (2.5mm A/F).

2.1.2 Rotate the battery pack through 90° in the direction of arrow 'B' and pull away from monitor.

Before reconnecting battery pack ensure that the contacts on both battery pack and monitor are clean, only clean cloth or paper wipes may be used. UNDER NO CIRCUMSTANCES MAY ABRASIVE CLEANING MATERIAL BE USED AS THIS WILL DAMAGE THE GOLD PLATING OF THE CONTACTS

To reconnect battery pack reverse the procedure for disconnection and ensure that the keyway 'C' on the monitor engages positively with the key slot on the top of the battery pack.

2.2 Battery Pack Contacts

The contacts on the top of the pack are slightly recessed to prevent accidental discharge if placed in contact with a metal surface, although care should be taken to ensure that accidental

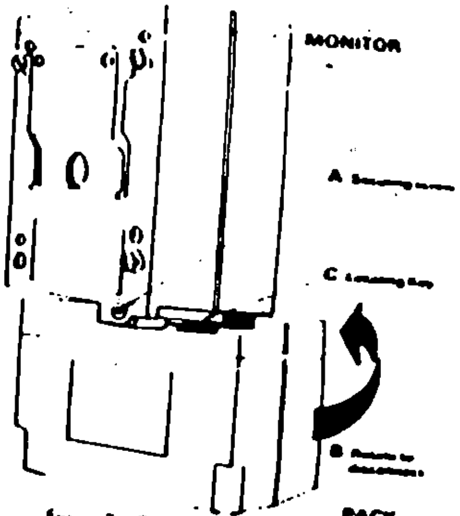


Figure 2 Connecting Battery Pack

shorting of these contacts does not occur when the battery pack is separated from the monitor. The recharging contacts at the base of the battery pack are electrically protected to ensure that the battery pack cannot be discharged through these contacts. This ensures that if the instrument or battery pack is stood on a wet surface, or if accidentally immersed, no discharge of the battery can occur through these contacts.

2.3 Charging the Battery

To ensure that the correct charge conditions are applied to the battery pack, only a Neotronics approved battery charger may be used. The Neotronics EXOTOX battery charger is designed to prevent battery damage due to overcharging. Before inserting the battery pack (with or without the monitor attached) ensure that the electrical contacts on the base of the battery pack are clean. **UNDER NO CIRCUMSTANCES MAY ANY ABRASIVE CLEANING MATERIAL BE USED.**

The battery pack may only be connected to the charger in the position indicated in Figure 3. Moulded in locators ensure correct orientation.

Press the battery pack, or complete monitor, into the charging recess on the battery charger; the weight of battery pack, or monitor, will ensure sufficient contact pressure. As soon as contact is made the red 'FAST' charge light on the battery charger will illuminate. If light does not illuminate ensure charger and battery contacts are clean and firmly press pack into charger.

The battery will be fully recharged from a discharged condition within five hours. Each hour of fast charge will provide about two hours of monitor use.

A built-in detection circuit automatically switches the charger to a 'SLOW' charge condition when sufficient charge has been given to the battery the red 'Fast' charge light automatically switches to a green 'Slow' charge light. The battery may be left on the charge for an indefinite period.

It is recommended that the battery pack is fully discharged at regular intervals, by leaving the monitor on until the low battery alarm sounds.

The monitor should then be fully recharged as per the above procedure. This will help in overcoming problems associated with Ni Cad battery charge/discharge memory patterns, which can reduce the charge capacity of the battery. To enable a precise check to be kept on battery pack performance, a record should be kept as illustrated at the back of this manual.

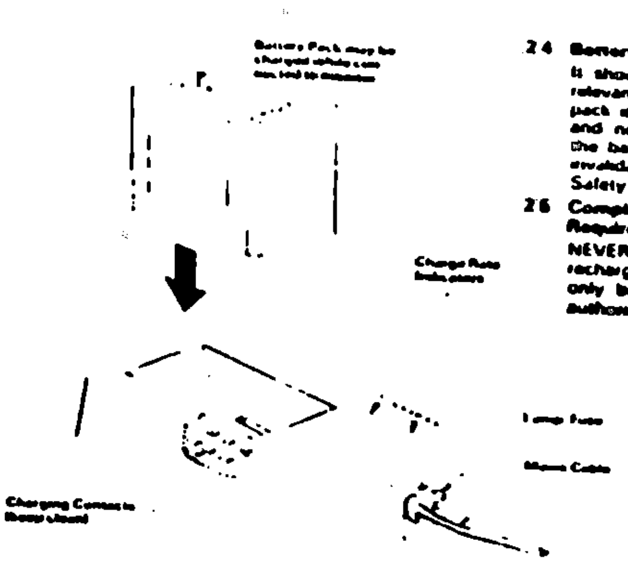


Figure 3 Single Way Battery Charger

2.4 Battery Replacement

It should be noted that, to comply with the relevant Intrinsic Safety standards, the battery pack is a sealed and encapsulated component and no attempt should be made to remove the batteries from the pack. Such action will invalidate the Neotronics guarantee and Intrinsic Safety certification.

2.5 Compliance with Intrinsic Safety Requirements

NEVER allow battery packs to be charged or recharged in hazardous atmospheres. This must only be carried out in safe atmospheres by authorised personnel.

CHAPTER 3 SUGGESTED ON-SITE PROCEDURES

3.1 Procedure prior to confined entry space

3.1.1 Check that the EXOTOX is fit for use

3.1.2 Switch on monitor

3.1.3 Ensure that any hand line being used is securely attached to the monitor harness or belt clip. Use handline to lower EXOTOX into working space

3.1.4 Lift access covers to assist with the ventilation of the work space, prior to entry

3.1.5 Ensure the means of access and escape are secure and fit for use in normal conditions

3.1.6 Lower the monitor into the work space, checking where necessary, various levels. The correct time (5 MINUTES) must be allowed at each check point to ensure that monitor carried out a complete sensing cycle, and responds to all possible gas dangers

3.1.7 Using the hand line, withdraw the monitor from the work space and observe any alarm indication. If no audible or visual alarm is operating the indication is that the work space is free of those gas hazards that the monitor is designed to measure

3.1.8 **If alarm indication occurs, the work space should not be entered under any circumstances. Refer to your Work Supervisor for further instructions**

Figure 4 How the EXOTOX should be Worn

3.1.9 If no alarm is indicated remove the EXOTOX from the hand-line and wear the monitor near the breathing zone (see Figure 4) prior to entry of each space.

3.2 Procedure While Working

Under most working conditions the monitor should be carried on the person at all times. The EXOTOX is designed specifically for personal monitoring, and working away from the monitor may lead to an alarm indication being unnoticed due, for example, to high noise levels in the working area. The size and weight of the monitor enable it to be carried comfortably for long periods without operator fatigue.

When carried, both audible and visual alarm conditions and LCD, are easily observed. In high noise environments encountered around pumps, or with fast flow conditions, use the acoustic earphone. This gives additional safeguard. If at any time while working within the confined space the monitor gives an alarm indication, for whatever reason, the confined space **MUST BE EVACUATED IMMEDIATELY.**

Point to Note

If, during the initial atmosphere testing prior to entry the monitor alarms, it must be withdrawn immediately. If upon examination, it indicates a hazardous atmosphere:

1 Switch off the monitor to re set the alarm condition.

2 Switch the monitor on.

3 Allow the monitor to auto check and then replace into the work space.

4 If the monitor again alarms and indicates a hazardous atmosphere, switch off the monitor and follow your local operating procedures for such conditions.

CHAPTER 4 GENERAL MAINTENANCE

4.1 The Outer Case

The outer casing of the monitor is moulded in heavy duty plastic and only requires wiping down with a clean damp cloth or paper. No solvents or other proprietary cleaners should be used, as these may damage the moulding material.

4.2 The Grill

Check that the grill covering the sensors on the front of the monitor, is clean and free from obstruction at all times.

4.3 The Straps

Check that all carrying straps and attachm points are clean, undamaged and fit for use

4.4 Storage

When not in use, monitors should be stored cool dry room on racks or hung by their carr straps or belt clip. Alternatively, monitors ma kept on charge when not in use

TABLE 1: CHARACTERISTICS OF TYPICAL GASES AND VAPOURS
(FOR OTHERS NOT LISTED CONSULT OUR TECHNICAL ADVISORY SERVICE)

FLAMMABLE SUBSTANCE	STANDARD SENSITIVITY % LEL	VOL %	SPECIFIC GRAVITY OF GAS AT 20°C
ACETIC ACID	36.8	2.00	2.10
ACETONE	38.6	1.00	2.03
ACETYLENE	35.2	0.80	0.91
AIR			1.00
AMMONIA	15.80	2.53	0.58
BENZENE	40.00	0.64	2.73
n BUTANE	34.0	0.65	2.03
n DECANE	61.0	0.40	4.97
DIPHENYL	80.0	0.48	1.30
ETHYL ALCOHOL	27.4	0.90	1.61
ETHYLENOXIDE	38.6	1.16	1.54
n HEPTANE	51.8	0.52	3.50
HYDROGEN	26.0	1.04	0.07
METHANE (Natural Gas)	20.0	1.06	0.56
METHYL ALCOHOL	23.2	1.00	1.12
PROPANE	36.2	0.80	1.54
TOLUENE	49.4	0.82	3.22
TRIMETHYL AMINE	41.2	0.82	2.08
XYLENE	61.0	0.56	3.70

For Model 40 EXOTOX Gas Monitor with Standard Calibration (20% Methane in Air)

CHAPTER 5 THE SENSORS

CALIBRATION USING THE NEOTRONICS CALIBRATION KIT

5.1 Preparation of Calibration Kit

Select the gas bottle required for the test.
Check that the bottle contains adequate gas pressure.
Connect tubing from the flow meter input to the gas bottle regulator.
Connect the flow meter output to the Calibration Aspirator Hood Port which will be nearest to the sensor under test. Depress the button on top of the Gas Bottle Regulator and ensure that the flow meter indicates 250 to 300 cm³/minute. Keep the button depressed for 10 seconds to purge the system.
Remove the Sensor Grill from the EXOTOX.
Attach the Calibration Aspirator Hood to the instrument.

ONLY ATTEMPT CALIBRATION OUTSIDE OR IN A WELL VENTILATED ENVIRONMENT.

The equipment is now set up for calibration of the EXOTOX.

5.2 Explosvie Gas Calibration Check (All Models)

To check the calibration of the flammable gas detection element use the Neotronics' standard calibration gas mixture of 1.15% Methane in Air with the following procedure

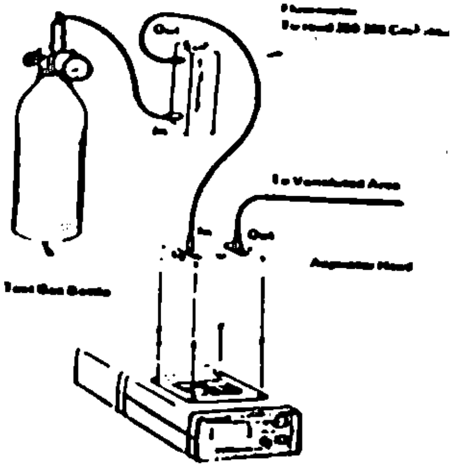


Figure 5 Calibration Kit Connection

5.2.1 Connect Calibration Kit as detailed in procedure 5.1.
Switch the EXOTOX ON and select Explosive display mode.

5.2.2 Depress the button on top of the Gas Bottle Regulator until either the EXOTOX goes into alarm condition (Audible, Display 'EXP' & 30%) or the maximum time limit is exceeded. The time limits for various models are listed in Figure 8. The time is measured from the depression of the button. If the results from this test fall outside those detailed in figure 8, the EXOTOX requires recalibration (procedure 5.3).

5.3 Explosive Gas Calibration Adjustment (All Models)

Only authorised, qualified personnel should attempt recalibration using the following procedure. Under no circumstances should unauthorised access to the calibration controls be allowed.

5.3.1 (Zero Adjustment)
Remove the sensor grill using a 2in Hexagonal Wrench (Allen Key)

5.3.2 Switch EXOTOX ON and select the Explosive Gas Display Mode.

5.3.3 With the monitor in a Flammable Gas Free Environment (Fresh Air), slowly adjust the 'Explosive Zero' potentiometer (Figure 7 'G') until a reading of zero percent is obtained on the display.

5.3.4 (Span Adjustment)
NOTE: The type of gas used to calibrate the Explosive Span should be a mixture of between 1 & 1.5% of Methane in Air (20 to 30% LEL) The EXOTOX displays the Methane concentration in its LEL form. For the purpose of calibration, the LEL is calculated as follows

% LEL value of gas = % age concentration by volume x 20

5.3.5 Connect the Calibration Kit as detailed in procedure 5.1.

5.3.6 Depress the button on top of the Gas Bottle Regulator for 1 minute and then slowly adjust the 'Exp Span' potentiometer (Figure 7 'H') until the display reads the figure calculated above ± 1% LEL. Turn OFF the gas.

5.3.7 Allow the EXOTOX to settle for 15 minutes and then recheck the zero. This may have changed during the calibration process. If necessary, repeat procedure 5.3.3
Replace the sensor grill

5.4 Oxygen Sensor Replacement and Recalibration (Models OF, OFH & OFC)

The low Oxygen alarm on the EXOTOX is set to activate at a concentration of 19% Oxygen in Air by Volume (19.5% U.S.)

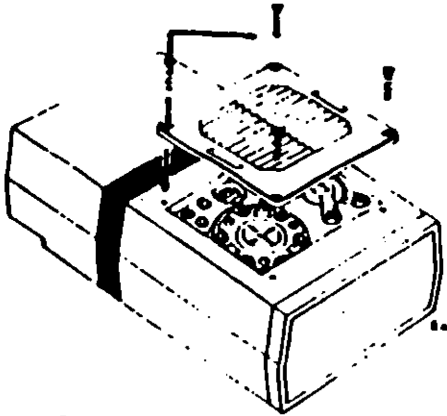


Figure 6 Sensor Grill
Removal and Replacement

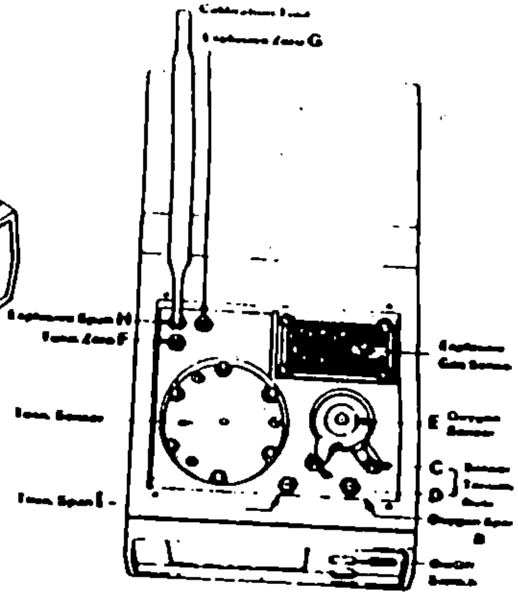


Figure 7 Calibration Controls

The response of the alarm may be checked simply using exhaled breath which, at a normal breathing rate, contains about 17% Oxygen by Volume.

At a constant temperature, the Oxygen sensor in the EXOTOX will remain stable. Ambient temperature changes of ± 10 degrees centigrade will cause small changes on the display (typically ± 0.4% Oxygen). If the EXOTOX is to be used consistently at temperatures other than normal surface ambient conditions, it is recommended that the monitor be recalibrated for 20.9% Oxygen in conditions equivalent to the workplace.

Recalibration may be achieved as follows.

5.4.1 Remove the sensor grill.
Ensure that the monitor is in a 'Fresh Air' type environment. Slowly adjust the Oxygen span control (Figure 7 'B') until the display indicates 20.9%. NOTE: Make sure that no exhaled breath enters the sensor chamber during the procedure.

5.4.2 If an indication of 20.9% cannot be obtained, the sensor needs replacement.

5.4.3 The Oxygen Sensor (Figure 7 'E') can be easily replaced by the removal of the two castle nuts (Figure 7 'C' & 'D'). The new sensor is again secured firmly in place by the castle nuts and washers.

5.4.4 After the sensor has been allowed a five minute settling in period (with the EXOTOX's power OFF), procedure step 5.4.1 must be carried out.

5.4.6 A 'HI' or 'LO' Oxygen display after a normal instrument power on and auto check cycle, indicates that the calibration is substantially incorrect.

5.5 Toxic Gas Zero Adjustment (Models OFH & OFC)

5.5.1 Switch the EXOTOX ON in a Toxic Gas Free Environment (Fresh Air) and select the Toxic Gas Display.

5.5.2 Slowly adjust the 'Toxic Zero' potentiometer ('F' in *Figure 7*) until the digital display reads zero ppm TOX.

5.6 Toxic Gas Alarm Check (Models OFH & OFC)

5.6.1 Connect Calibration Kit as detailed in procedure 5.1.
The Standard Gas to be used should be selected from Figure 8.

5.6.2 Depress the button on top of the Gas Bottle Regulator until the EXOTOX goes into alarm or the maximum time limit is exceeded. The time limits for various EXOTOX models are detailed in Figure 8. The time is measured from the depression of the button. If the results from this test falls outside those detailed in Figure 8, the EXOTOX requires recalibration.

5.7 Toxic Gas Sensor Recalibration (Models OFH & OFC)

Only authorised qualified personnel should attempt recalibration using the following procedure. Under no circumstances should unauthorised access to calibration controls be allowed.

5.7.1 Ensure that the toxic sensor zero is correctly set using procedure 5.5.

5.7.2 Connect the Calibration Kit as detailed in procedure 5.1.
The calibration test gases used for different models are detailed in Figures 9 & 10.

5.7.3 Depress the button on top of the Gas Bottle Regulator and keep it depressed for 10 minutes (for H_2S and 5 minutes for CO). Slowly adjust the 'Tox Span' potentiometer (Figure 7 'E') for a display indication the same as the concentration quoted by the Gas Bottle Manufacturer ± 1%.

5.7.4 Release the button and turn the regulator OFF. Disconnect the aspirator head and replace the sensor grill.
Allow the EXOTOX to settle for 15 minutes and then recheck the Toxic Zero as this may have changed slightly during the calibration process. If necessary, repeat procedure 5.5.

5.8 Sensor Warranty

The Toxic Sensors for H_2S and CO have a twelve month warranty. They can be expected to provide sensing for a period of two years of normal instrument use.

Explosive gas sensors (Pellistors) also have a twelve month warranty and can be expected to provide sensing for 3 years of normal instrument operation.

Oxygen Sensors have a six month warranty and can be expected to operate satisfactory for 9 months of normal instrument use.

FIGURE 8. EXOTOX ALARM REACTION TIMES

Model	Gas Mixture	Minimum Toxic Alarm Time	Minimum Exp' Alarm Time	Maximum Alarm Time
EXOTOX 20 MkII OFH	65–75 ppm H_2S	30 seconds		90 seconds
	1% Methane (20% LEL)		30 seconds	90 seconds
EXOTOX 20 MkII OFC	560–600 ppm CO	15 seconds		45 seconds
	1% Methane (20% LEL)		30 seconds	90 seconds
EXOTOX 40 MkII OFH	66–75 ppm H_2S	30 seconds		90 seconds
	1.15% Methane (23% LEL)		30 seconds	90 seconds
EXOTOX 40 MkII OFC	560–600 ppm CO	15 seconds		45 seconds
	1.15% Methane (23% LEL)		30 seconds	90 seconds
EXOTOX 20 MkII OF & F	1% Methane (20% LEL)		30 seconds	90 seconds
EXOTOX 40 MkII OF & F	1.15% Methane (23% LEL)		30 seconds	90 seconds

Note: All reaction times shown are for 20% LEL models. Models with different Explosive Alarm point are calibrated with different test gas mixtures. If in doubt, contact the Manufacturer's Service Department or your nearest distributor for further details.

FIGURE 9. STANDARD GASES FOR HYDROGEN SULPHIDE CALIBRATION

Alarm Point (PPM)	Gas Mixture to be Applied (PPM)
50	60–70
100	115–125

FIGURE 10. STANDARD GASES FOR CARBON MONOXIDE CALIBRATION

Alarm Point (PPM)	Gas Mixture to be Applied (PPM)
50	65–75
100	130–140
300	340–360
400	440–460
500	560–600

CHAPTER 6 ACCESSORIES

6.1 The Acoustic Earphone

This is for use when monitor is being operated in high background noise conditions, such as are found in pumping station sumps, wet wells or in conditions of fast flow. The ear piece is retained in place by a plastic loop, which may be reversed for use in either ear. The rubber connector at the end of the hose is inserted into position on the monitor as shown in Figure 8.

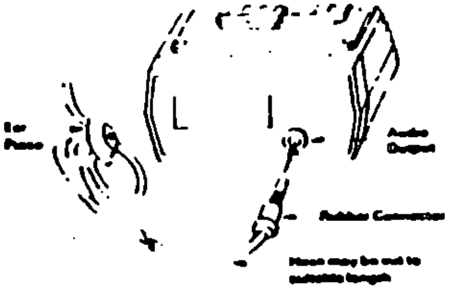


Figure 11 Earphone Connection

The connecting hose may, if necessary, b shortened to suit individual user need.

For reasons of hygiene, each user of th EXOTOX should have his own earphone. If r issuing an earphone to a new user, ensure that is cleaned and sterilized in a suitable steriliz medium such as domestic disinfectant and a ne replaceable earpiece fitted (see list of accessori

6.2 The Hand Aspirator

This attachment enables work space mospheres to be sampled without the necess of exposing the whole monitor to the work environment.

This attachment should be used if service duc surface trenches, supply pipes, etc., require g testing.

6.2.1 Remove aspirator grill attachment, aspira bulb and rubber hosing from case, and in the two mounting tongues into recess on monitor grill (see Figure 12). Ensure that aspirator hose is on the left side of the mon (viewed from the display end).

6.2.2 Pump the aspirator three times in fresh ai ensure that the aspirator and sensor hous are free from retained gas. Check that the line filter is not obstructed, this is indicated a slow return to normal shape after depress the aspirator bulb. Replace with recommend type (see Accessories List).

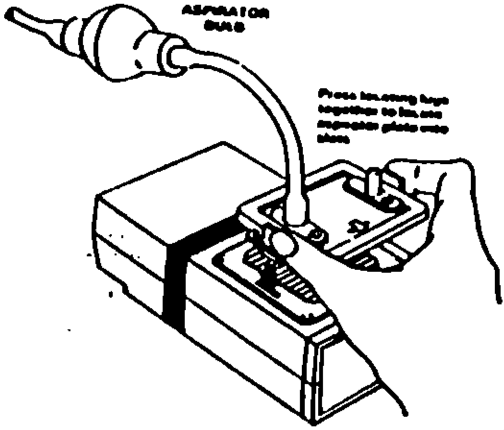


Figure 12 Aspirator Attachment

6.2.3 Always ensure that the end of the sampling hose, when inserted into the work space, is not in contact with standing liquids or solid deposits which could be accidentally drawn into the sampling hose

6.3 List of Accessories

300	0185	00	Earphone Extension
325	0347	00	10 Earpiece Replacements
300	0186	00	Aspirator Attachment
035	0020	00	Carrying Case for Aspirator Attachment
138	0014	00	Oxygen Sensor
138	0010	01	Hydrogen Sulphide Sensor
138	0009	01	Carbon Monoxide Sensor
080	0001	00	Calibration test Gas (1.5% Methane in Air
325	0346	00	Ni Cad Battery Pack
300	0187	00	Replacement Harness
300	0184	00	Single Way Battery Charger
300	0184	01	4 Way Battery Charger
085	0005	00	Calibration Tool
085	0006	01	Pkt 10 2.5mm A/F Hexagonal Wrench/Allen Keys
085	0002	01	Pkt 10 2.0mm A/F Hexagonal Wrench/Allen Keys
300	0128	00	Box 100 Filters for Aspirator Attachment
059	0017	01	EXOTOX Model 40 User Manual
059	0016	01	EXOTOX Model 40 User Card
059	0017	02	EXOTOX Model 40 User Manual (USA)
059	0018	05	EXOTOX Model 40 User Card (USA)

CHAPTER 7 RECORD

7.1 Keeping a Log for each Instrument

Records should be kept of each monitor and battery pack. This will ensure that correct information is recorded concerning sensor and battery life, and will further ensure that periodic checks of the monitor's performance have carried out

Two suggested monitor log sheets are illus below

INSTRUMENT SERIAL NUMBER			
SENSOR SERIAL NUMBER	DATE OF PURCHASE		
	SENSOR TYPE	DATE FITTED	DATE FAILED

BATTERY PACK SERIAL NO	BATTERY PACK		
	DATE FITTED	DATE FAILED	COMMENTS

Summary of Technical Specification

BASEEFA CERTIFICATE NO. EX 84254X

Dimensions
48mm × 98mm × 178mm (1.9" × 3.9" × 7.0")

Weight:
1.1Kg (2.4lb).

Battery Capacity:
10 hours minimum with charged batteries

Operating Temperature:
-15° to 40°C (5°F to 104°F)

Storage Temperature:
– 20° C to 45°C (– 4°F to 113°F)

Humidity:
0 to 99%, Non condensing.

LCD readout:
Percentage Oxygen: 0–35.0
Parts per million Toxic. H_2S 0–500, CO. 0–999
TWA at elapsed time
Elapsed time from instrument switch on alternately displaying – 'Hr' (hour) and decimal hours. 0–99.9 Hrs
Percentage LEL (LFL) Explosive Gas: 0–99

CONDUCTIVITY METER

Model 1484-00
1484-10/1484-11
1484-20/1484-30

Instruction Manual

Cole Parmer

7425 North Oak Park Avenue, Chicago, Illinois 60648

WARRANTY

All instruments are fully guaranteed against defective materials and workmanship for one year. Any attempted repair voids the warranty on this instrument.

RANGE 5, FROM 10,000 TO 20,000
MICROMHOS, IS NONLINEAR AND
SHOULD BE USED FOR RELATIVE
MEASUREMENTS ONLY.

CONDUCTIVITY METER

Table of Contents

CONDUCTIVITY METER

1.0 Introduction

The Bench/Portable Conductivity Meter measures total ionized substances in solution. The meter displays conductivity in five ranges from 0-20,000 micromhos/centimeter (μmhos/cm) and comes with a break-resistant probe that has an internal thermistor for automatic temperature compensation.

2.0 Specifications

Range (μmhos/cm):	0-2 0-20 0-200 0-2,000 0-20,000
Accuracy:	± 2% Full Scale
Temperature Compensation:	Automatic, 5-45°C
Sensor:	Dip Style — Gold Plated Electrodes
Power:	
Bench:	AC — 115 Volt Model or 230 Volt Model DC — Eight Size AA Batteries
Portable:	AC/DC Rechargeable NiCad Batteries 110 VAC or 220 VAC
Size:	
Bench:	8" W × 6" H × 6" D
Portable	12¼" W × 4¼" H × 8" D

3.0 Preliminary Set Up

3.1 Remove and inspect carton for the following:
- a. AC or DC Conductivity Meter
- b. Conductivity probe with six feet of cable
- c. Probe holder with support rod
- d. Instruction Manual
- e. Conductivity Standards — 10,000 μmhos, 1000 μmhos, 100 μmhos

3.2 Install the instrument in a location with adequate ventilation and freedom from vibration. In addition, close proximity to high voltage wires or transformers should be avoided.

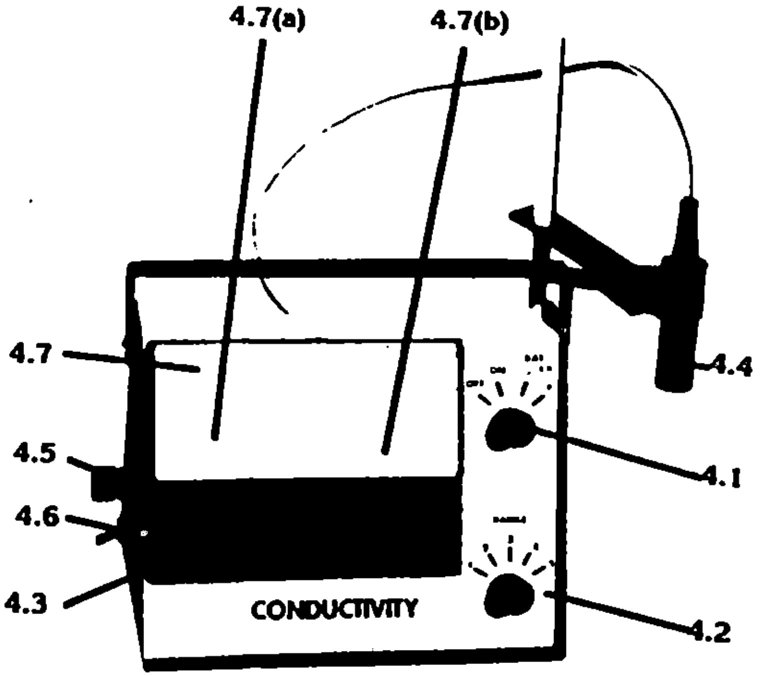
3.3 Turn top power switch to the Off position.

3.4 AC Models — Plug AC cord into appropriate electrical outlet. AC/DC Models should be allowed to charge for at least 24 hours to insure a fully charged battery.

3.5 Turn instrument On — both AC and DC units should be ready to use after a five minute warm-up.

3.6 Immerse and store probe in distilled or deionized water.

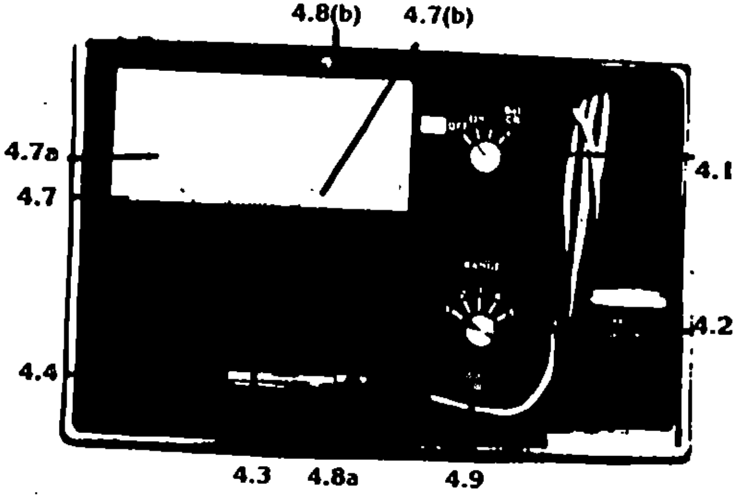
4.0 Front Panel



4.1 Function (Off/On/(− , +)BATT-CK)
- Off: Turns power Off to conserve battery/meter life
- ON: Activates meter
- (− , +) Batt CK: Each position will monitor the batteries(DC) or power supply (AC)

4.2 Range: Five position switch used to select proper conductivity scale

4.3 Zero adjustment screw
4.4 Conductivity probe
4.5 Fuse holder for 1/16 amp fuse (AC model only)
4.6 Line cord (AC model only)
4.7 Analog Meter Face: Containing 5 conductivity scales covering 0-20,000 μmhos/cm
  a. Range Selection
  b. Batt OK Zone — Acceptable zone when checking battery or power supply function
4.8 Front Panel Screws a&b: Two screws that fasten front panel to carrying case. Screws must be completely removed and panel lifted out to gain access to the rechargeable batteries.
4.9 Calibration Adjustment — Use to calibrate the meter using a conductivity standard (on back panel of bench model)



5.0 Checking Meter Function

5.1 Turn meter off — with flathead screwdriver turn the zero adjustment screw so that the needle reads zero
5.2 DC models — turn function switch to BATT CK − The needle should read in BATT OK zone. A reading below this zone indicates that the negative bank of batteries need to be replaced. Turn function switch to BATT CK + A reading below the BATT OK zone indicates that the positive bank of batteries need to be replaced.

5.3 AC models — turn function switch to BATT CK − or +. A reading below the BATT OK zone indicates power supply failure and the meter needs to be serviced.
5.4 AC/DC Models — Turn function switch to BATT CK − or +. A reading below the BATT OK zone indicates the batteries require recharging.
5.5 Turn instrument ON.

6.0 Sample Measurement

6.1 Turn the range switch to position 5. Rinse the probe with either distilled or deionized water.
6.2 Insert the probe into the unknown solution at least one inch without touching the sides or bottom of the container.
6.3 Decrease the range one step at a time until the meter reading is between 10% and 90% of full scale.
6.4 The probe will automatically compensate for sample temperatures between 5°C and 45°C. It may take several minutes for the reading to stabilize when the temperature of the solution is different from the test environment. The reading is taken after the output has stabilized.
6.5 When you measure low conductivity levels, the sample should be protected from the atmosphere since dissolved gases will rapidly change the conductivity.
6.6 After the measurement range has been determined, select a standard within that range and calibrate the meter at the selected range.
6.7 Re-measure the unknown solution.
6.8 The probe should be rinsed and stored in distilled water after each measurement.

7.0 Theory

7.1 Conductivity is the measurement of the amount of electrical current that will flow across two noble metal surfaces when a constant voltage is applied. Conductivity is a non-selective measurement with any charged ion contributing to the total conductivity. Organic compounds such as phenols, alcohols, oils, etc., do not dissosiate (ionize) in water and therefore have little or no effect on the conductivity.

Conductivity is normally expressed as micromhos per centimeter (µmhos/cm). In the International System of Units (SI), conductivity is expressed as millisiemens per meter, where 1 mS/m is equal to 10 µmhos/cm or 1 µS/cm is equal to 1 µmho/cm.

Conductivities of some common liquids:

Freshly distilled water	.5 to 2.0 µmhos/cm
Potable Water	50 to 1,500 µmhos/cm
Normal Saline	18,400 µmhos/cm

7.2 Conductivity can be used to determine concentration. A nearly linear relationship exists between conductivity and ion concentration for most ionic compounds below 2,000 µmhos/cm. As a result conductivity is often measured to determine total dissolved solids (TDS). It is important to note that this is only a valid methodology when the ionic solution is composed of a pure compound since the exact relationship between conductivity and concentration varies with each ionic compound.

Some examples of the relationship between concentration and conductivity are:

Salt	Concentration	Conductivity at 25°C
Calcium Carbonate ($CaCO_3$)	1 G/L (1000 ppm)	2300 µmhos/cm
Sodium Chloride (NaCl)	1 G/L (1000 ppm)	1990 µmhos/cm
Potassium Chloride (KCl)	1 G/L (1000 ppm)	1880 µmhos/cm

8.0 Troubleshooting

8.1 **Symptom:** Meter exhibits no response.

Action

a. Check power to meter.
  1. AC meter not plugged in
  2. AC meter has blown fuse
  3. Turn function switch to BATT CK to test power supply (AC) or batteries (DC)

Conclusion:

If the meter seems to be adequately powered, proceed to next step.

If meter needle does not register BATT OK when function is checked then:

— Replace batteries in DC model (negative bank only if battery is adequate in positive check, positive bank only if battery is adequate in negative check).

— Power supply needs to be serviced in AC model.

b. Check for electrical short.
  1. Turn meter on and turn range switch to position 5
  2. Hold a 100 ohm resistor across the metal surfaces at the end of the conductivity probe
  3. The meter needle should read at least 10,000 µmhos/cm

Conclusion:

If the meter needle does not deflect at all or deflects less than 10,000 µmhos/cm, then the meter needs to be serviced.

8.2 **Symptom:** Standard does not fall within acceptable limits.

Action:

a. Clean probe using a mild detergent to remove oil or debris.

b. Go to 8.1 b and check for electrical short.

Conclusion:

Meter needs to be serviced.

9.0 Parts List

Description	Part Number
Probe	300-7500-00
Meter	650-1070-00
½" Probe Cable Clamp	560-8916-00
Rod Clamp	536-0013-02
Probe Holder	538-0100-00
Test Tube Clip	559-1006-00
Nylon Rod	532-0011-00
100K Pot	634-0104-10
AC Unit:	
AC Power Board 115 Volts	600-0001-10
AC Power Board 230 Volts	600-0001-20
Power Cord	780-7136-00
Fuse Holder	567-2014-00
Fuse, 1/16 Amp	714-2062-00
Board	600-0017-03
DC Unit:	
Mercury Battery	709-0009-00
Battery	567-2182-00
Board	600-0017-03
AC/DC Unit:	
[1] Battery Holders	567-1290-00
[1] Battery Plate	534-0100-23
[1] Battery Plate Stands	538-0100-22
[1] Battery Straps	538-0100-21
Terminal Strip	734-8643-00
330 Ohm Resistor	631-0331-00
220 μF 16V Capacitor	627-2281-00
Rechargeable Batteries, 9V	706-0022-00
Battery Snaps	569-0745-00
[2] Charger, 115V	702-8113-00
[2] Charger, 230V	702-0504-20
Portable Case	526-2585-00
Board	600-0700-02
1484-00/1484-10/1484-11 1484-20/1484-30 Instruction Manual	175-1484-00
Model 70/700 Instruction Manual	175-7000-00

[1] To obtain total assembly — order all parts together.

[2] Make certain to select correct chargers for your unit.

APPENDIX G

USEPA CONTRACT LABORATORY PROGRAM NATIONAL FUNCTIONAL GUIDELINES FOR ORGANIC DATA REVIEW AND INORGANIC ANALYSES

USEPA CONTRACT LABORATORY PROGRAM

NATIONAL FUNCTIONAL GUIDELINES
FOR
ORGANIC DATA REVIEW

Multi-Media, Multi-Concentration (OLM01.0)

and

Low Concentration Water (OLC01.0)

DRAFT

December, 1990
Revised June, 1991

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INTRODUCTION

This document is designed to offer guidance on EPA Contract Laboratory Program (CLP) analytical data evaluation and review. In some applications it may be used as a Standard Operating Procedure (SOP). In other, more subjective areas, only general guidance is offered due to the complexities and uniqueness of data relative to specific samples. For example, areas where the application of specific SOPs are possible are primarily those in which definitive performance criteria are established. These criteria are concerned with specifications that are not sample dependent: they specify performance requirements that should fully be under a laboratory's control. These specific areas include blanks, calibration standards, performance evaluation standard materials, and instrument performance checks (tuning).

These Guidelines have been updated to include the requirements in the Statement of Work (SOW) for Organic Analysis Multi-Media Multi-Concentration (SOW OLM01.0), <u>and</u> the SOW for Low Concentration Water Organic Analysis (SOW OLC01.0). *To ensure that the data review guidelines that are unique to the Low Concentration Water SOW are easily identified, these requirements and procedures are presented in italics and contained within brackets ([ ]) throughout the document.*

This update includes changes to instrument performance checks (formerly referred to as tuning) including changes to instrument performance checks and calibration criteria as a result of the Response Factor Workgroup. Minor revisions to the Data Qualifier Definitions from the previous National Functional Guidelines are also included in this document.

This document is intended to assist in the <u>technical</u> review of analytical data generated through the CLP. Determining contract compliance is not the intended objective of these guidelines or the regional data review process. The data review process provides information on analytical limitations of data based on specific quality control (QC) criteria. In order to provide more specific useability statements, the reviewer must have a complete understanding of the intended use of the data. For this reason, it is recommended that whenever possible the reviewer obtain usability issues from the user prior to reviewing the data. When this is not possible, the user should be encouraged to communicate any questions to the reviewer.

At times, there may be an urgent need to use data which do not meet all contract requirements and technical criteria. Use of these data does <u>not</u> constitute either a new requirement standard or full acceptance of the data. Any decision to utilize data for which performance criteria have not been met is strictly to facilitate the progress of projects requiring the availability of the data. A contract laboratory submitting data which are out of specification may be required to rerun samples or resubmit data even if the previously submitted data have been utilized due to urgent program needs; data which do not meet specified requirements are never fully acceptable. The only exception to this requirement is in the area of requirements for individual sample analysis; if the nature of the sample itself limits the attainment of specifications, appropriate allowances must be made. The overriding concern of the Agency is to obtain data which are technically valid and legally defensible.

Appendix A is based on the Multi-media Multi-concentration SOW and contains appropriate contractual requirements and equations for verifying various calculations. Appendix B contains the corresponding contractual requirements and equations from the Low Concentration Water SOW. Appropriate equations are presented for easy reference and to allow the reviewer to verify calculations as needed. Contractual requirements are provided to facilitate comparisons with the technical requirements. For each analytical fraction, Appendix C contains a table comparing contractual requirements of the Multi-media, Multi-concentration with those of the Low Concentration Water SOWs. Appendix D contains proposed guidance for Tentatively Identified Compounds (VOA and SV), and Appendix E contains a glossary of commonly used terms.

The data review should include comments that clearly identify the problems associated with a Case or Sample Delivery Group and to state the limitations of the data. Documentation should include the sample number, analytical method, extent of the problem, and assigned qualifiers.

A data review narrative generally accompanies the laboratory data forwarded to the intended data recipient (client) or user to promote communication. A copy of the data review narrative should be submitted to the CLP Quality Assurance Coordinator (QAC), the Regional CLP Technical Project Officer (TPO) assigned oversight responsibility for the laboratory producing the data, and the Environmental Monitoring Systems Laboratory in Las Vegas, Nevada (EMSL-LV).

It is the responsibility of the data reviewer to notify the appropriate Regional CLP TPO concerning problems and deficiencies with regard to laboratory data. If there is an urgent requirement, the TPO may be contacted by telephone to expedite corrective action. It is recommended that all items for TPO action be presented at one time.

PRELIMINARY REVIEW

In order to use this document effectively, the reviewer should have a general overview of the sample delivery group (SDG) or case at hand. The exact number of samples, their assigned numbers, their matrix, and the number of laboratories involved in their analysis are essential information. Background information on the site is helpful but often this information may be difficult to locate. The site manager is the best source for answers to questions or further direction.

Contract Compliance Screening (CCS) is a source of summarized information regarding contract compliance. If available, it can be used to alert the reviewer to problems in the SDG data package.

Sample cases (SDGs) routinely have unique samples which require special attention by the reviewer. These include field blanks, field duplicates, and performance audit samples which need to be identified. The sampling records should provide:

1. Project Officer for site.

2. Complete list of samples with information on:

    a. sample matrix,
    b. field blanks,
    c. field duplicates,
    d. field spikes,
    e. QC audit samples,
    f. shipping dates, and
    g. laboratories involved.

The chain-of-custody record includes sample descriptions and date(s) of sampling. The reviewer must take into account lag times between sampling and receipt for analysis when assessing technical sample holding times.

The laboratory's SDG Narrative is another source of general information. Notable problems with matrices, insufficient sample volume for analysis or reanalysis, samples received in broken containers, and unusual events should be found in the SDG Narrative.

The SDG Narrative for the sample data package must include a Laboratory Certification Statement (exactly as stated in the SOW), signed by the laboratory manager or his designee. This statement authorizes the validation and release of the sample data results. In addition, the laboratory must also provide comments in the SDG narrative describing in detail any problems encountered in processing the samples in the data package.

For every data package, the reviewer must verify that the laboratory certification statement is present, exactly stated as in the SOW (i.e., verbatim to the statement in the SOW), and signed by the Laboratory Manager or designee. The reviewer must further verify that the data package is consistent with the laboratory's certified narrative. Also, the reviewer should check the comments provided in the narrative to determine if they are sufficient to describe and explain the associated problem.

DATA QUALIFIER DEFINITIONS

The following definitions provide brief explanations of the national qualifiers assigned to results in the data review process. If the Regions choose to use additional qualifiers, a complete explanation of those qualifiers should accompany the data review.

U	-	**The analyte was analyzed for, but was not detected above the reported sample quantitation limit.**
J	-	**The analyte was positively identified; the associated numerical value is the approximate concentration of the analyte in the sample.**
N	-	**The analysis indicates the presence of an analyte for which there is presumptive evidence to make a "tentative identification."**
NJ	-	**The analysis indicates the presence of an analyte that has been "tentatively identified" and the associated numerical value represents its approximate concentration.**
UJ	-	**The analyte was not detected above the reported sample quantitation limit. However, the reported quantitation limit is approximate and may or may not represent the actual limit of quantitation necessary to accurately and precisely measure the analyte in the sample.**
R	-	**The sample results are rejected due to serious deficiencies in the ability to analyze the sample and meet quality control criteria. The presence or absence of the analyte cannot be verified.**

VOLATILE DATA REVIEW

**** Data review guidelines that are unique to data generated through the Low Concentration Water SOW are contained within brackets ([ ]), and written in italics. ****

The volatile data requirements to be checked are listed below

I. Technical Holding Times (CCS - Contractual holding times only)

II. GC/MS Instrument Performance Check (CCS)

III. Initial Calibration (CCS)

IV. Continuing Calibration (CCS)

V. Blanks

VI. System Monitoring Compounds (Surrogate Spikes) (CCS)

VII. Matrix Spikes/Matrix Spike Duplicates

VIII. Laboratory Control Samples (CCS)

IX. Regional Quality Assurance and Quality Control

X. Internal Standards (CCS)

XI. Target Compound Identification

XII. Compound Quantitation and Reported Contract Required Quantitation Limits (CRQLs)

XIII. Tentatively Identified Compounds

XIV. System Performance

XV. Overall Assessment of Data

<u>NOTE</u>: "CCS" indicates that the contractual requirements for these items will also be checked by CCS; CCS requirements are not always the same as the data review criteria.

I. Technical Holding Times

A. **Review Items:** Form I VOA *[Form I LCV]*, EPA Sample Traffic Report and or chain-of-custody, raw data, and SDG Narrative.

B. Objective

The objective is to ascertain the validity of results based on the holding time of the sample from time of collection to time of analysis.

C. Criteria

Technical requirements for sample holding times have only been established for water matrices. The holding times for soils (and other non-aqueous matrices such as sediments, oily wastes, and sludge) are currently under investigation. When the results are available they will be incorporated into the data evaluation process. Additionally, results of holding time studies will be incorporated into the data review criteria as the studies are conducted and approved.

The holding time criteria for water samples, as stated in the current 40 CFR Part 136 (Clean Water Act) is as follows:

- For non-aromatic volatile compounds in cooled (@ 4°C) water samples, the maximum holding time is 14 days from sample collection.

  Maximum holding times for purgeable aromatic hydrocarbons in cooled (@ 4°C ± 2°C), acid-preserved (pH 2 or below) water samples is 14 days from sample collection.

  Water samples that have not been maintained at 4°C (± 2°C) and preserved to a pH of 2 or below should be analyzed within 7 days from sample collection. If insufficient ice is used to ship samples, the laboratory may receive samples with no ice left in the cooler. Under these circumstanes, the temperature of the samples may exceed 4°C.

It is further recommended that volatile compounds in properly preserved non-aqueous samples be analyzed within 14 days of sample collection.

The contractual maximum holding times, which differ from the technical maximum holding times, state that water and soil samples are to be analyzed within 10 days from the validated time of sample receipt (VTSR) at the laboratory.

D. Evaluation

Technical holding times are established by comparing the sampling dates on the EPA Sample Traffic Report with dates of analysis on Form I VOA *[Form I LCV]* and the raw data. Information contained in the complete SDG file (formerly called the purge file) should also be considered in the determination of holding times. Verify that the analysis dates on the Form Is and the raw data/SDG file are identical. Examine the sample records to determine if samples were preserved. If adequate documentation on sample preservation is not available, contact the sampler. If the sampler cannot be contacted, then it must be assumed that the samples are unpreserved. If there is no indication in the SDG narrative or the sample records that there was a problem with the samples (e.g., samples not maintained @ 4°C or containing headspace in the samples), then the integrity of samples can be assumed to be good. If it is indicated that there were problems with the samples, then the integrity of the sample may have been compromised and professional judgement should be used to evaluate the effect of the problem on the sample results.

E. **Action**

1. If technical holding times are exceeded, document in the data review narrative that holding times were exceeded and qualify the sample results as follows (also see Table 1):

    a. If there is no evidence that the samples were properly preserved and the technical holding times exceeded 7 days, qualify positive results for aromatic compounds with "J" and sample quantitation limits with "UJ". Use professional judgement to determine if and how non-aromatic volatile compounds should also be qualified.

    b. If the samples were properly preserved but the technical holding times exceeded 14 days, qualify positive results with "J" and sample quantitation limits with "UJ".

Table 1. Qualification of Volatile Analytes Based on Technical Holding Times

MATRIX	PRESERVED	> 7 DAYS	> 14 DAYS
Water	No	All Aromatics*	All Compounds
	Yes	None	All Compounds
Non-aqueous	No/Yes	Professional Judgement	Professional Judgement

* Reviewer should use professional judgement to determine if data for additional compounds require qualification.

2. If technical holding times are grossly exceeded (e.g., by greater than two times the required time for volatiles) either on the first analysis or upon re-analysis, the reviewer must use professional judgement to determine the reliability of the data and the effects of additional storage on the sample results. Should the reviewer determine that qualification is necessary, non-detected volatile target compounds may be qualified unusable (R). Positive results are considered approximates and are qualified with "J".

3. Due to limited information concerning holding times for non-aqueous samples, it is left to the discretion of the data reviewer to apply water holding times or other information that is available.

4. Whenever possible, the reviewer should comment on the effect of the holding time exceedance on the resulting data in the data review narrative.

5. When contractual and/or technical holding times are grossly exceeded, this should be noted for TPO action.

6. The reviewer should also be aware of the scenario in which the laboratory has exceeded the technical holding times, but met contractual holding times. In this case, the data reviewer should notify the Regional TPO (where samples were collected) and/or RSCC that shipment delays have occurred so that the field problem can be corrected. The reviewer may pass this information on to the laboratory's TPO, but should explain that contractually the laboratory met the requirements.

II. GC/MS Instrument Performance Check

A. Review Items: Form V VOA *[Form V LCV]*. BFB mass spectra and mass listing.

B. **Objective**

Gas chromatograph/mass spectrometer (GC/MS) instrument performance checks (formerly referred to as tuning) are performed to ensure mass resolution, identification, and to some degree, sensitivity. These criteria are not sample specific. Conformance is determined using standard materials, therefore, these criteria should be met in all circumstances.

C. **Criteria**

The analysis of the instrument performance check solution must be performed at the beginning of each 12-hour period during which samples or standards are analyzed. The instrument performance check, bromofluorobenzene (BFB) for volatile analysis, must meet the ion abundance criteria given below.

Bromofluorobenzene (BFB)

m/z	ION ABUNDANCE CRITERIA
50	8.0 - 40.0% of m/z 95
75	30.0 - 66.0% of m/z 95
95	Base peak, 100% relative abundance
96	5.0 - 9.0% of m/z 95
173	Less than 2.0% of m/z 174
174	50.0 - 120.0% of m/z 95
175	4.0 - 9.0% of mass 174
176	93.0 - 101.0% of m/z 174
177	5.0 - 9.0% of m/z 176

NOTE: All ion abundances must be normalized to m/z 95, the nominal base peak, even though the ion abundance of m/z 174 may be up to 120 percent that of m/z 95.

D. **Evaluation**

1. **Compare the data presented for each Instrument Performance Check (Form V VOA *[Form V LCV]*) with each mass listing submitted to ensure the following:**

   a. **Form V VOA *[Form V LCV]* is present and completed for each 12-hour period during which samples were analyzed.**

b. The laboratory has not made transcription errors between the data and the form. If there are major differences between the mass listing and the Form Vs, a more in-depth review of the data is required. This may include obtaining and reviewing additional information from the laboratory.

c. The appropriate number of significant figures has been reported (number of significant figures given for each ion in the ion abundance criteria column) and that rounding is correct.

d. The laboratory has not made calculation errors.

2. Verify from the raw data (mass spectral listing) that the mass assignment is correct and that the mass listing is normalized to m/z 95.

3. Verify that the ion abundance criteria was met. The criteria for m/z 173, 176, and 177 are calculated by normalizing to the specified m/z.

4. If possible, verify that spectra were generated using appropriate background subtraction techniques. Since the BFB spectrum is obtained from chromatographic peaks that should be free from coelution problems, background subtraction should be done in accordance with the following procedure. Three scans (the peak apex scan and the scans immediately preceding and following the apex) are acquired and averaged and background subtraction must be accomplished using a single scan prior to the elution of BFB.

NOTE: All instrument conditions must be identical to those used in the sample analysis. Background subtraction actions resulting in spectral distortions for the sole purpose of meeting the contract specifications are contrary to the quality assurance objectives and are therefore unacceptable.

E. Action

1. If the laboratory has made minor transcription errors which do not significantly affect the data, the data reviewer should make the necessary corrections on a copy of the form.

2. If the laboratory has failed to provide the correct forms or has made significant transcription or calculation errors, the Region's designated representative should contact the laboratory and request corrected data. If the information is not available, then the reviewer must use professional judgement to assess the data. The laboratory's TPO should be notified.

3. If mass assignment is in error (such as m/z 96 is indicated as the base peak rather than m/z 95), classify all associated data as unusable (R).

4. If ion abundance criteria are not met, professional judgement may be applied to determine to what extent the data may be utilized. Guidelines to aid in the application of professional judgement to this topic are discussed as follows:

The most important factors to consider are the empirical results that are relatively insensitive to location on the chromatographic profile and the type of instrumentation. Therefore, the critical ion abundance criteria for BFB are the m/z 95/96, 174/175, 174/176, and 176/177 ratios. The relative abundances of m/z 50 and 75 are of lower importance.

5. Decisions to use analytical data associated with BFB instrument performance checks not meeting contract requirements should be clearly noted on the data review narrative.

6. If the reviewer has reason to believe that instrument performance check criteria were achieved using techniques other than those described in II.D.4, then additional information on the instrument performance checks should be obtained. If the techniques employed are found to be at variance with the contract requirements, the performance and procedures of the laboratory may merit evaluation. Concerns or questions regarding laboratory performance should be noted for TPO action. For example, if the reviewer has reason to believe that an inappropriate technique was used to obtain background subtraction (such as background subtracting from the solvent front or from another region of the chromatogram rather than the BFB peak), then this should be noted for TPO action.

III. Initial Calibration

A. **Review Items:** Form VI VOA *[Form VI LCV]*, quantitation reports, and chromatograms.

B. **Objective**

Compliance requirements for satisfactory instrument calibration are established to ensure that the instrument is capable of producing acceptable qualitative and quantitative data for compounds on the volatile target compound list (TCL). Initial calibration demonstrates that the instrument is capable of acceptable performance in the beginning of the analytical run and of producing a linear calibration curve.

C. **Criteria**

1. Initial calibration standards containing both volatile target compounds and system monitoring compounds are analyzed at concentrations of 10, 20, 50, 100, and 200 ug/L at the beginning of each analytical sequence or as necessary if the continuing calibration acceptance criteria are not met. The initial calibration (and any associated samples and blanks) must be analyzed within 12 hours of the associated instrument performance check.

   [For data generated through the Low Concentration Water SOW: Initial calibration standards containing both volatile target compounds and surrogate are analyzed at concentrations of 1, 2, 5, 10, and 25 ug/L for non-ketones and 5, 10, 25, 50, and 125 ug/L for ketones at the beginning of each analytical sequence or as necessary if the continuing calibration acceptance criteria are not met. The initial calibration (and any associated samples and blanks) must be analyzed within 12 hours of the associated BFB tuning check.]

2. Separate initial calibrations must be performed for water samples (or medium level soil samples) and for low level soil samples. The calibration for water samples and medium level soil samples is performed with an unheated purge and the calibration for low level soil samples is performed with a heated purge.

3. Initial calibration standard Relative Response Factors (RRFs) for all volatile target compounds and system monitoring compounds (surrogates) must be greater than or equal to 0.05. (Contractual initial calibration RRF criteria are listed in Appendix A *[Appendix B]*)

4. The Percent Relative Standard Deviation (%RSD) from the initial calibration must be less than or equal to 30.0% for all compounds.

D. **Evaluation**

1. Verify that the correct concentration of standards were used for the initial calibration (i.e., 10, 20, 50, 100, and 200 ug/L for water).

   [Verify that the correct concentration of standards were used for the initial calibration (i.e., 1, 2, 5, 10, and 25 ug/L for non-ketones and 5, 10, 25, 50, and 125 ug/L for ketones).]

2. Verify that the correct initial calibration was used for water and medium level soil samples (i.e., unheated purge) and for low level soil samples (i.e., heated purge).

3. If any sample results were calculated using an initial calibration, verify that the correct standard (i.e., the 50 ug/L standard) was used for calculating sample results and that the samples were analyzed within 12 hours of the associated instrument performance check.

   [If any sample results were calculated using an initial calibration, verify that the correct standard (i.e., the 5 ug/L for non-ketones and 25 ug/L for ketones) was used for calculating sample results and that the samples were analyzed within 12 hours of the associated instrument performance check.]

4. Evaluate the initial calibration RRFs and $\overline{RRF}$ for all volatile target compounds and system monitoring compounds (surrogates):

   a. Check and recalculate the RRFs and $\overline{RRF}$ for at least one volatile target compound associated with each internal standard; verify that the recalculated value(s) agrees with the laboratory reported value(s).

   b. Verify that for all volatile target compounds and system monitoring compounds (surrogates), the initial calibration RRFs are greater than or equal to 0.05.

NOTE: Because historical performance data indicate poor response and/or erratic behavior, the volatile compounds in Table 2 have no contractual maximum %RSD criteria. Contractually they must meet a minimum RRF criterion of 0.01, however, **for data review purposes, the "greater than or equal to 0.05" criterion is applied to all volatile compounds.**

Table 2. Volatile Target Compounds Exhibiting Poor Response

Acetone
2-Butanone
Carbon disulfide
Chloroethane
Chloromethane
1,2-Dichloroethene (total) †
trans-1,2-Dichloroethene ‡
cis-1,2-Dichloroethene ‡
1,2-Dichloropropane
2-Hexanone
Methylene chloride
4-Methyl-2-pentanone
Toluene-d8 †
1,2-Dichloroethane-d4 †
1,2-Dibromo-3-chloropropane ‡

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† Multi-media, Multi-concentration only
‡ Low Concentration Water only

5. **Evaluate the %RSD for all volatile target compounds and system monitoring compounds (surrogates):**

   a. **Check and recalculate the %RSD for one or more volatile target compound(s); verify that the recalculated value(s) agrees with the laboratory reported value(s).**

b. Verify that all volatile target compounds have a %RSD of less than or equal to 30.0%. The contractual criteria for an acceptable initial calibration specifies that up to any 2 volatile target compounds may fail to meet minimum RRF or maximum %RSD as long as they have RRFs that are greater than or equal to 0.010, and %RSD of less than or equal to 40.0%. For data review purposes, however, all compounds must be considered for qualification when the %RSD exceeds the ± 30.0% criterion.

c. If the %RSD is greater than 30.0%, then the reviewer should use professional judgement to determine the need to check the points on the curve for the cause of the non-linearity. This is checked by eliminating either the high point or the low point and recalculating the %RSD.

6. If errors are detected in the calculations of either the RRFs or the %RSD, perform a more comprehensive recalculation.

E. Action

1. **All volatile target compounds, including the 12 "poor performers" will be qualified using the following criteria:**

   a. **If the %RSD is greater than 30.0% and all initial calibration RRFs greater than or equal to 0.05, qualify positive results with "J", and non-detected volatile target compounds using professional judgement.**

   b. **If any initial calibration RRF is less than 0.05, qualify positive results that have acceptable mass spectral identification with "J", using professional judgement, and non-detected analytes as unusable (R).**

2. At the reviewer's discretion, a more in-depth review to minimize the qualification of data can be accomplished by considering the following:

   a. If any of the required volatile compounds have a %RSD greater than 30.0%, and if eliminating either the high or the low point of the curve does not restore the %RSD to less than or equal to 30.0%:

      i. **Qualify positive results for that compound(s) with "J".**

      ii. **Qualify non-detected volatile target compounds based on professional judgement.**

   b. **If the high point of the curve is outside of the linearity criteria (e.g. due to saturation):**

      i. **No qualifiers are required for positive results in the linear portion of the curve.**

   ii. Qualify positive results outside of the linear portion of the curve with a "J".

   iii. No qualifiers are needed volatile target compounds that were not detected.

  c. If the low end of the curve is outside of the linearity criteria:

   i. No qualifiers are required for positive results in the linear portion of the curve.

   ii. Qualify low level positive results in the area of non-linearity with "J".

   iii. Qualify non-detected volatile target compounds based on professional judgement.

3. If the laboratory has failed to provide adequate calibration information, the designated representative should contact the laboratory and request the necessary information. If the information is not available, the reviewer must use professional judgement to assess the data.

4. Whenever possible, the potential effects on the data due to calibration criteria exceedance should be noted in the data review narrative.

5. If calibration criteria are grossly exceeded, this should be noted for TPO action.

IV. Continuing Calibration

A. **Review Items:** Form VII VOA *[Form VII LCV]*, quantitation reports, and chromatograms.

B. **Objective**

Compliance requirements for satisfactory instrument calibration are established to ensure that the instrument is capable of producing acceptable qualitative and quantitative data. Continuing calibration establishes the 12-hour relative response factors on which the quantitations are based and checks satisfactory performance of the instrument on a day-to-day basis.

C. **Criteria**

1. Continuing calibration standards containing both target compounds and system monitoring compounds (surrogates) are analyzed at the beginning of each 12-hour analysis period following the analysis of the instrument performance check and prior to the analysis of the method blank and samples.

2. The continuing calibration RRF for volatile target compounds and system monitoring compounds (surrogates) must be greater than or equal to 0.05.

3. The percent difference (%D) between the initial calibration $\overline{\text{RRF}}$ and the continuing calibration RRF must be within ± 25.0%.

[For data generated through the Low Concentration Water SOW: The percent difference (%D) between the initial calibration $\overline{\text{RRF}}$ and the continuing calibration RRF must be within ± 30.0%.]

D. **Evaluation**

1. Verify that the continuing calibration was run at the required frequency and that the continuing calibration was compared to the correct initial calibration.

2. Evaluate the continuing calibration RRF for all volatile target compounds and system monitoring compounds:

   a. Check and recalculate the continuing calibration RRF for at least one volatile target compounds associated with each internal standard; verify that the recalculated value(s) agrees with the laboratory reported value(s).

   b. Verify that all volatile target compounds and system monitoring compounds meet the RRF specifications.

<u>NOTE</u>: Because historical performance data indicate poor response and/or erratic behavior, the compounds listed in Table 2 (Section III.D.4) have no contractual maximum %D criteria. Contractually they must meet a minimum RRF criterion of 0.01, however, **for data review purposes, the "greater than or equal to 0.05" criterion is applied to all volatile compounds.**

3. Evaluate the %D between initial calibration $\overline{RRF}$ and continuing calibration RRF for one or more compound(s).

   a. Check and recalculate the %D for one or more volatile target compound(s) associated with each internal standard; verify that the recalculated value(s) agrees with the laboratory reported value(s).

   b. Verify that the %D is within ± 25.0% for all volatile target compounds and system monitoring compounds. Note those compounds which have a %D outside the ± 25.0% criterion. The contractual criteria for an acceptable continuing calibration specifies that up to any 2 volatile target compounds may fail to meet minimum RRF or maximum %D as long as they have RRFs that are greater than or equal to 0.010, and %D of less than or equal to 40.0%. For data review purposes, however, all compounds must be considered for qualification when the %D exceeds the ± 25.0% criterion.

4. If errors are detected in the calculations of either the continuing calibration RRF or the %D, perform a more comprehensive recalculation.

E. Action

1. The reviewer should use professional judgement to determine if it is necessary to qualify the data for any volatile target compound. If qualification of data is required, it should be performed using the following guidelines:

   a. If the %D is outside the ± 25.0% criterion and the continuing calibration RRF is greater than or equal to 0.05, qualify positive results with "J".

   b. If the %D is outside the ± 25.0% criterion and the continuing calibration RRF is greater than or equal to 0.05, qualify non-detected volatile target compounds with "UJ".

   c. If the continuing calibration RRF is less than 0.05, qualify positive results that have acceptable mass spectral identifications with "J" or use professional judgement.

   d. If the continuing calibration RRF is less than 0.05, qualify non-detected volatile target compounds as unusable (R).

2. If the laboratory has failed to provide adequate calibration information, the designated representative should contact the laboratory and request the necessary information. If the information is not available, the reviewer must use professional judgement to assess the data.

3. Whenever possible, the potential effects on the data due to calibration criteria exceedance should be noted in the data review narrative.

4. If calibration criteria are grossly exceeded, this should be noted for TPO action.

V. Blanks

A. **Review Items:** Form I VOA *[Form I LCV]*, Form IV VOA *[Form IV LCV]*, chromatograms, and quantitation reports.

B. **Objective**

The purpose of laboratory (or field) blank analysis is to determine the existence and magnitude of contamination resulting from laboratory (or field) activities. The criteria for evaluation of blanks apply to any blank associated with the samples (e.g., method blanks, instrument blanks, trip blanks, and equipment blanks). If problems with any blank exist, all associated data must be carefully evaluated to determine whether or not there is an inherent variability in the data, or if the problem is an isolated occurrence not affecting other data.

C. **Criteria**

1. No contaminants should be found in the blanks.

2. A method blank analysis must be performed after the calibration standards and once for every 12-hour time period beginning with the injection of BFB.

3. The method blank must be analyzed on each GC/MS system used to analyze samples for each type of analysis, i.e., unheated purge (water and medium level soil) and heated purge (low level soil).

4. An instrument blank should be analyzed after any sample that has saturated ions from a given compound to check that the blank is free of interference and the system is not contaminated.

[5. *For data generated through the Low Concentration Water SOW: A storage blank must be prepared upon receipt of the first samples from an SDG, and stored with samples until analysis. The storage blank must be analyzed once per SDG.]*

D. **Evaluation**

1. Review the results of all associated blanks on the forms and raw data (chromatograms and quantitation reports) to evaluate the presence of target and non-target compounds in the blanks.

2. Verify that a method blank analysis has been reported per matrix, per concentration level, for each 12-hour time period on each GC/MS system used to analyze volatile samples. The reviewer can use the Method Blank Summary (Form IV VOA *[Form IV LCV]*) to identify the samples associated with each method blank.

3. Verify that the instrument blank analysis has been performed following any sample analysis where a target analyte(s) is reported at high concentration(s).

[4. Verify that a storage blank has been analyzed and included with each SDG and that the storage blanks are free of contamination.]

E. Action

If the appropriate blanks were not analyzed with the frequency described in Criteria 2, 3, and 4, *[and 5]* then the data reviewer should use professional judgement to determine if the associated sample data should be qualified. The reviewer may need to obtain additional information from the laboratory. The situation should be noted for TPO action.

Action regarding unsuitable blank results depends on the circumstances and origin of the blank. Positive sample results should be reported unless the concentration of the compound in the sample is less than or equal to 10 times (10x) the amount in any blank for the common volatile laboratory contaminants (**methylene chloride, acetone, and 2-butanone**), or 5 times (5x) the amount for other volatile target compounds. In instances where more than one blank is associated with a given sample, qualification should be based upon a comparison with the associated blank having the highest concentration of a contaminant. The results must <u>not</u> be corrected by subtracting any blank value.

Specific actions are as follows:

1. If a volatile compound is found in a blank but <u>not</u> found in the sample, no action is taken. If the contaminants found are volatile target compounds (or interfering non-target compounds) at significant concentrations above the CRQL, then this should be noted for TPO action.

2. Any volatile compound detected in the sample (other than the common volatile laboratory contaminants), that was also detected in any associated blank, is qualified if the sample concentration is less than five times (5x) the blank concentration. The quantitation limit may also be elevated. Typically, the sample CRQL is elevated to the concentration found in the sample. The reviewer should use professional judgement to determine if further elevation of the CRQL is required. For the common volatile laboratory contaminants, the results are qualified by elevating the quantitation limit to the concentration found in the sample when the sample concentration is less than 10 times (10x) the blank concentration.

   The reviewer should note that blanks may not inv[illegible] the same weights, volumes, or dilution factors as the associated samples. These factors must be taken into consideration when applying the "5x" and "10x" criteria, such that a comparison of the total amount of contamination is actually made.

   Additionally, there may be instances where little or no contamination was present in the associated blanks, but qualification of the sample is deemed necessary. If the reviewer determines that the contamination is from a source other than the sample, he/she should qualify the data. Contamination introduced through dilution water is one example. Although it is not always possible to determine, instances of this occurring can be detected when contaminants are found in the diluted sample result, but are absent in the undiluted sample result. Since both results are not routinely reported, it may be impossible to verify this source of contamination. In this case, the "5x" or "10x" rules may not apply; the target compound should be reported as not detected, and an explanation of the data qualification should be provided in the data review narrative.

3. If gross contamination exists (i.e., saturated peaks by GC/MS), all affected compounds in the associated samples should be qualified as unusable (R) due to interference. This should be noted for TPO action if the contamination is suspected of having an effect on the sample results.

4. If inordinate numbers of other target compounds are found at low levels in the blank(s), it may be indicative of a problem and should be noted for TPO action.

5. The same consideration given to the target compounds should also be given to Tentatively Identified Compounds (TICs), which are found in both the sample and associated blank(s). (See VOA Section XII for TIC guidance.)

6. If an instrument blank was not analyzed following a sample analysis which contained an analyte(s) at high concentration(s), sample analysis results after the high concentration sample must be evaluated for carryover. Professional judgement should be used to determine if instrument cross-contamination has affected any positive compound identification(s). If instrument cross-contamination is suggested, then this should be noted for TPO action if the cross-contamination is suspected of having an effect on the sample results.

[7. *If contaminants are found in the storage blanks, the following action is recommended.*

   a. *The associated method blank data should be reviewed to determine if the contaminant(s) was also present in the method blank. If the analyte was present at a comparable level in the method blank, then the source of the contamination may be in the analytical system and the action recommended for the method blank would apply.*

   If the analyte was not present in the method blank, then the source of contamination may be in the storage and all associated samples should be considered for possible cross-contamination.

   b. *If the storage blank contains a volatile TCL compound(s) at a concentration greater than the CRQL, then all positive results for that compounds(s) should be qualified with "J". If the concentration level in the blank is significantly high, then positive sample results may require rejection and be qualified with "R". Non-detected volatile target compounds should not require qualification unless the contamination is so high that it interferes with the analysis of the non-detect compounds.]*

The following are examples of applying the blank qualification guidelines. Certain circumstances may warrant deviations from these guidelines.

Example 1: Sample result is greater than the Contract Required Quantitation Limit (CRQL), but is less than the 5x or 10x multiple of the blank result.

	Rule	
	10x	5x
Blank Result	7	7
CRQL	5	5
Sample Result	60	30
Final Sample Result	60U	30U

In the example for the "10x" rule, sample results less than 70 (or 10 x 7) would be qualified as not detected. In the case of the "5x" rule, sample results less than 35 (or 5 x 7) would be qualified as not detected.

Example 2: Sample result is less than the CRQL, and is also less than the 5x or 10x multiple of the blank result.

	Rule	
	10x	5x
Blank Result	6	6
CRQL	5	5
Sample Result	4J	4J
Final Sample Result	5U	5U

Note that data are not reported as 4U, as this would be reported as a detection limit below the CRQL.

Example 3: Sample result is greater than the 5x or 10x multiple of the blank result.

	Rule	
	10x	5x
Blank Result	10	10
CRQL	5	5
Sample Result	120	60
Final Sample Result	120	60

For both the "10x" and "5x" rules, sample results exceeded the adjusted blank results of 100 (or 10x10) and 50 (or 5x10), respectively.

VI. System Monitoring Compounds
(Surrogate Spikes)

A. **Review Items:** Form II VOA *[Form II LCV]*, quantitation reports, and chromatograms.

B. **Objective**

Laboratory performance on individual samples is established by means of spiking activities. All samples are spiked with system monitoring compounds (formerly referred to as surrogates) prior to sample purging. The evaluation of the results of these system monitoring compounds is not necessarily straightforward. The sample itself may produce effects due to such factors as interferences and high concentrations of analytes. Since the effects of the sample matrix are frequently outside the control of the laboratory and may present relatively unique problems, the evaluation and review of data based on specific sample results is frequently subjective and demands analytical experience and professional judgement. Accordingly, this section consists primarily of guidelines, in some cases with several optional approaches suggested.

C. **Criteria**

1. Three system monitoring compounds (1,2-dichloroethane-d4, bromofluorobenzene, and toluene-d8) are added to all samples and blanks to measure their recovery in environmental samples in sample and blank matrices.

   [For data generated through the Low Concentration Water SOW: A single surrogate, bromofluorobenzene, is added to all samples and blanks to measure the recovery in sample and blank matrices.]

2. Recoveries for system monitoring compounds *[surrogates]* in volatile samples and blanks must be within the limits specified in Appendix A *[Appendix B]* and the SOW.

D. **Evaluation**

1. Check raw data (e.g., chromatograms and quantitation reports) to verify the recoveries on the System Monitoring Compound Recovery Form - Form II VOA *[Surrogate Recovery Form - Form II LCV]*. Check for any calculation or transcription errors.

2. Check that the system monitoring compound *[surrogate]* recoveries were calculated correctly. The equation can be found in Appendix [illegible] *[Appendix B]*.

3. The following should be determined from the System Monitoring Compound *[Surrogate]* Recovery form(s):

   a. If any system monitoring *[surrogate]* compound(s) in the volatile fraction is out of specification, there should be a reanalysis to confirm that the non-compliance is due to sample matrix effects rather than laboratory deficiencies.

<u>NOTE</u>: When there are unacceptable system monitoring compound *[surrogate]* recoveries followed by successful re-analyses, the laboratories are required to report only the successful run.

b. The laboratory failed to perform acceptably if system monitoring compounds *[surrogate]* are outside criteria with no evidence of re-analysis. Medium soils must first be re-extractracted prior to re-analysis when this occurs.

c. Verify that no blanks have system monitoring compounds *[surrogate]* outside the criteria.

4. Any time there are two or more analyses for a particular sample, the reviewer must determine which are the best data to report. Considerations should include but are not limited to:

   a. System monitoring compound *[surrogate]* recovery (marginal versus gross deviation).

   b. Technical holding times.

   c. Comparison of the values of the target compounds reported in each sample analysis.

   d. Other QC information, such as performance of internal standards.

E. Action

Data are qualified based on system monitoring compounds *[surrogate]* results if the recovery of any volatile system monitoring compound *[surrogate]* is out of specification. For system monitoring compound *[surrogate]* recoveries out of specification, the following approaches are suggested based on a review of all data from the package, especially considering the apparent complexity of the sample matrix.

1. If a system monitoring compound *[surrogate]* in the volatile sample has a recovery greater than the upper acceptance limit (UL):

   a. Detected volatile target compounds are qualified "J.".

   b. Results for non-detected volatile target compounds should not be qualified.

2. If a system monitoring compound *[surrogate]* in the volatile sample has a recovery greater than or equal to 10% but less than the lower acceptance limit (LL):

   a. Detected volatile target compounds are qualified "J."

   b. For non-detected volatile target compounds, the sample quantitation limit is qualified as approximated (UJ).

3. If a system monitoring compound *[surrogate]* in a volatile sample shows less than 10% recovery:

   a. Detected volatile target compounds are qualified "J".

   b. Non-detected volatile target compounds may be qualified as unusable (R).

Table 3. Qualification of Volatile Analytes Based on System Monitoring Compound *[Surrogate]* Recoveries

	SMC/Surrogate Recovery		
	> UL	10% to LL	< 10%
Detected analytes	J	J	J
Non-detected analytes	No Qualification	UJ	R

4. In the special case of a blank analysis with system monitoring compounds *[surrogate]* out of specification, the reviewer must give special consideration to the validity of associated sample data. The basic concern is whether the blank problems represent an isolated problem with the blank alone, or whether there is a fundamental problem with the analytical process. For example, if one or more samples in the batch show acceptable system monitoring compound *[surrogate]* recoveries, the reviewer may choose to consider the blank problem to be an isolated occurrence. However, even if this judgment allows some use of the affected data, analytical problems should be noted for TPO action. Also note if there are potential contractual problems associated with the lack of reanalysis of samples that were out of specification.

5. Whenever possible, potential effects of the data resulting from system monitoring recoveries not meeting the advisory limits should be noted in the data review narrative.

VII. Matrix Spikes/Matrix Spike Duplicates
(Not Required for Low Concentration Water Data)

A. **Review Items:** Form III VOA-1 and VOA-2, chromatograms, and quantitation reports.

B. **Objective**

Data for matrix spike/matrix spike duplicates (MS/MSD) are generated to determine long-term precision and accuracy of the analytical method on various matrices and to demonstrate acceptable compound recovery by the laboratory at the time of sample analysis. These data alone cannot be used to evaluate the precision and accuracy of individual samples. However, when exercising professional judgement, this data should be used in conjunction with other available QC information.

C. **Criteria**

1. Matrix spikes (MS) and matrix spike duplicate (MSD) samples are analyzed at frequency of one MS and MSD per 20 samples of similar matrix.
2. Spike recoveries should be within the advisory limits provided on Form III VOA-1 and 2.
3. Relative percent difference (RPD) between MS and MSD recoveries must be within the advisory limits provided on Form III VOA-1 and VOA-2.

D. **Evaluation**

1. Verify that MS and MSD samples were analyzed at the required frequency and that results are provided for each sample matrix.
2. Inspect results for the MS/MSD Recovery on Form III VOA-1 and VOA-2 and verify that the results for recovery and RPD are within the advisory limits.
3. Verify transcriptions from raw data and verify calculations.
4. Check that the matrix spike recoveries and RPD were calculated correctly.
5. Compare %RSD results of non-spiked compounds between the original result, MS, and MSD.

E. **Action**

1. No action is taken on MS/MSD data alone. However, using informed professional judgment the data reviewer may use the MS and MSD results in conjunction with other QC criteria and determine the need for some qualification of the data.

2. The data reviewer should first try to determine to what extent the results of the MS/MSD affect the associated data. This determination should be made with regard to the MS/MSD sample itself as well as specific analytes for all samples associated with the MS/MSD.

3. In those instances where it can be determined that the results of the MS/MSD affect only the sample spiked, then qualification should be limited to this sample alone. However, it may be determined through the MS/MSD results that a laboratory is having a systematic problem in the analysis of one or more analytes, which affects all associated samples.

4. The reviewer must use professional judgement to determine the need for qualification of positive results of non-spiked compounds.

<u>NOTE</u>: If a field blank was used for the MS/MSD, a statement to that effect must be included for the TPO.

VIII. Laboratory Control Samples
(Low Concentration Water)

A. *Review Items: Form III LCV-1, LCS chromatograms and quantitation reports*

B. *Objective*

Data for laboratory control samples (LCS) are generated to provide information on the accuracy of the analytical method and on the laboratory performance.

C. *Criteria*

1. *A laboratory control sample (LCS) must be analyzed once per SDG and concurrently with the samples in the SDG.*

2. *The LCS contains the following volatile compounds, in addition to the required surrogate:*

   Vinyl chloride
   1,2-Dichloroethane
   Carbon tetrachloride
   1,2-Dichloropropane
   Trichloroethene
   1,1,2-Trichloroethane
   Benzene
   cis-1,3-Dichloropropene
   Bromoform
   Tetrachloroethene
   1,2-Dibromoethane
   1,4-Dichlorobenzene

3. *The percent recoveries for the LCS compounds must be within 60-140%. The LCS must meet this recovery criteria for the sample data to be accepted.*

4. *The criteria for surrogate recovery and internal standard performance also apply.*

D. *Evaluation*

1. *Verify that LCS samples were analyzed at the required frequency and that results are provided for each SDG.*

2. *Inspect results for the LCS Recovery on Form III LCV-1 and verify that the results for recovery are within the QC limits of 60 to 140 percent.*

3. *Verify transcriptions from raw data and verify calculations.*

4. *Check that the LCS recovery was calculated correctly by using the correct equation.*

E. *Action*

If the LCS criteria are not met, then the laboratory performance and method accuracy are in question. Professional judgement should be used to determine if the data should be qualified or rejected. The following guidance is suggested for qualifying sample data for which the associated LCS does not meet the required criteria.

1. *Action on the LCS recovery should be based on both the number of compounds that are outside of the recovery criteria and the magnitude of the exceedance of the criteria.*

2. *If the LCS recovery criteria are not met, then the LCS results should be used to qualify sample data for the specific compounds that are included in the LCS solution. Professional judgement should be used to*

qualify data for compounds other than those compounds that are included in the LCS. Professional judgement to qualify non-LCS compounds should take into account the compound class, compound recovery efficiency, analytical problems associated with each compound, and comparability in performance of the LCS compound to the non-LCS compound.

3. *If the LCS recovery is greater than 140%, then positive sample results for the affected compound(s) should be qualified with a "J".*

4. *If the mass spectral criteria are met but the LCS recovery is less than 60%, then the associated detected target compounds should be qualified "J" and the associated non-detected target compounds should be qualified "R".*

5. *If more than half of the compounds in the LCS are not within the required recovery criteria, then all of the associated detected target compounds should be qualified "J" and all associated non-detected target compounds should be qualified "R."*

6. *Action on non-compliant surrogate recovery and internal standard performance should follow the procedures provided in VI.E and X.E, respectively. Professional judgement should be used to evaluate the impact that non-compliance for surrogate recovery and internal standard performance in the LCS has on the associated sample data.*

7. *It should be noted for TPO action if a laboratory fails to analyze an LCS with each SDG, or if a laboratory consistently fails to generate acceptable LCS recoveries.]*

IX. Regional Quality Assurance and Quality Control

A. **Review Items:** Form I VOA *[Form I LCV]*, chromatograms, and quantitation reports.

B. **Objective**

Regional Quality Assurance and Quality Control (QA/QC) refer to any QA and/or QC samples initiated by the Region, including field duplicates, Performance Evaluation (PE) samples, blind spikes, and blind blanks. It is highly recommended that Regions adopt the use of these.

C. **Criteria**

Criteria are determined by each Region.

1. Performance evaluation sample frequency may vary.

   [For data generated through the Low Concentration Water SOW: A performance evaluation (PE) sample may be required as frequently as once per SDG.]

2. The analytes present in the PE sample must be correctly identified and quantified.

D. **Evaluation**

Evaluation procedures must follow the Region's SOP for data review. Each Region will handle the evaluation of PE samples on an individual basis. Results for PE samples should be compared to the acceptance criteria for the specific PE samples, if available.

E. **Action**

Any action must be in accordance with Regional specifications and the criteria for acceptable PE sample results. Unacceptable results for PE samples should be noted for TPO action.

X. Internal Standards

A. **Review Items:** Form VIII VOA *[Form VIII LCV]*, quantitation reports, and chromatograms

B. **Objective**

Internal Standards (IS) performance criteria ensures that GC/MS sensitivity and response are stable during each analysis.

C. **Criteria**

1. Internal standard area counts must not vary by more than a factor of two (-50% to +100%) from the associated calibration standard.

   [For data generated through the Low Concentration Water SOW: Internal standard area counts must not vary by more than a factor of ± 40.0% from the associated calibration standard.]

2. The retention time of the internal standard must not vary more than ±30 seconds from the retention time of the associated calibration standard.

   [For data generated through the Low Concentration Water SOW: The retention time of the internal standard must not vary more than ± 20.0 seconds from the retention time of the associated calibration standard.]

D. **Evaluation**

1. Check raw data (e.g., chromatograms and quantitation lists) to verify the internal standard retention times and areas reported on the Internal Standard Area Summary (Form VIII VOA *[Form VIII LCV]*).

2. Verify that all retention times and IS areas are within criteria.

3. If there are two analyses for a particular fraction, the reviewer must determine which are the best data to report. Considerations should include:

   a. Magnitude and direction of the IS area shift.

   b. Magnitude and direction of the IS retention time shift.

   c. Technical holding times.

   d. Comparison of the values of the target compounds reported in each fraction.

   e. Other QC.

E. Action

1. If an IS area count for a sample or blank is outside -50% or +100% of the area for associated standard:

   a. Positive results for compounds quantitated using that IS should be qualified with "J".

   b. Non-detected compounds quantitated using an IS area count greater than 100% should not be qualified.

   c. Non-detected compounds quantitated using an IS area count less than 50% are reported as the associated sample quantitation limit and qualified with "UJ".

   d. If extremely low area counts are reported, or if performance exhibits a major abrupt drop-off, then a severe loss of sensitivity is indicated. Non-detected target compounds should then be qualified as unusable (R).

   [If an IS area count for a sample or blank is outside ± 40.0% of the area for associated standard:

   a. Positive results for compounds quantitated using that IS should be qualified with "J".

   b. Non-detected compounds quantitated using an IS area count greater than 40% should not be qualified.

   c. Non-detected compounds quantitated using an IS area count less than 40% are reported as the associated sample quantitation limit and qualified with "UJ".

   d. If extremely low area counts are reported, or if performance exhibits a major abrupt drop-off, then a severe loss of sensitivity is indicated. Non-detected target compounds should then be qualified as unusable (R).]

2. If an IS retention time varies by more than 30 seconds:

   [If an IS retention time varies by more than 20.0 seconds:]

   The chromatographic profile for that sample must be examined to determine if any false positives or negatives exist. For shifts of a large magnitude, the reviewer may consider partial or total rejection of the data for that sample fraction. Positive results should not need to be qualified as "R" if the mass spectral criteria are met.

3. If the internal standards performance criteria are grossly exceeded, then this should be noted for TPO action. Potential effects on the data resulting from unacceptable internal standard performance should be noted in the data review narrative.

XI. Target Compound Identification

A. **Review Items:** Form I VOA *(Form I LCV)*, quantitation reports, mass spectra, and chromatograms.

B. **Objective**

The objective of the criteria for GC/MS qualitative analysis is to minimize the number of erroneous identifications of compounds. An erroneous identification can either be a false positive (reporting a compound present when it is not) or a false negative (not reporting a compound that is present).

The identification criteria can be applied more easily in detecting false positives than false negatives. More information is available for false positives due to the requirement for submittal of data supporting positive identifications. Negatives, or non-detected compounds, on the other hand represent an absence of data and are, therefore, more difficult to assess. One example of detecting false negatives is the not reporting of a Target Compound that is reported as a TIC.

C. **Criteria**

1. The relative retention times (RRTs) must be within ±0.06 RRT units of the standard RRT.

2. Mass spectra of the sample compound and a current laboratory-generated standard (i.e., the mass spectrum from the associated calibration standard) must match according to the following criteria:

   a. All ions present in the standard mass spectrum at a relative intensity greater than 10% must be present in the sample spectrum.

   [For data generated through the Low Concentration Water SOW: All ions present in the standard mass spectrum at a relative intensity greater than 25% must be present in the sample spectrum.]

   b. The relative intensities of these ions must agree within ± 20% between the standard and sample spectra. (Example: For an ion with an abundance of 50% in the standard spectrum, the corresponding sample ion abundance must be between 30% and 70%.)

   c. Ions present at greater than 10% in the sample mass spectrum but not present in the standard spectrum must be considered and accounted for.

   [For data generated through the Low Concentration Water SOW: Ions present at greater than 25% in the sample mass spectrum but not present in the standard spectrum must be considered and accounted for.]

D. Evaluation

1. Check that the RRT of reported compounds is within ± 0.06 RRT units of the standard RRT.

2. Check the sample compound spectra against the laboratory standard spectra to see that it meets the specified criteria.

3. The reviewer should be aware of situations (e.g., high concentration samples preceding low concentration samples) when sample carryover is a possibility and should use judgment to determine if instrument cross-contamination has affected any positive compound identification. The SOW specifies that an instrument blank must be run after samples in which a target analyte ion(s) saturates the detector.

   [The reviewer should be aware of situations when sample carryover is a possibility and should use judgment to determine if instrument cross-contamination has affected any positive compound identification. The SOW specifies that an instrument blank must be run after samples which contain target compounds at levels exceeding the initial calibration range (25 ug/L for non-ketones, 125 ug/L for ketones) or non-target compounds at concentrations greater than 100 ug/L or saturated ions from a compound (excluding the compound peaks in the solvent front).]

4. Check the chromatogram to verify that peaks are accounted for, i.e., major peaks are either identified as target compounds, TICs, system monitoring compounds *[surrogate]*, or internal standards.

E. Action

1. The application of qualitative criteria for GC/MS analysis of target compounds requires professional judgement. It is up to the reviewer's discretion to obtain additional information from the laboratory. If it is determined that incorrect identifications were made, all such data should be qualified as not detected (U) or unusable (R).

2. Professional judgement must be used to qualify the data if it is determined that cross-contamination has occurred.

3. Any changes made to the reported compounds or concerns regarding target compound identifications should be clearly indicated in the data review narrative. The necessity for numerous or significant changes should be noted for TPO action.

XII. Compound Quantitation and Reported CRQLs

A. **Review Items:** Form I VOA *[Form I LCV]*, sample preparation sheets, SDG narrative, quantitation reports, and chromatograms.

B. **Objective**

The objective is to ensure that the reported quantitation results and Contract Required Quantitation Limits (CRQLs) are accurate.

C. **Criteria**

1. Compound quantitation, as well as the adjustment of the CRQLs, must be calculated according to the correct equation.

2. Compound RRFs must be calculated based on the internal standard (IS) associated with that compound, as listed in Appendix A *[Appendix B]* (also as specified in the Statement of Work) for packed column analyses. For analyses performed by capillary column method (EPA Method 524.2), the target compounds will not necessarily be associated with the same internal standard as in the packed column, depending on the compound elution order. Quantitation must be based on the quantitation ion (m/z) specified in the SOW for both the IS and target analytes. The compound quantitation must be based on the RRF from the appropriate daily standard.

D. **Evaluation**

1. For all fractions, raw data should be examined to verify the correct calculation of all sample results reported by the laboratory. Quantitation lists and chromatograms should be compared to the reported positive sample results and quantitation limits. Check the reported values.

2. Verify that the correct internal standard, quantitation ion, and RRF were used to quantitate the compound. Verify that the same internal standard, quantitation ion, and RRF are used consistently throughout, in both the calibration as well as the quantitation process. For analyses performed by capillary column, the reviewer should use professional judgement to determine that the laboratory has selected the appropriate internal standard.

3. Verify that the CRQLs have been adjusted to reflect all sample dilutions and dry weight factors that are not accounted for by the method.

E. **Action**

1. If any discrepancies are found, the laboratory may be contacted by the designated representative to obtain additional information that could resolve any differences. If a discrepancy remains unresolved, the reviewer must use professional judgement to decide which value is the best value. Under these circumstances, the reviewer may determine

qualification of data is warranted. A description of the reasons for data qualification and the qualification that is applied to the data should be documented in the data review narrative.

2. Numerous or significant failures to accurately quantify the target compound or to properly evaluate and adjust CRQLs should be noted for TPO action.

XIII. Tentatively Identified Compounds

A. **Review Items:** Form I VOA-TIC *[Form I LCV-TIC]*, chromatograms, and library search printout and spectra for three TIC candidates.

B. **Objective**

Chromatographic peaks in volatile fraction analyses that are not target analytes, system monitoring compounds *[surrogate]*, or internal standards are potential tentatively identified compounds (TICs). TICs must be qualitatively identified by a National Institute of Standards and Technology (NIST) mass spectral library search and the identifications assessed by the data reviewer.

C. **Criteria**

For each sample, the laboratory must conduct a mass spectral search of the NIST library and report the possible identity for the 10 largest volatile fraction peaks which are not system monitoring compound, internal standard, or target compounds, but which have area or height greater than 10 percent of the area or height of the nearest internal standard. TIC results are reported for each sample on the Organic Analyses Data Sheet (Form I VOA-TIC).

[For data generated through the Low Concentration Water SOW: For each sample, the laboratory must conduct a mass spectral search of the NIST library and report the possible identity for the 10 largest volatile fraction peaks which are not surrogate, internal standard, or TCL compounds, but which have area greater than or equal to 40 percent of the area of the nearest internal standard. Estimated concentrations for TICs are calculated similarly to the TCL compounds, using total ion areas for the TIC and the internal standard, and assuming a relative response factor of 1.0. TIC results are reported for each sample on the Organic Analyses Data Sheet (Form I LCV-TIC).]

NOTE: Since the SOW revision of October 1986, the CLP does not allow the laboratory to report as tentatively identified compounds any target compound which is properly reported in another fraction. For example, late eluting volatile target compounds should not be reported as semivolatile TICs.

D. **Evaluation**

1. Guidelines for tentative identification are as follows:

   a. Major ions (greater than 10% relative intensity) in the reference spectrum should be present in the sample spectrum.

   [Major ions (greater than 25% relative intensity) in the reference spectrum should be present in the sample spectrum.]

   b. The relative intensities of the major ions should agree within ±20% between the sample and the reference spectra.

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

d. Ions present in the sample spectrum but not in the reference spectrum should be reviewed for possible background contamination, interference, or coelution of additional TIC or target compounds.

e. When the above criteria are not met, but in the technical judgement of the data reviewer or mass spectral interpretation specialist the identification is correct, the data reviewer may report the identification.

f. If in the data reviewer's judgement the identification is uncertain or there are extenuating factors affecting compound identifications, the TIC result may be reported as "unknown".

2. Check the raw data to verify that the laboratory has generated a library search for all required peaks in the chromatograms for samples and blanks.

3. Blank chromatograms should be examined to verify that TIC peaks present in samples are not found in blanks. When a low-level non-target compound that is a common artifact or laboratory contaminant is detected in a sample, a thorough check of blank chromatograms may require looking for peaks which are less than 10 percent of the internal standard height, but present in the blank chromatogram at similar relative retention time.

[Blank chromatograms should be examined to verify that TIC peaks present in samples are not found in blanks. When a low-level non-TCL compound that is a common artifact or laboratory contaminant is detected in a sample, a thorough check of blank chromatograms may require looking for peaks which are less than 40 percent of the internal standard area but present in the blank chromatogram at similar relative retention time.]

4. All mass spectra for every sample and blank must be examined.

5. Since TIC library searches often yield several candidate compounds having a close matching score, all reasonable choices must be considered.

6. The reviewer should be aware of common laboratory artifacts/contaminants and their sources (e.g., aldol condensation products, solvent preservatives, and reagent contaminants). These may be present in blanks and not reported as sample TICs.

Examples:

a. Common laboratory contaminants: CO_2 (m/z 44), siloxanes (m/z 73), diethyl ether, hexane, certain freons (1,1,2-trichloro-1,2,2-trifluoroethane or fluorotrichloromethane), and phthalates at levels less than 100 ug/L or 4000 ug/Kg.

b. Solvent preservatives such as cyclohexene which is a methylene chloride preservative. Related by-products include cyclohexanone, cyclohexenone, cyclohexanol, cyclohexenol, chlorocyclohexene, and chlorocyclohexanol.

c. Aldol condensation reaction products of acetone include: 4-hydroxy-4-methyl-2-pentanone, 4-methyl-2-penten-2-one, and 5,5-dimethyl-2(5H)-furanone.

7. Occasionally, a target compound may be identified in the proper analytical fraction by non-target library search procedures, even though it was not found on the quantitation list. If the total area quantitation method was used, the reviewer should request that the laboratory recalculate the result using the proper quantitation ion. In addition, the reviewer should evaluate other sample chromatograms and check library reference retention times on quantitation lists to determine whether the false negative result is an isolated occurrence or whether additional data may be affected.

8. Target compounds could be identified in more than one fraction. Verify that quantitation is made from the proper fraction.

9. Library searches should not be performed on internal standards or system monitoring compounds.

10. TIC concentration should be estimated assuming a RRF of 1.0.

E. Action

1. All TIC results should be qualified "NJ", tentatively identified, with approximated concentrations.

2. General actions related to the review of TIC results are as follows:

   a. If it is determined that a tentative identification of a non-target compound is not acceptable, the tentative identification should be changed to "unknown" or an appropriate identification.

   b. If all contractually required peaks were not library searched and quantitated, the designated representative could request these data from the laboratory.

3. TIC results which are not sufficiently above 10x the level in the blank should not be reported. (Dilutions and sample size must be taken into account when comparing the amounts present in blanks and samples.)

4. When a compound is not found in any blanks, but is a suspected artifact of common laboratory contaminant, the result may be qualified as unusable (R).

5. In deciding whether a library search result for a TIC represents a reasonable identification, professional judgment must be exercised. If there is more than one possible match, the result may be reported as "either compound X or compound Y." If there is a lack of isomer specificity, the TIC result may be changed to a non-specific isomer result (e.g., 1,3,5-trimethyl benzene to trimethyl benzene isomer) or to a compound class (e.g., 2-methyl, 3-ethyl benzene to substituted aromatic compound).

6. The reviewer may elect to report all similar compounds as a total. (e.g., All alkanes may be summarized and reported as total hydrocarbons.)

7. Other case factors may influence TIC judgements. If a sample TIC match is poor but other samples have a TIC with a good library match, similar relative retention time, and the same ions, identification information may be inferred from the other sample TIC results.

8. Physical constants, such as boiling point, may be factored into professional judgement of TIC results.

9. Any changes made to the reported data or any concerns regarding TIC identifications should be indicated in the data review narrative.

10. Failure to properly evaluate and report TICs should be noted for TPO action.

XIV. System Performance

A. **Review Items:** Form VIII VOA *[Form VIII LCV]*, Form III VOA-1 and VOA-2 *[Form III LCSV]*, and chromatograms.

B. **Objective**

During the period following Instrument Performance QC checks (e.g. blanks, tuning, calibration), changes may occur in the system that degrade the quality of the data. While this degradation would not be directly shown by QC checks until the next required series of analytical QC runs, a thorough review of the ongoing data acquisition can yield indicators of instrument performance.

C. **Criteria**

There are no specific criteria for system performance. Professional judgement should be applied to assess the system performance.

D. **Evaluation**

1. Abrupt, discrete shifts in the reconstructed ion chromatogram (RIC) baseline may indicate a change in the instrument's sensitivity or the zero setting. A baseline "shift" could indicate a decrease in sensitivity in the instrument or an increase in the instrument zero, possibly causing target compounds, at or near the detection limit, to miss detection. A baseline "rise" could indicate problems such as a change in the instrument zero, a leak, or degradation of the column.

2. Poor chromatographic performance affects both qualitative and quantitative results. Indications of substandard performance include:

    a. High RIC background levels or shifts in absolute retention times of internal standards.

    b. Excessive baseline rise at elevated temperature.

    c. Extraneous peaks.

    d. Loss of resolution.

    e. Peak tailing or peak splitting that may result in inaccurate quantitation.

[3. A drift in instrument sensitivity may occur during the 12-hour time period. This could be discerned by examination of the IS area on Form VIII LCV for trends such as a continuous or near-continuous increase or decrease in the IS area over time.

4. The results of the LCS analysis (Form III LCSV) may also be used to assess instrument performance.]

E. Action

Professional judgement must be used to qualify the data if it is determined that system performance has degraded during sample analyses. Any degradation of system performance which significantly affected the data should be documented for TPO action.

XV. Overall Assessment of Data

A. **Review Items:** Entire data package, data review results, and (if available) Quality Assurance Project Plan (QAPjP), and Sampling and Analysis Plan (SAP).

B. **Objective**

The overall assessment of a data package is a brief narrative in which the data reviewer expresses concerns and comments on the quality and, if possible, the useability of the data.

C. **Criteria**

Assess the overall quality of the data.

Review all available materials to assess the overall quality of the data, keeping in mind the additive nature of analytical problems.

D. **Evaluation**

1. Evaluate any technical problems which have not been previously addressed.

2. If appropriate information is available, the reviewer may assess the useability of the data to assist the data user in avoiding inappropriate use of the data. Review all available information, including the QAPjP (specifically the Data Quality Objectives), SAP, and communication with data user that concerns the intended use and desired quality of these data.

E. **Action**

1. Use professional judgement to determine if there is any need to qualify data which were not qualified based on the QC criteria previously discussed.

2. Write a brief narrative to give the user an indication of the analytical limitations of the data. Any inconsistency of the data with the SDG narrative should be noted for TPO action. If sufficient information on the intended use and required quality of the data are available, the reviewer should include his/her assessment of the useability of the data within the given context.

SEMIVOLATILE DATA REVIEW

*** *Data review guidelines that are unique to data generated through the Low Concentration Water SOW are contained within brackets ([ ]) and written in italics.* ***

The semivolatile data requirements to be checked are listed below:

I. Technical Holding Times (CCS - Contractual holding times only)

II. GC/MS Instrument Performance Check (CCS)

III. Initial Calibration (CCS)

IV. Continuing Calibration (CCS)

V. Blanks (CCS)

VI. Surrogate Spikes (CCS)

VII. Matrix Spikes/Matrix Spike Duplicates

VIII. Laboratory Control Samples (CCS)

IX. Regional Quality Assurance and Quality Control

X. Internal Standards (CCS)

XI. Target Compound Identification

XII. Compound Quantitation and Reported Contract Required Quantitation Limits (CRQLs)

XIII. Tentatively Identified Compounds

XIV. System Performance (CCS)

XV. Overall Assessment of Data

<u>NOTE</u>: "CCS" indicates that the contractual requirements for these items will also be checked by CCS; CCS requirements are not always the same as the data review criteria.

I. Technical Holding Times

A. **Review Items:** Form I SV-1 and SV-2 *[Form I LCSV-1 and LCSV-2]*, EPA Sample Traffic Report and/or chain-of-custody, raw data, and sample extraction sheets.

B. **Objective**

The objective is to ascertain the validity of results based on the holding time of the sample from time of collection to time of sample extraction and analysis.

C. **Criteria**

Technical requirements for sample holding times have only been established for water matrices. The holding times for soils (and other non-aqueous matrices such as sediments, oily wastes, and sludge) are currently under investigation. When the results are available they will be incorporated into the data evaluation process. Additionally, results of holding time studies will be incorporated into the data review criteria as the studies are conducted and approved.

The holding time criteria for water samples, as stated in the current 40 CFR Part 136 (Clean Water Act) is as follows:

> For semivolatile compounds in cooled (@ 4°C) water samples the maximum holding time is 7 days from sample collection to extraction and 40 days from sample extraction to analysis.

It is recommended that semivolatile compounds in non-aqueous samples be extracted within 14 days of sample collection.

The contractual holding times, which differ from the technical holding times, state that water samples are to be extracted within 5 days from the validated time of sample receipt (VTSR) at the laboratory, and soil samples are to be extracted within 10 days from the VTSR. Also, contractually both water and soil sample extracts must be analyzed within 40 days of sample extraction. However, the contractual delivery due date is 35 days from the VTSR.

[For data generated through the Low Concentration SOW: The contractual delivery due date is 14 days from the VTSR.]

D. **Evaluation**

Technical holding times for sample extraction are established by comparing the sampling date on the EPA Sample Traffic Report with the dates of extraction on Form I SV-1 and SV-2 *[Form I LCSV-1 and LCSV-2]* and the sample extraction sheets. To determine if the samples were analyzed within the holding time after extraction, compare the dates of extraction on the sample extraction sheets with the dates of analysis on Form I SV-1 and SV-2 *[Form I LCSV-1 and LCSV-2]*.

Verify that the traffic report indicates that the samples were received intact and iced. If the samples were not iced or there were any problems with the samples upon receipt, then discrepancies in the sample condition could effect the data.

E. Action

1. If technical holding times are exceeded, flag all positive results as estimated "J" and sample quantitation limits as estimated "UJ" and document that holding times were exceeded.

2. If technical holding times are grossly exceeded, either on the first analysis or upon re-analysis, the reviewer must use professional judgement to determine the reliability of the data and the effects of additional storage on the sample results. The reviewer may determine that positive results or the associated quantitation limits are approximates and should be qualified with "J" or "UJ", respectively. The reviewer may determine that non-detect data are unusable (R).

3. Due to limited information concerning holding times for soil samples, it is left to the discretion of the data reviewer to apply water holding time criteria to soil samples. Professional judgement is required to evaluate holding times for soil samples.

4. Whenever possible, the reviewer should comment on the effect of the holding time exceedance on the resulting data in the data review narrative.

5. When contractual and/or technical holding times are exceeded, this should be noted as an action item for the TPO.

6. The reviewer should also be aware of the scenario in which the laboratory has exceeded the technical holding times, but met contractual holding times. In this case, the data reviewer should notify the Regional TPO (where samples were collected) and/or RSCC that shipment delays have occurred so that the field problem can be corrected. The reviewer may pass this information on to the laboratory's TPO, but should explain that contractually the laboratory met the requirements.

II. GC/MS Instrument Performance Check

A. **Review Items:** Form V SV *[Form V LCSV]*, and DFTPP mass spectra and mass listing.

B. **Objective**

Gas chromatograph/mass spectrometer (GC/MS) instrument performance checks (formerly referred to as tuning) are performed to ensure mass resolution, identification and, to some degree, sensitivity. These criteria are not sample specific. Conformance is determined using standard materials, therefore, these criteria should be met in all circumstances.

C. **Criteria**

The analysis of the instrument performance check solution must be performed at the beginning of each 12-hour period during which samples or standards are analyzed. The instrument performance check, decafluorotriphenylphosphine (DFTPP) for volatile analysis, must meet the ion abundance criteria given below.

Decafluorotriphenylphosphine (DFTPP)

m/z	ION ABUNDANCE CRITERIA
51	30.0 - 80.0% of m/z 198
68	Less than 2.0% of m/z 69
69	Present
70	Less than 2.0% of m/z 69
127	25.0 - 75.0% of m/z 198
197	Less than 1.0% of m/z 198
198	Base peak, 100% relative abundance
199	5.0 - 9.0% of m/z 198
275	10.0 - 30.0% of m/z 198
365	Greater than 0.75% of m/z 198
441	Present, but less than m/z 443
442	40.0 - 110.0% of m/z 198
443	15.0 - 24.0% of m/z 442

NOTE: All ion abundances must be normalized to m/z 198, the nominal base peak, even though the ion abundances of m/z 442 may be up to 110 percent that of m/z 198.

D. **Evaluation**

1. Compare the data presented on each GC/MS Instrument Performance Check (Form V SV *[Form V LCSV]*) with each mass listing submitted and ensure the following:

    a. Form V SV *[Form V LCSV]* is present and completed for each 12-hour period during which samples were analyzed.

b. The laboratory has not made any transcription errors between the data and the form. If there are major differences between the mass listing and the Form Vs, a more in-depth review of the data is required. This may include obtaining and reviewing additional information from the laboratory.

c. The appropriate number of significant figures has been reported (number of significant figures given for each ion in the ion abundance criteria column) and that rounding is correct.

d. The laboratory has not made any calculation errors.

2. Verify from the raw data (mass spectral listing) that the mass assignment is correct and that the mass is normalized to m/z 198.

3. Verify that the ion abundance criteria was met. The criteria for m/z 68, 70, 441, and 443 are calculated by normalizing to the specified m/z.

4. If possible, verify that spectra were generated using appropriate background subtraction techniques. Since the DFTPP spectrum is obtained from chromatographic peaks that should be free from coelution problems, background subtraction should be done in accordance with the following procedure. Three scans (the peak apex scan and the scans immediately preceding and following the apex) are acquired and averaged and background subtraction must be accomplished using a single scan prior to the elution of DFTPP.

NOTE: All instrument conditions must be identical to those used in the sample analysis. Background subtraction actions resulting in spectral distortions for the sole purpose of meeting the contract specifications are contrary to the quality assurance objectives and are therefore unacceptable.

E. Action

1. If the laboratory has made minor transcription errors which do not significantly affect the data, the data reviewer should make the necessary corrections on a copy of the form.

2. If the laboratory has failed to provide the correct forms or has made significant transcription or calculation errors, the Region's designated representative should contact the laboratory and request corrected data. If the information is not available, then the reviewer must use professional judgement to assess the data. The laboratory's TPO should be notified.

3. If mass assignment is in error (such as m/z 199 is indicated as the base peak rather than m/z 198), classify all associated data as unusable (R).

4. If ion abundance criteria are not met, professional judgement may be applied to determine to what extent the data may be utilized. Guidelines to aid in the application of professional judgement in evaluating ion abundance criteria are discussed as follows:

a. Some of the most critical factors in the DFTPP criteria are the non-instrument specific requirements that are also not unduly affected by the location of the spectrum on the chromatographic profile. The m/z ratios for 198/199 and 442/443 are critical. These ratios are based on the natural abundances of carbon 12 and carbon 13 and should always be met. Similarly, the relative abundances for m/z 68, 70, 197, and 441 indicate the condition of the instrument and the suitability of the resolution adjustment and are very important. Note that all of the foregoing abundances relate to adjacent ions; they are relatively insensitive to differences in instrument design and position of the spectrum on the chromatographic profile.

b. For the ions at m/z 51, 127, and 275, the actual relative abundance is not as critical. For instance, if m/z 275 has 40% relative abundance (criteria: 10.0-30.0%) and other criteria are met, then the deficiency is minor.

c. The relative abundance of m/z 365 is an indicator of suitable instrument zero adjustment. If relative abundance for m/z 365 is zero, minimum detection limits may be affected. On the other hand, if m/z 365 is present, but less than the 0.75% minimum abundance criteria, the deficiency is not as serious.

5. Decisions to use analytical data associated with DFTPP instrument performance checks not meeting contract requirements should be clearly noted in the data review narrative.

6. If the reviewer has reason to believe that instrument performance check criteria were achieved using techniques other than those specified in the SOW and II.D.4 above, additional information on the DFTPP instrument performance checks should be obtained. If the techniques employed are found to be at variance with contract requirements, the procedures of the laboratory may merit evaluation. Concerns or questions regarding laboratory performance should be noted for TPO action. For example, if the reviewer has reason to believe that an inappropriate technique was used to obtain background subtraction (such as background subtracting from the solvent front or from another region of the chromatogram rather than the DFTPP peak), then this should be noted for TPO action.

III. Initial Calibration

A. Review Items: Form VI SV-1 and SV-2 *[Form VI LCSV-1 and LCSV-2]*, quantitation reports, and chromatograms.

B. Objective

Compliance requirements for satisfactory instrument calibration are established to ensure that the instrument is capable of producing acceptable qualitative and quantitative data for compounds on the semivolatile Target Compound List (TCL). Initial calibration demonstrates that the instrument is capable of acceptable performance in the beginning of the analytical run and of producing a linear calibration curve.

C. Criteria

1. Initial calibration standards containing both semivolatile target compounds and surrogates are analyzed at concentrations of 20, 50, 80, 120, and 160 ug/L at the beginning of each analytical sequence or as necessary if the continuing calibration acceptance criteria are not met. The initial calibration (and any associated samples and blanks) must be analyzed within 12 hours of the associated instrument performance check.

   [For data generated through the Low Concentration SOW: Initial calibration standards containing both semivolatile TCL compounds and surrogates are analyzed at concentrations of 5, 10, 20, 50, and 80 ug/L at the beginning of each analytical sequence or as necessary if the continuing calibration acceptance criteria are not met. The initial calibration (and any associated samples and blanks) must be analyzed within 12 hours of the associated DFTPP tuning check. The following nine compounds require initial calibration at 20, 50, 80, 100, and 120 ug/L: 2,4-dinitrophenol, 2,4,5-trichlorophenol, 2-nitroaniline, 3-nitroaniline, 4-nitroaniline, 4-nitrophenol, 4,6-dinitro-2-methylphenol, pentachlorophenol, and 2,4,6-tribromophenol (surrogate).]

2. Minimum Relative Response Factor (RRF) criteria must be greater than or equal to 0.05. Contractual RRF criteria are listed in Appendix A *[Appendix B]*.

3. The Percent Relative Standard Deviations (%RSD) for the RRFs in the initial calibration must be less than or equal to 30%.

D. Evaluation

1. Verify that the correct concentration of standards were used for the initial calibration (i.e., 20, 50, 80, 120, and 160 ug/L). For the eight compounds with higher CRQLs, only a four-point initial calibration is required (i.e., 50, 80, 120, and 160 ug/L).

   [Verify that the correct concentration of standards were used for the initial calibration (i.e., 5, 10, 20, 50, and 80 ug/L). For the nine compounds listed in III.C.1. with higher CRQLs, verify that a five point initial calibration at 20, 50, 80, 100, and 120 ug/L was performed.]

iii. No qualifiers are needed for non-detected target compounds.

c. If the low end of the curve is outside of the linearity criteria:

i. No qualifiers are required for positive results in the linear portion of the curve.

ii. Qualify low level positive results in the area of non-linearity with "J".

iii. Qualify non-detected semivolatile target compounds using professional judgement.

3. If the laboratory has failed to provide adequate calibration information, the designated representative should contact the laboratory and request the necessary information. If the information is not available, the reviewer must use professional judgement to assess the data.

4. Whenever possible, the potential effects on the data due to calibration criteria exceedance should be noted in the data review narrative.

5. If calibration criteria are grossly exceeded, this should be noted for TPO action.

IV. Continuing Calibration

A. **Review Items:** Form VII SV-1 and SV-2 *[Form VII LCSV-1 and LCSV-2]*, quantitation reports, and chromatograms.

B. **Objective**

Compliance requirements for satisfactory instrument calibration are established to ensure that the instrument is capable of producing acceptable qualitative and quantitative data for semivolatile target compounds. Continuing calibration establishes the 12-hour relative response factors on which the quantitations are based and checks satisfactory performance of the instrument on a day-to-day basis.

C. **Criteria**

1. Continuing calibration standards containing both target compounds and surrogates are analyzed at the beginning of each 12-hour analysis period following the analysis of the instrument performance check and prior to the analysis of blanks and samples.

2. The minimum Relative Response Factors (RRF) for semivolatile target compounds and surrogates must be greater than or equal to 0.05.

3. The percent difference (%D) between the initial calibration $\overline{RRF}$ and the continuing calibration RRF must be within ± 25.0% for all target compounds.

D. **Evaluation**

1. Verify that the continuing calibration was run at the required frequency and that the continuing calibration was compared to the correct initial calibration.

2. Evaluate the continuing calibration RRF for all semivolatile target compounds and surrogates.

   a. Check and recalculate the continuing calibration RRF for at least one semivolatile target compound for each internal standard; verify that the recalculated value(s) agrees with the laboratory reported value(s).

   b. Verify that all semivolatile target compounds and surrogates have RRFs within specifications.

NOTE: Because historical performance data indicate poor response and/or erratic behavior, the compounds in Table 4 (Section III.D.3) have no contractual maximum %D criteria. Contractually they must meet a minimum RRF criterion of 0.01, however, **for data review purposes, the "greater than or equal to 0.05" criterion is applied to all semivolatile compounds.**

3. Evaluate the %D between initial calibration $\overline{RRF}$ and continuing calibration RRF for one or more semivolatile compounds.

   a. Check and recalculate the %D for at least one semivolatile target compound for each internal standard; verify that the recalculated value agrees with the laboratory reported value(s).

   b. Verify that the %D is within the ± 25.0% criterion, for all semivolatile target compounds and surrogates. Note those compounds which have a %D outside the ± 25.0% criterion. The contractual criteria for an acceptable continuing calibration specifies that up to any 4 semivolatile target compounds may fail to meet minimum RRF or maximum %D as long as they have RRFs that are greater than or equal to 0.010, and %D of less than or equal to 40.0%. For data review purposes, however, all compounds must be considered for qualification when the %D exceeds the ± 25.0% criterion.

4. If errors are detected in the calculations of either the continuing calibration RRF or the %D, perform a more comprehensive recalculation.

E. Action

1. The reviewer should use professional judgement to determine if it is necessary to qualify the data for any semivolatile target compound. If qualification of data is required, it should be performed using the following guidelines:

   a. If the %D is outside the ± 25.0% criterion and the continuing calibration RRF is greater than or equal to 0.05, qualify positive results "J".

   b. If the %D is outside the ± 25.0% criterion and the continuing calibration RRF is greater than or equal to 0.05, qualify non-detected semivolatile target compounds "UJ".

   c. If the continuing calibration RRF is less than 0.05, qualify positive results that have acceptable mass spectral identification with "J" or use professional judgement.

   d. If the continuing calibration RRF is less than 0.05, qualify non-detected semivolatile target compounds as unusable (R).

2. If the laboratory has failed to provide adequate calibration information, the designated representative should contact the laboratory and request the necessary information. If the information is not available, the reviewer must use professional judgement to assess the data.

3. Whenever possible, the potential effects on the data due to calibration criteria exceedance should be noted in the data review narrative.

4. If calibration criteria are grossly exceeded, this should be noted for TPO action.

V. Blanks

A. **Review Items:** Form I SV-1 and SV-2 *[Form I LCSV-1 and LCSV-2]*. Form IV SV *[Form IV LCSV]*, chromatograms, and quantitation reports.

B. **Objective**

The purpose of laboratory (or field) blank analyses is to determine the existence and magnitude of contamination problems resulting from laboratory (or field) activities. The criteria for evaluation of blanks apply to any blank associated with the samples (e.g., method blanks, instrument blanks, trip blanks, and equipment blanks). If problems with <u>any</u> blank exist, all associated data must be carefully evaluated to determine whether or not there is an inherent variability in the data, or if the problem is an isolated occurrence not affecting other data.

C. **Criteria**

1. No contaminants should be found in the blanks.

2. The method blank must be analyzed on each GC/MS system used to analyze that specific group or set of samples.

D. **Evaluation**

1. Review the results of all associated blank, Form I SV-1 and SV-2, and raw data (chromatograms and quantitation reports) to evaluate the presence of target and non-target compounds in the blanks.

2. Verify that a method blank analysis has been reported per matrix, per concentration level, for each extraction batch and for each GC/MS system used to analyze semivolatile samples. The reviewer can use the Method Blank Summary (Form IV SV) to assist in identifying samples associated with each method blank.

E. **Action**

If the appropriate blanks were not analyzed with the frequency described above, then the data reviewer should use professional judgement to determine [illegible]e associated sample data should be qualified. The reviewer may need to obtain additional information from the laboratory. The situation should be noted for TPO action.

Action in the case of unsuitable blank results depends on the circumstances and origin of the blank. Positive sample results should be reported unless the concentration of the compound in the sample is less than or equal to 10 times (10x) the amount in any blank for the common phthalate contaminants, or 5 times the amount for other compounds. In instances where more than one blank is associated with a given sample, qualification should be based upon a comparison with the associated blank having the highest concentration of a contaminant. The results must <u>not</u> be corrected by subtracting any blank value.

Specific actions are as follows:

1. If a semivolatile compound is found in a blank but not found in the sample, no action is taken. If the contaminants found are volatile target compounds (or interfering non-target compounds) at significant concentrations above the CRQL, then this should be noted for TPO action.

2. Any semivolatile compound detected in the sample (other than the common phthalate contaminants), that was also detected in any associated blank, is qualified if the sample concentration is less than five times (5x) the blank concentration. The quantitation limit may also be elevated. Typically, the sample CRQL is elevated to the concentration found in the sample. The reviewer should use professional judgement to determine if further elevation of the CRQL is required. For phthalate contaminants, the results are qualified "U" by elevating the sample quantitation limit to the sample concentration when the sample result is less than 10x the blank concentration.

   The reviewer should note that blanks may not involve the same weights, volumes, or dilution factors as the associated samples. These factors must be taken into consideration when applying the "5x" and "10x" criteria, such that a comparison of the total amount of contamination is actually made.

   Additionally, there may be instances where little or no contamination was present in the associated blanks, but qualification of the sample was deemed necessary. Contamination introduced through dilution is one example. Although it is not always possible to determine, instances of this occurring can be detected when contaminants are found in the diluted sample result, but are absent in the undiluted sample result. Since both results are not routinely reported, it may be impossible to verify this source of contamination. However, if the reviewer determines that the contamination is from a source other than the sample, he/she should qualify the data. In this case, the "5x" or "10x" rules may not apply; the sample value should be reported as a non-detect. An explanation of the rationale used for this determination should be provided in the narrative accompanying the Regional Data Assessment Summary.

3. If gross contamination exists (i.e., saturated peaks by GC/MS), all affected compounds in the associated samples should be qualified as unusable (R), due to interference. This should be noted for TPO action if the contamination is suspected of having an effect on the sample results.

4. If inordinate amounts of other target compounds are found at low levels in the blank(s), it may be indicative of a problem and should be noted for TPO action.

5. The same consideration given to the target compounds should also be given to Tentatively Identified Compounds (TICs) which are found in both the sample and associated blank(s). (See SV Section XII for TIC guidance.)

6. If an instrument blank was not analyzed following a sample analysis which contained an analyte(s) at high concentration(s), sample analysis results after the high concentration sample must be evaluated for carryover. Professional judgement should be used to determine if instrument cross-contamination has affected any positive compound

identification(s). If instrument cross-contamination is suggested, then this should be noted for TPO action if the cross-contamination is suspected of having an effect on the sample results.

The following are examples of applying the blank qualification guidelines. Certain circumstances may warrant deviations from these guidelines.

Example 1: Sample result is greater than the Contract Required Quantitation Limit (CRQL), but is less than the 5x or 10x multiple of the blank result.

	Rule	
	10x	5x
Blank Result	7	7
CRQL	5	5
Sample Result	60	30
Qualified Sample Result	60U	30U

In the example for the "10x" rule, sample results less than 70 (or 10 x 7) would be qualified as non-detects. In the case of the "5x" rule, sample results less than 35 (or 5 x 7) would be qualified as non-detects.

Example 2: Sample result is less than CRQL, and is also less than the 5x or 10x multiple of the blank result.

	Rule	
	10x	5x
Blank Result	6	6
CRQL	5	5
Sample Result	4J	4J
Qualified Sample Result	5U	5U

Note that data are not reported as 4U, as this would be reported as a detection limit below the CRQL.

Example 3: Sample result is greater than the 5x or 10x multiple of the blank result.

	Rule	
	10x	5x
Blank Result	10	10
CRQL	5	5
Sample Result	120	60
Qualified Sample Result	120	60

For both the "10x" and "5x" rules, sample results exceeded the adjusted blank results of 100 (or 10x10) and 50 (or 5x10), respectively.

VI. Surrogate Spikes

A. **Review Items:** Form II SV-1 and SV-2 *[Form II LCSV]*, chromatograms, and quantitation reports

B. Objective

Laboratory performance on individual samples is established by means of spiking activities. All samples are spiked with surrogate compounds prior to sample preparation. The evaluation of the results of these surrogate spikes is not necessarily straightforward. The sample itself may produce effects due to such factors as interferences and high concentrations of analytes. Since the effects of the sample matrix are frequently outside the control of the laboratory and may present relatively unique problems, the evaluation and review of data based on specific sample results is frequently subjective and demands analytical experience and professional judgment. Accordingly, this section consists primarily of guidelines, in some cases with several optional approaches suggested.

C. **Criteria**

1. Surrogate spikes, 4 acid compounds (3 required and 1 advisory) and 4 base/neutral compounds (3 required and 1 advisory) are added to all samples and blanks to measure their recovery in sample and blank matrices.

   [For data generated through the Low Concentration SOW: Surrogate spikes, 3 acid compounds and 3 base/neutral compounds, are added to all samples and blanks to measure their recovery in sample and blank matrices.]

2. Surrogate spike recoveries for semivolatile samples and blanks must be within the limits specified on in Appendix A and on Form II SV-1 and SV-2.

   [For data generated through the Low Concentration SOW: Surrogate spike recoveries for semivolatile samples and blanks must be within the limits specified in Appendix B and on Form II LCSV.]

D. **Evaluation**

1. Check raw data (e.g., chromatograms and quantitation reports) to verify the surrogate spike recoveries on the Surrogate Recovery Form II SV-1 and SV-2 *[Form II LCSV]*. Check for any transcription or calculation errors.

2. Check that the surrogate spike recoveries were calculated correctly. The equation can be found in Appendix A *[Appendix B]*.

3. The following should be determined from the Surrogate Recovery form(s):

   a. If any two base/neutral <u>or</u> acid surrogates are out of specification, or if any one base/neutral or acid extractable surrogate has a recovery of less than 10%, then there should be a reanalysis to confirm that the non-compliance is due to sample matrix effects rather than laboratory deficiencies.

NOTE: When there are unacceptable surrogate recoveries followed by successful re-analyses, the laboratories are required to report only the successful run.

b. The laboratory has failed to perform satisfactorily if surrogate recoveries are out of specification and there is no evidence of reinjection of the extract, or reextraction and reanalysis (if reinjection fails to resolve the problem).

c. Verify that no blanks have surrogates recoveries outside the criteria.

4. Any time there are two or more analyses for a particular fraction the reviewer must determine which are the best data to report. Considerations should include but are not limited to:

   a. Surrogate recovery (marginal versus gross deviation).

   b. Technical holding times.

   c. Comparison of the values of the target compounds reported in each fraction.

   d. Other QC information, such as performance of internal standards.

E. Action

Data are not qualified with respect to surrogate recovery unless two or more semivolatile surrogates, within the same fraction (base/neutral or acid fraction), are out of specification. For surrogate spike recoveries out of specification, the following approaches are suggested based on a review of all data from the case, especially considering the apparent complexity of the sample matrix.

1. If two or more surrogates in either semivolatile fraction (base/neutral or acid fraction) have a recovery greater than the upper acceptance limit (UL):

   a. Specify the fraction that is being qualified, i.e. acid, base/neutral, or both.

   b. Detected semivolatile target compounds are qualified "J."

   c. Results for non-detected semivolatile target compounds should not be qualified.

2. If two or more surrogates in either semivolatile fraction have a recovery greater than or equal to 10% but less than the lower acceptance limit (LL):

   a. Specify the fraction that is being qualified, i.e. acid, base/neutral, or both.

   b. Detected semivolatile target compounds are qualified "J."

   c. For non-detected semivolatile target compounds, the sample quantitation limit is qualified as approximated (UJ).

3. If any surrogate in either semivolatile fraction show less than 10% recovery:

   a. Specify the fraction that is being qualified, i.e. acid, base/neutral, or both.

   b. Detected semivolatile target compounds are qualified "J".

   c. Non-detected semivolatile target compounds may be qualified as unusable (R).

Table 5. Qualification of Semivolatile Analytes Based on Surrogate Recoveries

	Surrogate Recovery		
	> UL	10% to LL	< 10%
Detected analytes	J	J	J
Non-detected analytes	No Qualification	UJ	R

4. In the special case of a blank analysis with surrogates out of specification, the reviewer must give special consideration to the validity of associated sample data. The basic concern is whether the blank problems represent an isolated problem with the blank alone, or whether there is a fundamental problem with the analytical process. For example, if one or more samples in the batch show acceptable surrogate recoveries, the reviewer may choose to consider the blank problem to be an isolated occurrence. However, even if this judgement allows some use of the affected data, analytical problems should be noted for TPO action. Also note if there are potential contractual problems associated with the lack of re-analysis of samples that were out of specification.

5. Whenever possible, the potential effects of the data resulting from system monitoring recoveries not meeting the advisory limits should be noted in the data review narrative.

VII. Matrix Spikes/Matrix Spike Duplicates
(Not Required for Low Concentration Water Data)

A. **Review Items:** Form III SV-1 and SV-2, chromatograms, and quantitation reports.

B. Objective

Data for matrix spikes/matrix spike duplicates (MS/MSD) are generated to determine long-term precision and accuracy of the analytical method on various matrices and to demonstrate acceptable compound recovery by the laboratory at the time of sample analysis. These data alone cannot be used to evaluate the precision and accuracy of individual samples. However, when exercising professional judgement, this data should be used in conjunction with other available QC information.

C. **Criteria**

1. Matrix spikes and matrix spike duplicate samples are analyzed at frequency of one MS and MSD per 20 samples of similar matrix.
2. Matrix spike and matrix spike duplicate recoveries should be within the advisory limits established on Form III SV-1 and SV-2.
3. The Relative Percent Differences (RPDs) between matrix spike and matrix spike duplicate recoveries should be within the advisory limits listed on Form III SV-1 and SV-2.

D. **Evaluation**

1. Verify that MS and MSD samples were analyzed at the required frequency and that results are provided for each sample matrix.
2. Inspect results for the MS/MSD Recovery on Form III SV-1 and SV-2 and verify that the results for recovery and RPD are within the advisory limits.
3. Verify transcriptions from raw data and verify calculations.
4. Check that the recoveries and RPD were calculated correctly.
5. Compare results (%RSD) of non-spiked compounds between the original result, MS, and MSD.

E. **Action**

1. No action is taken on MS/MSD data alone. However, using informed professional judgment the data reviewer may use the matrix spike and matrix spike duplicate results in conjunction with other QC criteria and determine the need for some qualification of the data.
2. The data reviewer should first try to determine to what extent the results of the MS/MSD effect the associated data. This determination should be made with regard to the

MS/MSD sample itself as well as specific analytes for all samples associated with the MS/MSD.

3. In those instances where it can be determined that the results of the MS/MSD effect only the sample spiked, then qualification should be limited to this sample alone. However, it may be determined through the MS/MSD results that a laboratory is having a systematic problem in the analysis of one or more analytes, which affects all associated samples.

4. The reviewer must use professional judgement to determine the need for qualification of positive results of non-spiked compounds.

NOTE: If a field blank was used for the MS/MSD, a statement to that effect must be included for TPO action.

VIII. Laboratory Control Samples
(Low Concentration Water)

A. *Review Items: Form III LCSV, LCS chromatograms and quantitation reports.*

B. *Objective*

Data for laboratory control samples (LCS) are generated to provide information on the accuracy of the analytical method and the laboratory performance.

C. *Criteria*

1. *Laboratory control samples are analyzed at frequency of once per 20 samples per SDG. The LCS must be prepared and analyzed concurrently with the samples in the SDG.*

2. *LCS percent recoveries must be within the QC limits provided on Form III LCSV. The LCS must meet the recovery criteria for the sample data to be accepted.*

3. *The LCS contains the following semivolatile target compounds, in addition to the required surrogates:*

Phenol
2-Chlorophenol
4-Chloroaniline
2,4,6-Trichlorophenol
bis(2-Chloroethyl)ether
N-Nitroso-di-n-propylamine
Hexachloroethane
Isophorone
1,2,4-Trichlorobenzene
Naphthalene
2,4-Dinitrotoluene
Diethylphthalate
N-Nitrosodiphenylamine
Hexachlorobenzene
Benzo(a)pyrene

4. *The criteria for surrogate recovery and internal standard performance also apply.*

D. *Evaluation*

1. *Verify that LCS samples were analyzed at the required frequency.*

2. *Inspect the results for LCS Recovery on Form III LCSV and verify that the results for recovery are within the advisory limits.*

3. *Verify transcriptions from raw data and verify calculations.*

4. *Check that the recoveries were calculated correctly.*

E. *Action*

If the LCS criteria are not met, then the laboratory performance and method accuracy are in question. Professional judgement should be used to determine if the data should be qualified or rejected. The following guidance is suggested for qualifying sample data for which the associated LCS does not meet the required criteria.

1. *Action on the LCS recovery should be based on both the number of compounds that are outside of the recovery criteria and the magnitude of the exceedance of the criteria.*

2. *If the LCS recovery criteria are not met, then the LCS results should be used to qualify sample data for the specific compounds that are included in the LCS solution. Professional judgement should be used to*

qualify data for compounds other than those compounds that are included in the LCS. Professional judgement to qualify non-LCS compounds should take into account the compound class, compound recovery efficiency, analytical problems associated with each compound, and comparability in performance of the LCS compound to the non-LCS compound.

3. *If the LCS recovery is greater than 140%, then positive sample results for the affected compound(s) should be qualified with a "J".*

4. *If the mass spectral criteria are met but the LCS recovery is less than 60%, then the associated detected target compounds should be qualified "J" and the associated non-detected target compounds should be qualified "R".*

5. *If more than half of the compounds in the LCS are not within the required recovery criteria, then all of the associated detected target compounds should be qualified "J" and all associated non-detected target compounds should be qualified "R."*

6. *Action on non-compliant surrogate recovery and internal standard performance should follow the procedures provided in VI.E and X.E, respectively. Professional judgement should be used to evaluate the impact that non-compliance for surrogate recovery and internal standard performance in the LCS has on the associated sample data.*

7. *It should be noted for TPO action if a laboratory fails to analyze an LCS with each SDG, or if a laboratory consistently fails to generate acceptable LCS recoveries.]*

IX. Regional Quality Assurance and Quality Control

A. **Review Items:** Form I SV-1 and SV-2 *[Form I LCSV-1 and LCSV-2]*, chromatograms, quantitation report, traffic report and raw data for Regional QC samples.

B. **Objective**

Regional Quality Assurance and Quality Control (QA/QC) refer to any QA and/or QC initiated by the Region, including field duplicates, Regional Performance Evaluation (PE) samples, blind spikes, and blind blanks. It is highly recommended that Regions adopt the use of these.

C. **Criteria**

Criteria are determined by each Region.

1. Performance evaluation sample frequency may vary.

   [For data generated through the Low Concentration SOW: A performance evaluation (PE) sample may be required as frequently as once per SDG.]

2. The analytes present in the PE sample must be correctly identified and quantified.

D. **Evaluation**

Evaluation procedures must follow the Region's SOP for data review. Each Region will handle the evaluation of PE samples on an individual basis. Results for PE samples should be compared to the acceptance criteria for the specific PE samples, if available.

E. **Action**

Any action must be in accordance with Regional specifications and the criteria for acceptable PE sample results. Unacceptable results for PE samples should be noted for TPO action.

X. Internal Standards

A. **Review Items:** Form VIII SV-1 and SV-2 *[Form VIII LCSV-1 and LCSV-2]*, quantitation reports, and chromatograms.

B. **Objective**

Internal Standards (IS) performance criteria ensure that GC/MS sensitivity and response are stable during every analytical run.

C. **Criteria**

1. Internal standard area counts for samples and blanks must not vary by more than a factor of two (- 50% to + 100%) from the associated calibration standard.

   [For data generated through the Low Concentration Water SOW: Internal standard area counts must not vary by more than a factor of ± 40.0% from the associated calibration standard.]

2. The retention time of the internal standards in samples and blanks must not vary by more than ± 30 seconds from the retention time of the associated calibration standard.

   [For data generated through the Low Concentration SOW: The retention time of the internal standards in samples and blanks must not vary by more than ±20.0 seconds from the retention time of the associated calibration standard.]

D. **Evaluation**

1. Check raw data (e.g., chromatograms and quantitation lists) for samples and blanks to verify the internal standard retention times and areas reported on the Internal Standard Area Summary (Forms VIII SV-1, VIII SV-2 *[Form VIII LCSV-1 and LCSV-2]*).

2. Verify that all retention times and IS areas are within the required criteria.

3. If there are two analyses for a particular fraction, the reviewer must determine which are the best data to report. Considerations should include:

   a. **Magnitude and direction of the IS area [illegible]**

   b. **Magnitude and direction of the IS retention time shift.**

   c. **Technical holding times.**

   d. **Comparison of the values of the target compounds reported in each fraction.**

E. **Action**

1. **If an IS area count for a sample or blank is outside - 50% or + 100% of the area for the associated standard:**

a. Positive results for compounds quantitated using that IS should be qualified with "J".

b. Non-detected compounds quantitated using an IS area count greater than 100% should not be qualified.

c. Non-detected compounds quantitated using an IS area count less than 50% are reported as the associated sample quantitation limit and qualified with "UJ".

d. If extremely low area counts are reported, or if performance exhibits a major abrupt drop-off, then a severe loss of sensitivity is indicated. Non-detected target compounds should then be qualified as unusable (R).

[If an IS area count for a sample or blank is outside ± 40.0% of the area for associated standard:

a. Positive results for compounds quantitated using that IS should be qualified with "J".

b. Non-detected compounds quantitated using an IS area count greater than 40% should not be qualified.

c. Non-detected compounds quantitated using an IS area count less than 40% are reported as the associated sample quantitation limit and qualified with "UJ".

d. If extremely low area counts are reported, or if performance exhibits a major abrupt drop-off, then a severe loss of sensitivity is indicated. Non-detected target compounds should then be qualified as unusable (R).]

2. If an IS retention time varies by more than 30 seconds:

[If an IS retention time varies by more than 20.0 seconds:]

The chromatographic profile for that sample must be examined to determine if any false positives or negatives exist. For shifts of a large magnitude, the reviewer may consider partial or total rejection (R) of the data for that sample fraction. Positive results should not need to be qualified with "R" if the mass spectral criteria are met.

3. If the internal standards performance criteria are grossly exceeded, then this should be noted for TPO action. Potential effects on the data resulting from unacceptable internal standard performance should be noted in the data review narrative.

XI. Target Compound Identification

A. **Review Items:** Form I SV-1 and SV-2 *[Form I LCSV-1 and LCSV-2]*, quantitation reports, mass spectra, and chromatograms.

B. **Objective**

Qualitative criteria for compound identification have been established to minimize the number of erroneous identifications of compounds. An erroneous identification can either be a false positive (reporting a compound present when it is not) or a false negative (not reporting a compound that is present).

The identification criteria can be applied much more easily in detecting false positives than false negatives. More information is available due to the requirement for submittal of data supporting positive identifications. Negatives, or non-detected compounds, on the other hand represent an absence of data and are, therefore, much more difficult to assess. One example of detecting false negatives is the reporting of a Target Compound as a TIC.

C. **Criteria**

1. Compound must be within ± 0.06 relative retention time (RRT) units of the standard RRT.

2. Mass spectra of the sample compound and a current laboratory-generated standard must match according to the following criteria:

   a. All ions present in the standard mass spectrum at a relative intensity greater than 10% must be present in the sample spectrum

   [For data generated through the Low Concentration SOW: All ions present in the standard mass spectrum at a relative intensity greater than 25% must be present in the sample spectrum.]

   b. The relative intensities of these ions must agree within ±20% between the standard and sample spectra. (Example: For an ion with an abundance of 50% in the standard spectrum, the corresponding sample ion abundance must be between 30% and 70%.)

   c. Ions present at greater than 10% in the sample mass spectrum but not present in the standard spectrum must be considered and accounted for.

   [For data generated through the Low Concentration SOW: Ions present at greater than 25% in the sample mass spectrum but not present in the standard mass spectrum must be considered and accounted for.]

D. **Evaluation**

1. Check that the RRT of reported compounds is within ± 0.06 RRT units of the standard relative retention time.

2. Check that the sample compound spectra against the laboratory standard spectra to verify that its meets the specified criteria.

3. The reviewer should be aware of situations (e.g., high concentration samples preceding low concentration samples) when sample carryover is a possibility and should use judgment to determine if instrument cross-contamination has affected any positive compound identification.

4. Check the chromatogram to verify that peaks are accounted for, i.e., major peaks are either identified as target compounds, TICs, surrogates, or internal standards.

E. Action

1. The application of qualitative criteria for GC/MS analysis of target compounds requires professional judgement. It is up to the reviewer's discretion to obtain additional information from the laboratory. If it is determined that incorrect identifications were made, all such data should be qualified as not detected (U) or unusable (R).

2. Professional judgement must be used to qualify the data if it is determined that cross-contamination has occurred.

3. Any changes made to the reported compounds or concerns regarding target compound identifications should be clearly indicated in the data review narrative. The necessity for numerous or significant changes should be noted for TPO action.

XII. Compound Quantitation and Reported CRQLS

A. **Review Items:** Form I SV-1 and SV-2 *[Form I LCSV-1 and LCSV-2]*, sample preparation sheets, case narrative, sample clean-up sheets, quantitation reports, and chromatograms.

B. **Objective**

The objective is to ensure that the reported quantitation results and Contract Required Quantitation Limits (CRQLs) for semivolatile target compounds are accurate.

C. **Criteria**

1. Compound quantitation, as well as the adjustment of the CRQL, must be calculated according to the correct equation.

2. Compound area responses must be calculated based on the internal standard (IS) associated with that compound, as listed in Appendix A *[Appendix B]* (also as specified in the Statement of Work). Quantitation must be based on the quantitation ion (m/z) specified in the SOW for both the IS and target analytes. The compound quantitation must be based on the RRF from the appropriate daily calibration standard.

D. **Evaluation**

1. For all fractions, raw data should be examined to verify the correct calculation of all sample results reported by the laboratory. Quantitation lists, chromatograms, and sample preparation log sheets should be compared to the reported positive sample results and quantitation limits. Check the reported values.

2. Verify that the correct internal standard, quantitation ion, and RRF were used to quantitate the compound. Verify that the same internal standard, quantitation ion, and RRF are used consistently throughout the calibration and quantitation processes.

3. Verify that the CRQLs have been adjusted to reflect all sample dilutions, concentrations, splits, clean-up activities, and dry weight factors that are not accounted for by the method.

E. **Action**

1. If there are any discrepancies found, the laboratory may be contacted by the designated representative to obtain additional information that could resolve any differences. If a discrepancy remains unresolved, the reviewer must use professional judgement to decide which value is the best value. Under these circumstances, the reviewer may determine qualification of data is warranted. Decisions made on data quality should be included in the data review narrative. A description of the reasons for data qualification and the qualification that is applied to the data should be documented in the data review narrative.

2. Numerous or significant failures to accurately quantify the target compound or to properly evaluate and adjust CRQLs should be noted for TPO action.

XIII. Tentatively Identified Compounds

A. **Review Items:** Form I SV-TIC *[Form I LCSV-TIC]*, chromatograms, and library search printout with spectra for three TIC candidates.

B. **Objective**

Chromatographic peaks in semivolatile fraction analyses that are not target analytes, surrogates, or internal standards are potential tentatively identified compounds (TICs). TICs must be qualitatively identified by a National Institute of Standards and Technology (NIST) mass spectral library search and the identifications assessed by the data reviewer.

C. **Criteria**

For each sample, the laboratory must conduct a mass spectral search of the NIST library and report the possible identity for the 20 largest semivolatile fraction peaks which are not surrogate, internal standard, or target compounds, but which have area or height greater than 10 percent of the area or height of the nearest internal standard. TIC results are reported for each sample on the Organic Analyses Data Sheet (Form I SV-TIC).

[For data generated through the Low Concentration SOW: For each sample, the laboratory must conduct a mass spectral search of the NIST library and report the possible identity for the 20 largest semivolatile fraction peaks which are not surrogates, internal standards, or TCL compounds, but which have an area greater than 50 percent of the area of the nearest internal standard. Estimated concentrations for TICs are calculated similarly to the TCL compounds, using total ion areas for the TIC and the internal standard, and assuming a relative response factor of 1.0. TIC results are reported for each sample on the Organic Analyses Data Sheet (Form I LCSV-TIC).]

NOTE: Since the SOW revision of October 1986, the CLP does not allow the laboratory to report as tentatively identified compounds any target compound which is properly reported in another fraction. For example, late eluting volatile target compounds should not be reported as semivolatile TICs.

D. **Evaluation**

1. Guidelines for tentative identification are as follows:

   a. **Major ions (greater than 10% relative intensity) in the reference spectrum should be present in the sample spectrum.**

   [Major ions (greater than 25% relative intensity) in the reference spectrum should be present in the sample spectrum.]

   b. **The relative intensities of the major ions should agree within ±20% between the sample and the reference spectra.**

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

d. Ions present in the sample spectrum but not in the reference spectrum should be reviewed for possible background contamination, interference, or coelution of additional TIC or target compounds.

e. When the above criteria are not met, but in the technical judgment of the data reviewer or mass spectral interpretation specialist the identification is correct, the data reviewer may report the identification.

f. If in the data reviewer's judgment the identification is uncertain or there are extenuating factors affecting compound identifications, the TIC result may be reported as "unknown".

2. Check the raw data to verify that the laboratory has generated a library search for all required peaks in the chromatograms for samples and blanks.

[Check the raw data to verify that the laboratory has generated a library search for all required peaks in the chromatograms for samples and blanks with areas greater than or equal to 50 percent of the area of the nearest internal standard.]

3. Blank chromatograms should be examined to verify that TIC peaks present in samples are not found in blanks. When a low-level non-target compound that is a common artifact or laboratory contaminant is detected in a sample, a thorough check of blank chromatograms may require looking for peaks which are less than 10 percent of the internal standard height, but present in the blank chromatogram at a similar relative retention time.

[Blank chromatograms should be examined to verify that TIC peaks present in samples are not found in blanks. When a low-level non-TCL compound that is a common artifact or laboratory contaminant is detected in a sample, a thorough check of blank chromatograms may require looking for peaks which have areas less than 50 percent of the internal standard area, but present in the blank chromatogram at a similar relative retention time.]

4. All mass spectra for each sample and blank must be examined.

5. Since TIC library searches often yield several candidate compounds having a close matching score, all reasonable choices should be considered.

6. The reviewer should be aware of common laboratory artifacts/contaminants and their sources (e.g., aldol condensation products, solvent preservatives, and reagent contaminants). These may be present in blanks and not reported as sample TICs.

Examples:

a. Common laboratory contaminants: CO_2 (m/z 44), siloxanes (m/z 73), diethyl ether, hexane, certain freons (1,1,2-trichloro-1,2,2-trifluoroethane or fluorotrichloromethane), and phthalates at levels less than 100 ug/L or 4000 ug/Kg.

b. Solvent preservatives, such as cyclohexene which is a methylene chloride preservative. Related by-products include cyclohexanone, cyclohexenone, cyclohexanol, cyclohexenol, chlorocyclohexene, and chlorocyclohexanol.

c. Aldol reaction products of acetone include: 4-hydroxy-4-methyl-2-pentanone, 4-methyl-2-penten-2-one, and 5,5-dimethyl-2(5H)-furanone.

7. Occasionally, a target compound may be identified as a TIC in the proper analytical fraction by non-target library search procedures, even though it was not found on the quantitation list. If the total area quantitation method was used, the reviewer should request that the laboratory recalculate the result using the proper quantitation ion. In addition, the reviewer should evaluate other sample chromatograms and check library reference retention times on quantitation lists to determine whether the false negative result is an isolated occurrence or whether additional data may be affected.

8. Target compounds may be identified in more than one fraction. Verify that quantitation is made from the proper fraction.

9. Library searches should not be performed on internal standards or surrogates.

10. TIC concentration should be estimated assuming a RRF of 1.0.

E. Action

1. All TIC results should be qualified "NJ", tentatively identified, with approximated concentrations.

2. General actions related to the review of TIC results are as follows:

   a. If it is determined that a tentative identification of a non-target compound is not acceptable, the tentative identification should be changed to "unknown" or an appropriate identification.

   b. If all contractually required peaks were not library searched and quantitated, the designated representative could request these data from the laboratory.

3. TIC results which are not sufficiently above the level in the blank should not be reported. (Dilutions and sample size must be taken into account when comparing the amounts present in blanks and samples.)

4. When a compound is not found in any blanks, but is a suspected artifact of common laboratory contamination, the result may be qualified as unusable (R).

5. In deciding whether a library search result for a TIC represents a reasonable identification, professional judgment must be exercised. If there is more than one possible match, the result may be reported as "either compound X or compound Y." If there is a lack of isomer specificity, the TIC result may be changed to a non-specific isomer result (e.g., 1,3,5-trimethyl benzene to trimethyl benzene isomer) or to a compound class (e.g., 2-methyl, 3-ethyl benzene to substituted aromatic compound).

6. The reviewer may elect to report all similar isomers as a total. (All alkanes may be summarized and reported as total hydrocarbons.)

7. Other case factors may influence TIC judgments. If a sample TIC match is poor but other samples have a TIC with a good library match, similar relative retention time, and the same ions, identification information may be inferred from the other sample TIC results.

8. Physical constants, such as boiling point, may be factored into professional judgment of TIC results.

9. Any changes made to the reported data or any concerns regarding TIC identifications should be indicated in the data review narrative.

10. Failure to properly evaluate and report TICs should be noted for TPO action.

XIV. System Performance

A. **Review Items:** Form III SV-1 and SV-2 *[Form III LCSV]*, Form VIII SV-1 and SV-2 *[Form VIII LCSV-1 and LCSV-2]*, and chromatograms.

B. **Objective**

During the period following Instrument Performance QC checks (e.g. blanks, tuning, calibration), changes may occur in the system that degrade the quality of the data. While this degradation would not be directly shown by QC checks until the next required series of analytical QC runs, a through review of the ongoing data acquisition can yield indicators of instrument performance.

C. **Criteria**

There are no specific criteria for system performance. Professional judgement should be used to assess the system performance.

D. **Evaluation**

1. Abrupt, discrete shifts in the reconstructed ion chromatogram (RIC) baseline may indicate a change in the instrument's sensitivity or the zero setting. A baseline shift could indicate a decrease in sensitivity in the instrument or an increase in the instrument zero, possibly causing target compounds at or near the detection limit to be non-detects. A baseline "rise" could indicate problems such as a change in the instrument zero, a leak, or degradation of the column.

2. Poor chromatographic performance affects both qualitative and quantitative results. Indications of substandard performance include:

    a. High RIC background levels or shifts in absolute retention times of internal standards.

    b. Excessive baseline rise at elevated temperature.

    c. Extraneous peaks.

    d. Loss of resolution as suggested between by factors such as non-resolution of 2,4- and 2,5- dinitrotoluene.

    e. Peak tailing or peak splitting that may result in inaccurate quantitation.

[3. A drift in instrument sensitivity may occur during the 12-hour time period. This could be discerned by examination of the IS area on Form VIII LCSV-1 and LCSV-2 for trends such as a continuous or near-continuous increase or decrease in the IS area over time.

4. The results of the LCS analysis (Form III LCSV) may also be used to assess instrument performance.]

E. Action

Professional judgement must be used to qualify the data if it is determined that system performance has degraded during sample analyses. Any degradation of system performance which significantly affected the data should be documented for TPO action.

XV. Overall Assessment of Data

A. **Review Items:** Entire data package, data review results, and (if available) Quality Assurance Project Plan (QAPjP), and Sampling and Analysis Plan (SAP).

B. **Objective**

The overall assessment of a data package is a brief narrative in which the data reviewer expresses concerns and comments on the quality and, if possible, the useability of the data.

C. **Criteria**

Assess the overall quality of the data.

Review all available materials to assess the overall quality of the data, keeping in mind the additive nature of analytical problems.

D. **Evaluation**

1. Evaluate any technical problems which have not been previously addressed.

2. Review all available materials to assess the overall quality of the data, keeping in mind the additive nature of analytical problems.

3. If appropriate information is available, the reviewer may assess the useability of the data to assist the data user in avoiding inappropriate use of the data. Review all available information, including the QAPjP (specifically the Data Quality Objectives), SAP, and communication with data user that concerns the intended use and desired quality of the data.

E. **Action**

1. Use professional judgement to determine if there is any need to qualify data which were not qualified based on the QC criteria previously discussed.

2. Write a brief narrative to give the user an indication of the analytical limitations of the data. Any inconsistency of that data with the SDG Narrative should be noted for TPO action. If sufficient information on the intended use and required quality of the data are available, the reviewer should include his/her assessment of the useability of the data within the given context.

APPENDIX A

CONTRACTUAL REQUIREMENTS AND EQUATIONS

MULTI-MEDIA, MULTI-CONCENTRATION - MM/MC
(OLM01.0)

DRAFT 12/90
Revised 6/91

MULTI-MEDIA, MULTI-CONCENTRATION
CONTRACTUAL REQUIREMENTS AND EQUATIONS FOR VOLATILE DATA REVIEW

II. GC/MS Instrument Performance Check

Use equation II.1 to verify that the laboratory has not made errors the calculation of the percent relative abundance.

$$\% \ Relative \ Abundance = \frac{abundance \ of \ X}{abundance \ of \ Y} \times 100\% \qquad (II.1)$$

For example, the percent relative abundance of m/z 96 (X) relative to m/z 95 (Y) is calculated as follows:

$$\% \ Relative \ Abundance = \frac{abundance \ of \ m/z \ 96}{abundance \ of \ m/z \ 95} \times 100\%$$

III. Initial Calibration

Data Review Criteria: All volatile target compounds and system monitoring compounds must have a Relative Response Factor (RRF) of greater than or equal to 0.05 and a percent relative standard deviation (%RSD) of less than or equal to 30%.

Contractual Criteria: The maximum %RSD for volatile compounds is 20.5% and the minimum RRF criteria vary as specified in the Table A.1 (The volatile compounds listed separately in Table 2 on page 13 are not contractually required to meet a maximum %RSD but do have to meet a contractual minimum RRF of 0.010). The contractual criteria for an acceptable initial calibration specifies that up to any 2 volatile target compounds may fail to meet minimum RRF or maximum %RSD as long as they have RRFs that are greater than or equal to 0.010, and %RSD of less than or equal to 40.0%.

Table A.1 Minimum RRF Criteria for Volatile Target Compunds

Volatile Compound	**Minimum RRF**
Bromomethane	0.100
Vinyl chloride	0.100
1,1-Dichloroethene	0.100
1,1-Dichloroethane	0.200
Chloroform	0.200
1,2-Dichloroethane	0.100
1,1,1-Trichloroethane	0.100
Carbon tetrachloride	0.100
Bromodichloromethane	0.200
cis-1,3-Dichloropropene	0.200

Table A.1 Minimum RRF Criteria for Volatile Target Compunds (continued)

Volatile Compound	Minimum RRF
Trichloroethene	0.300
Dibromochloromethane	0.100
1,1,2-Trichloroethane	0.100
Benzene	0.500
trans-1,3-Dichloropropene	0.100
Bromoform	0.100
Tetrachloroethene	0.200
1,1,2,2-Tetrachloroethane	0.500
Toluene	0.400
Chlorobenzene	0.500
Ethylbenzene	0.100
Styrene	0.300
Xylenes (total)	0.300
Bromofluorobenzene	0.200

Initial calibration RRFs and $\overline{RRF}$ are calculated using equations III.1 and III.2.

$$RRF = \frac{A_x}{A_{is}} \times \frac{C_{is}}{C_x} \qquad \text{(III.1)}$$

$$\overline{RRF} = \frac{\sum_{i=1}^{5} RRF_i}{5} \qquad \text{(III.2)}$$

where:

RRF_i = "i"th Relative Response Factor
A = Area of the characteristic ion (EICP) measured
C = Concentration
is = Internal standard
x = Analyte of interest

The %RSD is calculated using equations III.3 and III.4.

$$\sigma = \sqrt{\sum_{i=1}^{n} \frac{(x_i - \bar{x})^2}{(n-1)}} \qquad \text{(III.3)}$$

$$\%RSD = \frac{\sigma}{\bar{x}} \times 100 \qquad \text{(III.4)}$$

where:

σ = Standard deviation of 5 relative response factors
$\bar{x}$ = Mean of 5 relative response factors

IV. Continuing Calibration

Data Review Criteria: All compounds must be considered for qualification when the %D exceeds the ± 25.0% criterion.

Contratual Criteria: The percent difference (%D) between the initial calibration $\overline{RRF}$ and the continuing calibration RRF is ± 25% for all compounds listed in Table A.1. The contractual criteria for an acceptable continuing calibration specifies that up to any 2 volatile target compounds may fail to meet minimum RRF or maximum %D as long as they have RRFs that are greater than or equal to 0.010, and %D of less than or equal to 40.0%.

Check the continuing calibration RRF calculations for volatile target compounds using equation III.1. The %D between initial calibration $\overline{RRF}$ and continuing calibration RRF is calculated using equation IV.1.

$$\% D = \frac{\overline{RRF}_I - RRF_C}{\overline{RRF}_I} \times 100\% \qquad \text{(IV.1)}$$

where:

$\overline{RRF}_I$ = average relative response factor from initial calibration.
RRF_C = relative response factor from continuing calibration standard.

VI. System Monitoring Compounds

The volatile system monitoring compounds (surrogates) and their contractual recovery limits are listed in Table A.2.

Table A.2 System Monitoring Compound Contractual Requirements

System Monitoring Compound	%Recovery Limits	
	Water Samples	Soil Samples
SMC1 Toluene-d_8	88 - 110	84 - 138
SMC2 Bromofluorobenzene	86 - 115	59 - 113
SMC3 1,2-Dichloroethane-d_4	76 - 114	70 - 121

Use equation VI.1 to check that the system monitoring compound recoveries were calculated correctly:

$$\% \ Recovery = \frac{Concentration/amount\ found}{Concentration/amount\ spiked} \times 100\% \qquad (VI.1)$$

VII. Matrix Spikes/Matrix Spike Duplicates

The matrix spike/matrix spike duplicate contractual requirements are listed in Table A.3.

Table A.3 MS/MSD Contractual Requirements

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

Verify that the matrix spike recoveries and RPD were calculated correctly using equations VII.1 and VII.2.

$$\% \ Recovery = \frac{SSR - SR}{SA} \times 100\% \qquad (VII.1)$$

where:

SSR = Spiked sample result
SR = Sample result
SA = Spike added

$$RPD = \frac{|MSR - MSDR|}{1/2\ (MSR + MSDR)} \times 100\% \qquad (VII.2)$$

where:

RPD = Relative percent difference
MSR = Matrix spike recovery
$MSDR$ = Matrix spike duplicate recovery

IX. Internal Standards

Table A.4 contains the volatile internal standards and their corresponding target compounds. These criteria have been established for packed columns only. Specific criteria for capillary columns have not been included in the SOW at this time.

Table A.4 Internal Standards and Their Corresponding Target Compounds

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	Bromoform	1,1,2,2-Tetrachloroethane
Methylene Chloride	1,2-Dichloropropane	Toluene
Acetone	trans-1,3-Dichloropropene	Chlorobenzene
Carbon Disulfide	Trichloroethene	Ethylbenzene
1,1-Dichloroethene	Dibromochloromethane	Styrene
1,1-Dichloroethane	1,1,2-Trichloroethane	Total Xylenes
1,2-Dichloroethene(total)	Benzene	Bromofluorobenzene (SMC)
Chloroform	cis-1,3-Dichloropropene	Toluene-d_8 (SMC)
1,2-Dichloroethane	Bromoform	
2-Butanone		
1,2-Dichloroethane-d_4 (SMC)		

SMC = System Monitoring Compound

XI. Compound Quantitation and Reported Contract Required Quantitation Limits (CRQLs)

Check the reported positive sample results and quantitation limits with the quantitation lists and chromatograms using equations XI.1, XI.2, or XI.3. Characteristic ions for the volatile target compounds are contained in Table A.5. Characteristic ions for System Monitoring Compounds and Internal Standards are contained in Table A.6.

Concentration for waters:

$$ug/L = \frac{A_x \times I_s \times Df}{A_{is} \times RRF \times V_o} \qquad \text{(XI.1)}$$

Concentration for low level soils:
(Dry weight basis)

$$ug/Kg = \frac{A_x \times I_s}{A_{is} \times RRF \times W_s \times D} \qquad \text{(XI.2)}$$

Concentration for medium level soils:
(Dry weight basis)

$$ug/Kg = \frac{A_x \times I_s \times V_t \times 1000 \times Df}{A_{is} \times RRF \times V_a \times W_s \times D} \qquad \text{(XI.3)}$$

where:

Ax = area of characteristic ion (EICP) for compound being measured
A_{is} = area of characteristic ion (EICP) for the internal standard
I_s = amount of internal standard added (ng)
RRF = daily response factor for compound being measured
V_o = volume of water purged (mL)
W_s = weight of sample (g)
D = (100 - % moisture)/100% - conversion to dry weight
V_t = volume of methanol (mL)†
V_i = volume of extract added (uL) for purging
Df = dilution factor‡
V_a = volume of the aliquot of the methanol extract (uL) added to reagent water for purging

† This volume is typically 10.0 mL, even though only 1.0 mL is transferred to the vial. See the SOW for more details.

‡ The dilution factor for analysis of soil/sediment samples for volatiles by the medium level method is defined as the ratio of the number of microliters (uL) of methanol added to the reagent water for purging (V_a) to the number of microliters of the methanol extract of the sample contained in volume V_a. If no dilution is performed, then the dilution factor equals 1.0.

The CRQL for a diluted sample should be calculated as follows:

Adjusted CRQL = Non-adjusted CRQL x Sample Dilution Factor (XI.4)

For example, the adjusted CRQL for a water sample with a 10U non-diluted CRQL and a 1 to 100 dilution (100.0 dilution factor) would be 1000U, according to the following calculation:

$$1000U = 10U \times 100$$

The CRQL adjustment for dry weight for a soil sample should be calculated as follows:

$$Dry\ Weight\ CRQL = \frac{Non\text{-}adjusted\ CRQL}{\left(\frac{100\ -\ \%moisture}{100}\right)} \qquad (XI.5)$$

For example, the dry weight CRQL for a soil sample with a 10U non-adjusted CRQL and a 10% moisture would be 11U, according to the following calculation:

$$11U = \frac{10U}{\left(\frac{100\ -\ 10}{100}\right)}$$

Table A.5 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-Dichloropropane	63	65, 114
trans-1,3-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

Table A.5 Characteristic Ions for Volatile Target Compounds (Continued)

Analyte	Primary Ion*	Secondary Ion(s)
Tetrachloroethene	164	129, 131, 166
Toluene	92	92
Chlorobenzene	112	114
Ethyl benzene	106	91
Styrene	104	78, 103
Total Xylenes	106	91

** While m/z 43 is used for quantitation of 2-Butanone, m/z 72 must be present for positive identification.

* The primary ion should be used unless interferences are present, in which case, a secondary ion may be used.

Table A.6 Characteristic Ions for System Monitoring Compounds and Internal Standards for Volatile Organic Compounds

Compound	Primary Ion	Secondary Ion(s)
SYSTEM MONITORING COMPOUNDS		
4-Bromofluorobenzene	95	174, 176
1,2-Dichloroethane-d_4	65	102
Toluene-d_8	98	70, 100
INTERNAL STANDARDS		
Bromochloromethane	128	49, 130, 51
1,4-Difluorobenzene	114	63, 88
Chlorobenzene-d_5	117	82, 119

MULTI-MEDIA, MULTI-CONCENTRATION
CONTRACTUAL REQUIREMENTS AND EQUATIONS FOR SEMIVOLATILE DATA REVIEW

II. GC/MS Instrument Performance Check

Use equation II.1 to verify that the laboratory has not made errors in the calculation of the percent relative abundance.

For example, the percent relative abundance of m/z 443 (X) relative to m/z 442 (Y) is calculated as follows:

$$\%\ Relative\ Abundance = \frac{abundance\ of\ m/z\ 443}{abundance\ of\ m/z\ 442} \times 100\%$$

III. Initial Calibration

<u>Data Review Criteria:</u> All semivolatile target compounds and surrogates must have a Relative Response Factor (RRF) of greater than or equal to 0.05 and a percent relative standard deviation (%RSD) of less than or equal to 30%.

<u>Contractual Criteria:</u> The maximum %RSD for most semivolatile compounds is 20.5% and the minimum RRF criteria vary as specified in Table A.7 (The semivolatile compounds listed separately in Table 4 on page 52 are not contractually required to meet a maximum %RSD but do have to meet a contractual minimum RRF of 0.010). The contractual criteria for an acceptable initial calibration specifies that up to any 4 semivolatile target compounds may fail to meet minimum RRF or maximum %RSD as long as they have RRFs that are greater than or equal to 0.010, and %RSD of less than or equal to 40.0%.

Table A.7 Minimum RRF Criteria for Semivolatile Target Compounds

Semivolatile Compounds	Minimum RRF
Phenol	0.800
bis(-2-Chloroethyl)ether	0.700
2-Chlorophenol	0.800
1,3-Dichlorobenzene	0.600
1,4-Dichlorobenzene	0.500
1,2-Dichlorobenzene	0.400
2-Methylphenol	0.700
4-Methylphenol	0.600
N-Nitroso-di-propylamine	0.500
Hexachloroethane	0.300
Nitrobenzene	0.200
Isophorone	0.400
2-Nitrophenol	0.100
2,4-Dimethylphenol	0.200
bis(-2-Chloroethoxy)methane	0.300

Table A.7 Minimum RRF Criteria for Semivolatile Target Compounds (Continued)

Semivolatile Compounds	Minimum RRF
2,4-Dichlorophenol	0.200
1,2,4-Trichlorobenzene	0.200
Naphthalene	0.700
4-Chloro-3-methylphenol	0.200
2-Methylnaphthalene	0.400
2,4,6-Trichlorophenol	0.200
2,4,5-Trichlorophenol	0.200
2-Chloronaphthalene	0.800
Acenaphthylene	1.300
2,6-Dinitrotoluene	0.200
Acenaphthene	0.800
Dibenzofuran	0.800
2,4-Dinitrotoluene	0.200
4-Chlorophenyl-phenylether	0.400
Fluorene	0.900
4-Bromophenyl-phenylether	0.100
Hexachlorobenzene	0.100
Pentachlorophenol	0.050
Phenanthrene	0.700
Anthracene	0.700
Fluoranthene	0.600
Pyrene	0.600
Benzo(a)anthracene	0.800
Chrysene	0.700
Benzo(b)fluoranthene	0.700
Benzo(k)fluoranthene	0.700
Benzo(a)pyrene	0.700
Indeno(1,2,3-cd)pyrene	0.500
Dibenz(a,h)anthracene	0.400
Benzo(g,h,i)perylene	0.500
Nitrobenzene-d_5	0.200
2-Fluorobiphenyl	0.700
Terphenyl-d_{14}	0.500
Phenol-d_5	0.800
2-Fluorophenol	0.600
2-Chlorophenol-d_4	0.800
1,2-Dichlorobenzene-d_4	0.400

Initial calibration RRF and $\overline{RRF}$ are calculated using equations III.1 and III.2; %RSD is calculated using equations III.3 and III.4.

IV. Continuing Calibration

Data Review Criteria: All semivolatile target compounds should meet a %D criterion of ± 25%.

Contractual Criteria: The percent difference (%D) between the initial calibration $\overline{RRF}$ and the continuing calibration RRF is ± 25.0% for the compounds listed in Table A.4. The contractual criteria for an acceptable continuing calibration specifies that up to any 4 semivolatile target compounds may fail to meet minimum RRF or maximum %D as long as they have RRFs that are greater than or equal to 0.010, and %D of less than or equal to 40.0%.

Check the continuing calibration RRF calculations for semivolatile target compounds using equation III.1, and evaluate the %D between initial calibration $\overline{RRF}$ and continuing calibration RRF using equation IV.1.

VI. Surrogate Spikes

The semivolatile surrogate compounds and their contractual recovery limits are listed in Table A.8.

Table A.8 Semivolatile Surrogate Requirements

Surrogate	%Recovery Limits	
	Water Samples	Soil Samples
S1 Nitrobenzene-d_5	35 - 114	23 - 120
S2 2-Fluorobiphenyl	43 - 116	30 - 115
S3 Terphenyl-d_{14}	33 - 141	18 - 137
S4 Phenol-d_5	10 - 110	24 - 113
S5 2-Fluorophenol	21 - 110	25 - 121
S6 2,4,6-Tribromophenol	10 - 123	19 - 122
S7 2-Chlorophenol-d_4	33 - 110*	20 - 130*
S8 1,2-Dichlorobenzene-d_4	16 - 110*	20 - 130*

* Advisory limits

Use equation VI.1 to verify that the surrogate recoveries were calculated correctly.

VII. Matrix Spikes/Matrix Spike Duplicates

The matrix spike/matrix spike duplicate contractual requirements are listed in Table A.9.

Verify that the matrix spike recoveries and RPD were calculated correctly using equations VII.1 and VII.2.

IX. Internal Standards

Table A.10 contains the semivolatile internal standards and their corresponding target compounds.

Table A.9 Semivolatile MS/MSD Contractual Requirements

Compound	%R - Water	%R - Soil	RPD - Water	RPD - Soil
Phenol	12 - 110	26 - 90	≤ 42	≤ 35
2-Chlorophenol	27 - 123	25 - 102	≤ 40	≤ 50
1,4-Dichlorobenzene	36 - 97	28 - 104	≤ 28	≤ 27
N-Nitroso-di-n-propylamine	41 - 116	41 - 126	≤ 38	≤ 38
1,2,4-Trichlorobenzene	39 - 98	38 - 107	≤ 28	≤ 23
4-Chloro-3-methylphenol	23 - 97	26 - 103	≤ 42	≤ 33
Acenaphthene	46 - 118	31 - 137	≤ 31	≤ 19
4-Nitrophenol	10 - 80	11 - 114	≤ 50	≤ 50
2,4-Dinitrotoluene	24 - 96	28 - 89	≤ 38	≤ 47
Pentachlorophenol	9 - 103	17 - 109	≤ 50	≤ 47
Pyrene	26 - 127	35 - 142	≤ 31	≤ 36

Table A.10 Semivolatile Internal Standards and Their Corresponding Target Compounds

1,4-Dichlorobenzene-d_4	Naphthalene-d_8	Acenaphthene-d_{10}
Phenol	Nitrobenzene	Hexachlorocyclopentadiene
bis(2-Chloroethyl)ether	Isophorone	2,4,6-Trichlorophenol
2-Chlorophenol	2-Nitrophenol	2,4,5-Trichlorophenol
1,3-Dichlorobenzene	2,4-Dimethylphenol	2-Chloronaphthalene
1,4-Dichlorobenzene	bis(2-Chloroethoxy)methane	2-Nitroaniline
1,2-Dichlorobenzene	2,4-Dichlorophenol	Dimethyl phthalate
2-Methylphenol	1,2,4-Trichlorobenzene	Acenaphthylene
2,2'-oxybis-(1-Chloropropane)	Naphthalene	3-Nitroaniline
4-Methylphenol	4-Chloroaniline	Acenaphthene
N-Nitroso-Di-n-propylamine	Hexachlorobutadiene	2,4-Dinitrophenol
Hexachloroethane	4-Chloro-3-methylphenol	4-Nitrophenol
2-Fluorophenol (surr)	2-Methylnaphthalene	Dibenzofuran
Phenol-d_5 (surr)	Nitrobenzene-d_5 (surr)	2,4-Dinitrotoluene
2-Chlorobenzene-d_4 (surr)		2,6-Dinitrotoluene
1,2-Dichlorobenzene-d_4 (surr)		Diethyl phthalate
		4-Chlorophenyl-phenyl ether
		Fluorene
		4-Nitroaniline
		2-Fluorobiphenyl (surr)
		2,4,6-Tribromophenol (surr)

Phenanthrene-d_{10}	Chrysene-d_{12}	Perylene-d_{12}
4,6-Dinitro-2-methylphenol	Pyrene	Di-n-octyl phthalate
N-Nitrosodiphenylamine	Butylbenzyl phthalate	Benzo(b)fluoranthene
4-Bromophenyl phenyl ether	3,3'-Dichlorobenzidine	Benzo(k)fluoranthene
Hexachlorobenzene	Benzo(a)anthracene	Benzo(a)pyrene
Pentachlorophenol	bis(2-Ethylhexyl)phthalate	Indeno(1,2,3-cd)pyrene
Phenanthrene	Chrysene	Dibenz(a,h)anthracene
Carbazole	Terphenyl-d_{14} (surr)	Benzo(g,h,i)perylene
Anthracene		
Di-n-butyl phthalate		
Fluoranthene		

surr = surrogate compound

XI. Compound Quantitation and Reported Contract Required Quantitation Limits (CRQLs)

Check the reported positive sample results and quantitation limits with the quantitation lists and chromatograms using equations XI.6, XI.7, or XI.8. Equation XI.4 should be used to adjust the CRQL for a diluted sample, and equation XI.5 should be used to adjust the CRQL for a soil sample. Characteristic ions for semivolatile target compounds are contained in Table A.11. Characteristic ions for semivolatile surrogates and internal standards are contained in Table A.12. Characteristic ions for pesticides and Aroclors are contained in Table A.13.

Concentration for waters:

$$ug/L = \frac{A_x \times I_s \times V_t \times Df}{A_{is} \times RRF \times V_o \times V_i} \qquad (XI.6)$$

Concentration for soils/sediments:
(Dry weight basis)

$$ug/Kg = \frac{A_x \times I_s \times V_t \times Df}{A_{is} \times RRF \times V_i \times W_s \times D} \qquad (XI.7)$$

where:

Ax = area of characteristic ion (EICP) for compound being measured
A_{is} = area of characteristic ion (EICP) for the internal standard
I_s = amount of internal standard added (ng)
RRF = daily relative response factor for compound being measured
V_o = volume of water extracted (mL)
V_i = volume of extract injected (uL)
V_t = volume of concentrated extract (uL)
Df = dilution factor†
D = (100 - % moisture)/100% - conversion to dry weight
W_s = weight of sample (g)

† **The dilution factor for analysis of water samples for semivolatiles by the method specified in SOW OLM01.0 is calculated using equation XI.8. If no dilution is performed, then the dilution factor equals 1.0.**

$$Df = \frac{uL\ of\ the\ most\ concentated\ extract\ used + uL\ of\ clean\ solvent}{uL\ of\ the\ most\ concentrated\ extract\ used} \qquad (XI.8)$$

Table A.11 Characteristic Ions for Semivolatile Target Compounds

Analyte	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

Table A.11 Characteristic Ions for Semivolatile Target Compounds (Continued)

Parameter	Primary Ion	Secondary Ion(s)
2-Nitroaniline	65	92, 138
Dimethyl phthalate	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
Carbazole	167	166, 139
Di-n-butylphthalate	149	150, 104
Fluoranthene	202	101, 100
Pyrene	202	101, 100
Butylbenzylphthalate	149	91, 206
3,3'-Dichlorobenzidine	252	254, 126

Table A.11 Characteristic ions for Semivolatile Target Compounds (Continued)

Analyte	Primary Ion	Secondary Ion(s)
Benz(a)anthracene	228	229, 226
bis(2-Ethylhexyl)phthalate	149	167, 279
Chrysene	228	226, 229
Di-n-Octyl phthalate	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

Table A.12 Characteristic Ions for Semivolatile Surrogates and Internal Standards

Analyte	Primary Ion	Secondary Ion(s)
SURROGATES		
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
2-Chlorophenol-d_4	132	68, 134
1,2-Dichlorobenzene-d_4	152	115, 150
INTERNAL STANDARDS		
1,4-Dichlorobenzene-d_4	152	115
Naphthalene-d_8	136	68
Acenapthene-d_{10}	164	162, 160
Phenanthrene-d_{10}	188	94, 80
Chrysene-d_{12}	240	120, 236
Perylene-d_{12}	264	260, 265

Table A.13 Characteristic Ions for Pesticides/Aroclors

Analyte	Primary Ion	Secondary Ion(s)
alpha-BHC	183	181, 109
beta-BHC	181	183, 109
delta-BHC	183	181, 109
gamma-BHC (Lindane)	183	181, 109
Heptachlor	100	272, 274
Aldrin	66	263, 220
Heptachlor epoxide	353	355, 351
Endosulfan I	195	339, 341
Dieldrin	79	263, 279
4,4'-DDE	246	248, 176
Endrin	263	82, 81
Endrin ketone	317	67, 319
Endrin aldehyde	67	250, 345
Endosulfan II	337	339, 341
4,4'-DDD	235	237, 165
Endosulfan sulfate	272	387, 422
4,4'-DDT	235	237, 165
Methoxychlor	227	228
Chlordane (alpha and/or gamma)	373	375, 377
Toxaphene	159	231, 233
Arochlor-1016	222	260, 292
Arochlor-1221	190	222, 260
Arochlor-1232	190	222, 260
Arochlor-1242	222	256, 292
Arochlor-1248	292	362, 326
Arochlor-1254	292	362, 326
Arochlor-1260	360	362, 394

APPENDIX B

CONTRACTUAL REQUIREMENTS AND EQUATIONS

LOW CONCENTRATION WATER - LCW
(OLC01.0)

DRAFT 12/90
Revised 6/91

LOW CONCENTRATION WATER
CONTRACTUAL REQUIREMENTS AND EQUATIONS FOR VOLATILE DATA REVIEW

II. GC/MS Instrument Performance Check

Use equation II.1 to verify that the laboratory has not made errors the calculation of the percent relative abundance.

$$\%\ Relative\ Abundance = \frac{abundance\ of\ X}{abundance\ of\ Y} \times 100\% \qquad (II.1)$$

For example, the percent relative abundance of m/z 96 (X) relative to m/z 95 (Y) is calculated as follows:

$$\%\ Relative\ Abundance = \frac{abundance\ of\ m/z\ 96}{abundance\ of\ m/z\ 95} \times 100\%$$

III. Initial Calibration

<u>Data Review Criteria</u>: All volatile target compounds and system monitoring compounds must have a Relative Response Factor (RRF) of greater than or equal to 0.05 and a percent relative standard deviation (%RSD) of less than or equal to 30%.

<u>Contractual Criteria</u>: The maximum %RSD for most volatile compounds is 20.5% and the minimum RRF criteria vary as specified in the following table (The volatile compounds listed separately in Table 2 on page 13 are not contractually required to meet a maximum %RSD but do have to meet a contractual minimum RRF of 0.010). The contractual criteria for an acceptable initial calibration specifies that up to <u>any</u> 2 volatile target compounds may fail to meet minimum RRF or maximum %RSD as long as they have RRFs that are greater than or equal to 0.010, and %RSD of less than or equal to 40.0%.

Initial calibration RRFs and $\overline{RRF}$ are calculated using equations III.1 and III.2.

$$RRF = \frac{A_x}{A_{is}} \times \frac{C_{is}}{C_x} \qquad (III.1)$$

$$\overline{RRF} = \frac{\sum_{i=1}^{5} RRF_i}{5} \qquad (III.2)$$

where:

RRF_i = "i"th Relative Response Factor
A = Area of the characteristic ion (EICP) measured
C = Concentration
is = Internal standard
x = Analyte of interest

Table B.1. Technical Acceptance Criteria for Initial and Continuing Calibration for Volatile Organic Compounds

Target Volatile Compound	Minimum RRF	Maximum %RSD	%D
Benzene	0.500	30.0	±30.0
Bromochloromethane	0.100	30.0	±30.0
Bromodichloromethane	0.200	30.0	±30.0
Bromoform	0.100	30.0	±30.0
Bromomethane	0.100	30.0	±30.0
Carbon tetrachloride	0.100	30.0	±30.0
Chlorobenzene	0.500	30.0	±30.0
Chloroform	0.200	30.0	±30.0
Dibromochloromethane	0.100	30.0	±30.0
1,2-Dibromoethane	0.100	30.0	±30.0
1,2-Dichlorobenzene	0.400	30.0	±30.0
1,3-Dichlorobenzene	0.600	30.0	±30.0
1,4-Dichlorobenzene	0.500	30.0	±30.0
1,1-Dichloroethane	0.200	30.0	±30.0
1,2-Dichloroethane	0.100	30.0	±30.0
1,1-Dichloroethene	0.100	30.0	±30.0
cis-1,3-Dichloropropene	0.200	30.0	±30.0
trans-1,3-Dichloropropene	0.100	30.0	±30.0
Ethylbenzene	0.100	30.0	±30.0
Styrene	0.300	30.0	±30.0
1,1,2,2-Tetrachloroethane	0.500	30.0	±30.0
Tetrachloroethene	0.200	30.0	±30.0
Toluene	0.400	30.0	±30.0
1,1,1-Trichloroethane	0.100	30.0	±30.0
1,1,2-Trichloroethane	0.100	30.0	±30.0
Trichloroethene	0.300	30.0	±30.0
Vinyl Chloride	0.100	30.0	±30.0
Xylenes (total)	0.300	30.0	±30.0
4-Bromofluorobenzene	0.200	30.0	±30.0

The %RSD is calculated using equations III.3 and III.4.

$$\sigma = \sqrt{\sum_{i=1}^{n} \frac{(x_i-\bar{x})^2}{(n-1)}} \qquad \text{(III.3)}$$

$$\%RSD = \frac{\sigma}{\bar{x}} \times 100 \qquad \text{(III.4)}$$

where:

σ = Standard deviation of 5 relative response factors

$\bar{x}$ = Mean of 5 relative response factors

IV. Continuing Calibration

Data Review Criteria: All volatile target compounds should meet a %D criterion of ± 30%.

Contractual Criteria: The percent difference (%D) between the initial calibration $\overline{\text{RRF}}$ and the continuing calibration RRF is ± 30% for all compounds listed in Table B.1 (Page B-2). The contractual criteria for an acceptable continuing calibration specifies that up to any 2 volatile target compounds may fail to meet minimum RRF or maximum %D as long as they have RRFs that are greater than or equal to 0.010, and %D of less than or equal to 40.0%.

Check the RRF calculations for volatile target compounds using equation III.1 (Page B-1). The %D between initial calibration $\overline{\text{RRF}}$ and continuing calibration RRF is calculated using equation IV.1.

$$\%\ D = \frac{\overline{RRF}_I - RRF_C}{\overline{RRF}_I} \times 100\% \qquad \text{(IV.1)}$$

where:

$\overline{RRF}_i$ = average relative response factor from initial calibration.

RRF_c = relative response factor from continuing calibration standard.

VI. Surrogate Spikes

The volatile surrogate compound and the contractual recovery limits are listed below.

Surrogate Spike	%Recovery Limits
BFB Bromofluorobenzene	80 - 120

Use equation VI.2 to check that the surrogate percent recovery was calculated correctly:

$$\% \ Recovery = \frac{Q_D}{Q_A} \times 100\% \qquad (VI.2)$$

where:

Q_D = Quantity determined by analysis.
Q_A = Quantity added to samples/blanks.

VII. Laboratory Control Samples (LCS)

Laboratory Control Sample compounds are listed in Table B.2. The contractual percent recovery limits are from **60 to 140 percent**. However, these limits may eventually be expanded by the Agency during the period of performance if the limits are found to be too restrictive.

Table B.2 Volatile Laboratory Control Sample Compounds

Vinyl Chloride
1,2-Dichloroethane
Carbon Tetrachloride
1,2-Dichloropropane
Trichloroethene
1,1,2-Trichloroethane
Benzene
cis-1,3-Dichloropropene
Bromoform
Tetrachloroethene
1,2-Dibromoethane
1,4-Dichlorobenzene

Check that the LCS recovery was calculated correctly by using equation VI.2.

IX. Internal Standards

Table B.3 contains the volatile internal standards and their corresponding target compounds.

Table B.3. Volatile Internal Standards and Their Corresponding Target Compounds

1,4-Difluorobenzene	Chlorobenzene-d_5	1,4-Dichlorobenzene d_4
Chloromethane	1,1,1-Trichloroethane	Bromoform
Bromomethane	Carbon tetrachloride	1,2-Dibromo-3-chloropropane*
Vinyl Chloride	Bromodichloromethane	1,2-Dichlorobenzene*
Chloroethane	1,2-Dichloropropane	1,3-Dichlorobenzene*
Bromochloromethane*	cis-1,3-Dichloropropene	1,4-Dichlorobenzene*
Methylene Chloride	Trichloroethene	
Acetone	Dibromochloromethane	
Carbon disulfide	1,1,2-Trichloroethane	
1,1-Dichloroethene	Benzene	
1,1-Dichloroethane	trans-1,3-Dichloropropene	
cis-1,2-Dichloroethene**	4-Methyl-2-Pentanone	
trans-1,2-Dichloroethene**	2-Hexanone	
Chloroform	Tetrachloroethene	
1,2-Dichloroethane	1,1,2,2-Tetrachloroethane	
2-Butanone	1,2-Dibromoethane*	
4-Bromofluorobenzene (surr)	Toluene	
	Chlorobenzene	
	Ethylbenzene	
	Styrene	
	Total Xylenes	

* compounds not on Multi-media, Multi-concentration TCL

** on Multi-media, Multi-concentration TCL as total 1,2-Dichloroethene

XI. Compound Quantitation and Reported Contract Required Quantitation Limits (CRQLs)

Check the reported positive sample results and quantitation limits with the quantitation lists and chromatograms using equation XI.1. Primary and secondary Quantitation ions are listed in Table B.4 (Page B-7).

$$ug/L = \frac{A_x \times I_s \times D_f}{A_{is} \times RRF \times V_o} \qquad \text{(XI.1)}$$

where:

Ax = area of characteristic ion (EICP) for compound being measured
A_{is} = area of characteristic ion for the internal standard
I_s = amount of internal standard added (ng)
RRF = relative response factor for compound being measured
V_o = volume of water purged (mL)
Df = dilution factor

The CRQL for a diluted sample should be calculated as follows:

$$\text{Adjusted CRQL} = \text{Non-adjusted CRQL} \times \text{Sample Dilution Factor} \qquad (XI.4)$$

For example, the adjusted CRQL for a water sample with a 10U non-diluted CRQL and a 1 to 100 dilution (100.0 dilution factor) would be 1000U, according to the following calculation:

$$1000U = 10U \times 100$$

TABLE B.4 Volatile Quantitation Ions

Volatile Target Compounds	Primary Quantitation Ion	Secondary Ions
Acetone	43	58
Benzene	78	---
Bromochloromethane	128	49, 130
Bromodichloromethane	83	85, 127
Bromoform	173	175, 254
Bromomethane	94	96
2-Butanone	43	72*
Carbon disulfide	76	78
Carbon tetrachloride	117	119
Chlorobenzene	112	77, 114
Chloroethane	64	66
Chloroform	83	85
Chloromethane	50	52
Dibromochloromethane	129	127
1,2-Dibromo-3-chloropropane	75	155, 157
1,2-Dibromoethane	107	109, 188
1,2-Dichlorobenzene	146	111, 148
1,3-Dichlorobenzene	146	111, 148
1,4-Dichlorobenzene	146	111, 148
1,1-Dichloroethane	63	65, 83
1,2-Dichloroethane	62	98
1,1-Dichloroethene	96	61, 63
cis-1,2-Dichloroethene	96	61, 98
trans-1,2-Dichloroethene	96	61, 98
1,2-Dichloropropane	63	112
cis-1,3-Dichloropropene	75	77

* Quantitation of this analyte is based on m/z 43 but m/z 72 must be present in the spectrum.

TABLE B.4 Volatile Quantitation Ions (Continued)

Volatile Target Compounds	Primary Quantitation Ion	Secondary Ions
trans-1,3-Dichloropropene	75	77
Ethylbenzene	91	106
2-Hexanone	43	58, 57, 100
Methylene chloride	84	86, 49
4-Methyl-2-pentanone	43	58, 100
Styrene	104	78
1,1,2,2-Tetrachloroethane	83	131, 85
Tetrachloroethene	166	168, 129
Toluene	91	92
1,1,1-Trichloroethane	97	99, 61
1,1,2-Trichloroethane	97	83, 85, 99, 132, 134
Trichloroethene	95	130, 132
Vinyl chloride	62	64
Xylenes (total)	106	91
SURROGATE COMPOUND AND INTERNAL STANDARDS:		
4-Bromofluorobenzene	95	174, 176
Chlorobenzene-d_5	117	82, 119
1,4-Dichlorobenzene-d_4	150	115, 152
1,4-Difluorobenzene	114	63, 88

LOW CONCENTRATION WATER
CONTRACTUAL REQUIREMENTS AND EQUATIONS FOR SEMIVOLATILE DATA REVIEW

II. GC/MS Instrument Performance Check

Use equation II.1 (Page B-1) to verify that the laboratory has not made errors the calculation of the percent relative abundance.

For example, the percent relative abundance of m/z 443 (X) relative to m/z 442 (Y) is calculated as follows:

$$\%\ Relative\ Abundance = \frac{abundance\ of\ m/z\ 443}{abundance\ of\ m/z\ 442} \times 100\%$$

III. Initial Calibration

<u>Data Review Criteria:</u> All semivolatile target compounds and surrogates must have a Relative Response Factor (RRF) of greater than or equal to 0.05 and a percent relative standard deviation (%RSD) of less than or equal to 30%.

<u>Contractual Criteria:</u> The maximum %RSD for most semivolatile compounds is 20.5% and the minimum RRF criteria vary as specified in the following table (The semivolatile compounds listed separately in table 4 on page 52 are not contractually required to meet a maximum %RSD but do have to meet a contractual minimum RRF of 0.010). The contractual criteria for an acceptable initial calibration specifies that up to <u>any</u> 4 semivolatile target compounds may fail to meet minimum RRF or maximum %RSD as long as they have RRFs that are greater than or equal to 0.010, and %RSD of less than or equal to 40.0%.

Initial calibration RRFs and $\overline{RRF}$ are calculated using equations III.1 and III.2 (Page B-1); %RSD is calculated using equations III.3 and III.4 (Page B-3).

IV. Continuing Calibration

<u>Data Review Criteria:</u> All semivolatile target compounds should meet a %D criterion of ± 25%.

<u>Contractual Criteria:</u> The percent difference (%D) between the initial calibration $\overline{RRF}$ and the continuing calibration RRF is ± 25.0% for the compounds listed in Table B.5. The contractual criteria for an acceptable continuing calibration specifies that up to <u>any</u> 4 semivolatile target compounds may fail to meet minimum RRF or maximum %D as long as they have RRFs that are greater than or equal to 0.010, and %D of less than or equal to 40.0%.

Check the RRF calculations for semivolatile target compounds using equation III.1 (Page B-1), and evaluate the %D between initial calibration $\overline{RRF}$ and continuing calibration RRF using equation IV.1 (Page B-3).

Table B.5. Acceptance Criteria for Initial and Continuing Calibration for Semivolatile Organic Compounds

Semivolatile Compounds	Minimum RRF	Maximum %RSD	%D
Phenol	0.800	20.5	±25.0
bis(-2-Chloroethyl)ether	0.700	20.5	±25.0
2-Chlorophenol	0.700	20.5	±25.0
2-Methylphenol	0.700	20.5	±25.0
4-Methylphenol	0.600	20.5	±25.0
N-Nitroso-di-n-propylamine	0.500	20.5	±25.0
Hexachloroethane	0.300	20.5	±25.0
Nitrobenzene	0.200	20.5	±25.0
Isophorone	0.400	20.5	±25.0
2-Nitrophenol	0.100	30.0	±30.0
2,4-Dimethylphenol	0.200	30.0	±30.0
bis(2-Chloroethoxy)methane	0.300	20.5	±25.0
2,4-Dichlorophenol	0.200	20.5	±25.0
1,2,4-Trichlorobenzene	0.200	20.5	±25.0
Naphthalene	0.700	20.5	±25.0
4-Chloro-3-methylphenol	0.200	20.5	±25.0
2-Methylnaphthalene	0.400	20.5	±25.0
2,4,6-Trichlorophenol	0.200	20.5	±25.0
2,4,5-Trichlorophenol	0.200	20.5	±25.0
2-Chloronaphthalene	0.800	20.5	±25.0
Acenaphthylene	1.300	20.5	±25.0
Acenaphthene	0.800	20.5	±25.0
Dibenzofuran	0.800	20.5	±25.0
2,4-Dinitrotoluene	0.200	30.0	±30.0
2,6-Dinitrotoluene	0.200	20.5	±25.0
4-Chlorophenyl-phenylether	0.400	20.5	±25.0
Fluorene	0.900	20.5	±25.0
4-Bromophenyl-phenylether	0.100	20.5	±25.0
Hexachlorobenzene	0.100	20.5	±25.0
Pentachlorophenol	0.050	20.5	±25.0
Phenanthrene	0.700	20.5	±25.0
Anthracene	0.700	20.5	±25.0
Fluoranthene	0.600	20.5	±25.0

Table B.5. Acceptance Criteria for Initial and Continuing Calibration for Semivolatile Organic Compounds (continued)

Semivolatile Compounds	Minimum RRF	Maximum %RSD	%D
Pyrene	0.600	20.5	±25.0
Benz(a)anthracene	0.800	20.5	±25.0
Chrysene	0.700	20.5	±25.0
Benzo(b)fluoranthene	0.700	20.5	±25.0
Benzo(k)fluoranthene	0.700	20.5	±25.0
Benzo(a)pyrene	0.700	20.5	±25.0
Indeno(1,2,3-cd)pyrene	0.500	20.5	±25.0
Dibenz(a,h)anthracene	0.400	20.5	±25.0
Benzo(g,h,i)perylene	0.500	20.5	±25.0
Phenol-d_5 (surr)	0.800	20.5	±25.0
2-Fluorophenol (surr)	0.600	20.5	±25.0
Terphenyl-d_{14} (surr)	0.500	20.5	±25.0
2-Fluorobiphenyl (surr)	0.700	20.5	±25.0

VI. Surrogate Spikes

Surrogate spike recoveries for semivolatile samples and blanks must be within the limits specified in Table B.6.

Table B.6 Semivolatile Surrogate Recovery Requirements

Surrogate Compound	% Recovery
Nitrobenzene-d_5	40 - 112
2-Fluorobiphenyl	42 - 110
p-Terphenyl-d_{14}	24 - 140
Phenol-d_5	17 - 113
2-Fluorophenol	16 - 110
2,4,6-Tribromophenol	18 - 126

Use equation VI.2 to verify that the surrogate recoveries were calculated correctly.

VII. Laboratory Control Samples (LCS)

The percent recovery for each of the compounds in the LCS spiking solution must be within the recovery limits listed in Table B.7. However, these limits may eventually be expanded by the Agency during the period of performance if the limits are found to be too restrictive.

Table B.7 Semivolatile Laboratory Control Sample Compounds and Recevery Limits

Compound	%Recovery
Phenol	44 - 120
2-Chlorophenol	58 - 110
4-Chloroaniline	35 - 98
2,4,6-Trichlorophenol	65 - 110
bis(2-Chloroethyl)ether	64 - 110
N-Nitroso-di-n-propylamine	34 - 102
Hexachloroethane	32 - 77
Isophorone	49 -110
1,2,4-Trichlorobenzene	44 - 96
Naphthalene	56 - 160
2,4-Dinitrotoluene	61 - 140
Diethylphthalate	76 - 104
N-Nitrosodiphenylamine	35 - 120
Hexachlorobenzene	30 - 95
Benzo(a)pyrene	55 - 92

Check that the recoveries were calculated correctly by using equation VI.2 (Page B-4).

IX. Internal Standards

Table B.8 (Page B-14) contains the semivolatile internal standards and their corresponding target compounds.

XI. Compound Quantitation and Reported Contract Required Quantitation Limits (CRQLs)

Check the reported positive sample results and quantitation limits with the quantitation lists and chromatograms using equation XI.6. Equation XI.4 (Page B-6) should be used to adjust the CRQL for a diluted sample. Table B.9 (Page B-15,16,17) contains the semivolatile primary and secondary Quantitation ions.

$$ug/L = \frac{A_x \times I_s \times V_t \times Df}{A_{is} \times RRF \times V_o \times V_i} \qquad \text{(XI.6)}$$

where:

Ax = area of characteristic ion (EICP) for compound being measured
A_{is} = area of characteristic ion (EICP) for the internal standard
I_s = amount of internal standard added (ng)
RRF = daily relative response factor for compound being measured
V_o = volume of water extracted (mL)
V_i = volume of extract injected (uL)
V_t = volume of concentrated extract (uL)
Df = dilution factor

Table B.8. Semivolatile Internal Standards and Their Corresponding Target Compounds

1,4-Dichlorobenzene-d_4	Naphthalene-d_8	Acenaphthene-d_{10}
Phenol	Nitrobenzene	Hexachlorocyclopentadiene
bis(2-Chloroethyl)ether	Isophorone	2,4,6-Trichlorophenol
2-Chlorophenol	2-Nitrophenol	2,4,5-Trichlorophenol
2-Methylphenol	2,4-Dimethylphenol	2-Chloronaphthalene
2,2'-oxybis-(1-Chloropropane)	bis(2-Chloroethoxy)methane	2-Nitroaniline
4-Methylphenol	2,4-Dichlorophenol	Dimethyl phthalate
N-Nitroso-di-n-propylamine	1,2,4-Trichlorobenzene	Acenaphthylene
2-Fluorophenol (surr)	Naphthalene	3-Nitroaniline
Phenol-d_5 (surr)	4-Chloroaniline	Acenaphthene
	Hexachlorobutadiene	2,4-Dinitrophenol
	4-Chloro-3-methylphenol	4-Nitrophenol
	2-Methylnaphthalene	Dibenzofuran
	Nitrobenzene-d_5 (surr)	2,4-Dinitrotoluene
		2,6-Dinitrotoluene
		Diethyl phthalate
		4-Chlorophenyl-phenyl ether
		Fluorene
		4-Nitroaniline
		2-Fluorobiphenyl (surr)
		2,4,6-Tribromophenol (surr)

surr = surrogate compound

Phenanthrene-d_{10}	Chrysene-d_{12}	Perylene-d_{12}
4,6-Dinitro-2-methylphenol	Pyrene	Di-n-octyl phthalate
N-Nitrosodiphenylamine	Butylbenzyl phthalate	Benzo(b)fluoranthene
4-Bromophenyl phenyl ether	3,3'-Dichlorobenzidine	Benzo(k)fluoranthene
Hexachlorobenzene	Benzo(a)anthracene	Benzo(a)pyrene
Pentachlorophenol	bis(2-Ethylhexyl)phthalate	Indeno(1,2,3-cd)pyrene
Phenanthrene	Chrysene	Dibenz(a,h)anthracene
Anthracene	Terphenyl-d_{14} (surr)	Benzo(g,h,i) perylene
Di-n-butyl phthalate		
Fluoranthene		

surr = surrogate compound

Table B.9 Semivolatile Quantitation Ions

Analyte	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-n-propylamine	70	42, 101, 130
Hexachloroethane	117	201, 199
Nitrobenzene	77	123, 65
Isophorone	82	95, 138
2-Nitrophenol	139	65, 109
2,4-Dimethylphenol	107	121, 122
bis(-2-Chloroethoxy)methane	93	95, 123
2,4-Dichlorophenol	162	164, 98
1,2,4-Trichlorobenzene	180	182, 145
Naphthalene	128	129, 127
4-Chloroaniline	127	129
Hexachlorobutadiene	225	223, 227
4-Chloro-3-methylphenol	107	144, 142
2-Methylnaphthalene	142	141
Hexachlorocyclopentadiene	237	235, 272
2,4,6-Trichlorophenol	196	198, 200
2,4,5-Trichlorophenol	196	198, 200
2-Chloronaphthalene	162	164, 127
2-Nitroaniline	65	92, 138

Table B.9 Semivolatile Quantitation Ions (Continued)

Analyte	Primary Ion	Secondary Ion(s)
Dimethyl phthalate	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-octyl phthalate	149	---

Table B.9 Semivolatile Quantitation Ions (Continued)

Analyte	Primary Ion	Secondary Ion(s)
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		
Phenol-d_5	99	42, 71
2-Fluorophenol	112	64
2,4,6-Tribromophenol	330	332, 141
d-5 Nitrobenzene	82	128, 54
2-Fluorobiphenyl	172	171
Terphenyl	244	122, 212
Internal Standards		
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

APPENDIX C

CONTRACTUAL REQUIREMENT COMPARISON TABLES

Table C.1. Comparison of Requirements for
Volatile Data Review

REQUIREMENT	MULTI-MEDIA, MULTI-CONCENTRATION	LOW CONCENTRATION WATERS
Target Compound List	33 Target Compounds	40 Target Compounds
Data Turnaround	35 days	14 days
Technical Holding Time	7 days if not preserved 14 days if preserved	7 days if not preserved 14 days if preserved
Initial Calibration	5 levels: 10 - 200 ug/L	5 levels: 1 - 25 ug/L (5 - 125 for Ketones)
Continuing Calibration	mid-level: 50 ug/L	mid-level: 5 ug/L (25 for Ketones)
Blanks	Method Blanks Instrument Blanks	Method Blanks Instrument Blanks Storage Blanks
SMC/Surrogates	SMC: 1,2-Dichloroethane-d_4 Bromofluorobenzene Toluene-d_8	Surrogate: Bromofluorobenzene
MS/MSD	Frequency: 1 per 20 samples, per matrix	N/A
LCS	N/A	1 per SDG
Regional QA/QC	PEs - variable	PEs - 1 per SDG
Internal Standards	IS Area: - 50% to + 100% IS RT Shift: ± 30 sec. 3 compounds: Chlorobenzene-d_5 1,4-Difluorobenzene Bromochloromethane	IS Area: ± 40% IS RT Shift: ± 20 sec. 3 compounds: Chlorobenzene-d_5 1,4-Difluorobenzene 1,4-Dichlorobenzene
CRQL	10 ppb (water/low soil) 1200 ppb (med soil)	1 - 5 ug/L
TICs	largest 10 ≥10% of nearest IS	largest 10 ≥40% of nearest IS

Table C.2: Comparison of Requirements for
Semivolatile Data Review

REQUIREMENT	MULTI-MEDIA, MULTI-CONCENTRATION	LOW CONCENTRATION WATERS
Target Compound List	64 Target Compounds	60 Target Compounds
Data Turnaround	35 days	14 days
Technical Holding Time	Extraction - 5 days Analysis - 40 days after extraction	Extraction - 5 days Analysis - 40 days after extraction
Initial Calibration	5 levels: 20 - 160 ug/L	5 levels: varies
Continuing Calibration	mid-level: 50 ug/L	mid-level: varies
Blanks	Method Blanks Instrument Blanks	Method Blanks Instrument Blanks Storage Blanks
Surrogates	8 compounds	6 compounds
MS/MSD	Frequency: 1 per 20 samples, per matrix	N/A
LCS	N/A	1 per SDG
Regional QA/QC	PEs - variable	PEs - 1 per SDG
Internal Standards	IS Area: - 50% to + 100% IS RT Shift: ± 30 sec.	IS Area: - 50% to 100% IS RT Shift: ± 20 sec.
CRQLs	10 - 50 ppb (water) 330 - 1700 ppb (low soil) 10,000 - 50,000 (med soil)	5 - 20 ug/L
TICs	largest 20 ≥10% of nearest IS	largest 20 ≥50% of nearest IS

APPENDIX D

PROPOSED GUIDANCE FOR TENTATIVELY IDENTIFIED COMPOUNDS (VOA AND SV)

Proposed Guidance for Tentatively Identified Compounds (VOA)

A. **Review Items:** Form I VOA-TIC, chromatograms, library search printout and spectra for three TIC candidates, and GC retention time data.

B. **Objective**

Chromatographic peaks in volatile analyses that are not TCL compounds, system monitoring compounds, or internal standards are potential tentatively identified compounds (TICs) or library search compounds (LSCs). TICs must be qualitatively identified by a library search of the National Institute of Standards and Technology (NIST) mass spectral library, and the identifications assessed by the data reviewer.

C. **Criteria**

For each sample, the laboratory must conduct a library search of the NIST mass spectral library and report the possible identity for the 10 largest volatile fraction peaks which are not surrogates, internal standards, or TCL compounds, but which have a peak area greater than 40 percent of the peak area of the nearest internal standard. TIC results are reported for each sample on the Organic Analysis Data Sheet (Form I VOA-TIC).

Note: Since the SOW revision of October 1986, the CLP does not allow the laboratory to report as tentatively identified compounds any TCL compound which is properly reported in another fraction. (For example, late eluting volatile TCL compounds must not be reported as semivolatile TICs.)

D. **Evaluation**

1. Guidelines for Tentative Identification are as follows:

The interpretation of library search compounds (LSCs) is one of the aspects of data review which calls for the fullest exercise of professional judgement. The reviewer must be thoroughly familiar with the principles and practice of mass spectral interpretation and of gas chromatography. Because the interpretation process is labor-intensive, it is important to document the process involved in arriving at a tentative identification.

Worksheets for "Tentative Identification of Library Search Compounds" are provided in Appendix B for the volatile GC/MS fractions to assist in generating the information needed to make a reasonable tentative identification of the LSCs.

The process involved in tentatively identifying a library search compound may be summarized as follows:

a. Identify all samples in the related group (Case, SAS or SDG) in which the unknown compound occurs. Calculation of relative retention times (RRT) and comparison of RRT and mass spectral data across samples is extremely helpful in identifying unknowns that occur repeatedly in related samples. Use one worksheet per unknown for all samples in which it occurs.

b. Inspect the library search spectrum retrieved for each unknown, to determine if detailed mass spectral interpretation is necessary. Often, it is obvious that the correct match is among the spectra retrieved for the unknown from the several samples in which it is found. It may only be necessary to check the unknown's RRT versus a reference list of VOA (generated under similar conditions and after accounting for bias in the sample) to arrive at a satisfactory tentative identification. Some references are provided. If a reference RRT is not available, then a comparison of the unknown's RRT or boiling point to the RRT or boiling point of a closely related compound may also provide a satisfactory tentative identification. Within a compound class, retention time increases with increasing boiling point.

c. In the event that serious ambiguity still exists after examining the library spectra and RRT data, a full mass spectral interpretation can narrow down the possibilities. While a full discussion of manual mass spectral interpretation is beyond the scope of this document, several key points may be mentioned as important objects:

- Determine a likely molecular weight. Depending on the unknown, the MW may or may not be apparent due to the extent of fragmentation. The MW of the retrieved library spectra, interpreted in light of the RRT, may be helpful if the molecular ion is not present.

- Determine the isotope ratios (M+1)/M, (M+2)/M, (M+4)/M, etc. (where M is the molecular ion) and determine a short list of possible molecular formulas. Isotope ratios will also reveal the presence of S, Cl, and Br.

- Calculate the total number of rings-plus-double-bonds in the unknown by applying the following equation to the likely molecular formulas, to determine the degree of unsaturation.

  Number of rings-plus-double bonds (r+db):

  $$(r+db) = C - \frac{H}{2} - \frac{X}{2} + \frac{N}{2} + 1$$

  where:
  C = no. of carbons
  H = no. of hydrogens
  X = no. of halogens
  N = no. of nitrogens

  Note: oxygen and sulfur do not need to be accounted for. An aromatic ring counts as four rings and double bonds.

- Calculate the mass losses represented by major peaks in the unknown spectrum, and relate these to the fragmentation of neutral moieties from the molecular ion or other daughter ions.

- Using the information gathered on molecular weight, molecular formula, degree of unsaturation, and mass losses in the unknown spectrum, combined with the RRT data, give as precise a description of the unknown as possible, including an exact identification if it is justified.

d. In the event that the unknown spectrum is not that of a pure compound, mass spectral interpretation may not be possible. However, in some instances, a mixed spectrum may be recognized as two compounds having very similar relative retention times. Target compounds, surrogates and internal standards may also be responsible for extra ions in an unknown spectrum.

2. Check the raw data to verify that the laboratory has generated a library search spectrum for all required peaks in the chromatograms for samples and blanks.

3. Blank chromatograms should be examined to verify that TIC peaks present in samples are not found in blanks. When a low-level non-TCL compound that is a common artifact or laboratory contaminant is detected in a sample, a thorough check of blank chromatograms may require looking for peaks which are less than 40 percent of the internal standard peak area or height, but present in the blank chromatogram at similar relative retention time.

4. All mass spectra for every sample and blank must be examined.

5. The reviewer should be aware of common laboratory artifacts/contaminants and their sources (e.g., aldol condensation products, solvent preservatives, and reagent contaminants). These may be present in blanks and not reported as sample TICs.

Examples:

a. Common laboratory contaminants: CO_2 (m/z 44), siloxanes (m/z 73), diethyl ether, hexane, certain freons (1,1,2-trichloro-1,2,2-trifluoroethane or fluorotrichloromethane), and phthalates at levels less than 100 ug/L or 4000 ug/Kg.

b. Solvent preservatives such as cyclohexene which is a methylene chloride preservative. Related by-products include cyclohexanone, cyclohexenone, cyclohexanol, cyclohexenol, chlorocyclohexene, and chlorocyclohexanol.

c. Aldol condensation reaction products of acetone include: 4-hydroxy-4-methyl-2-pentanone, 4-methyl-2-penten-2-one, and 5,5-dimethyl-2(5H)-furanone.

6. Occasionally, a TCL compound may be identified in the proper analytical fraction by non-target library search procedures, even though it was not found on the quantitation list. If the total area quantitation method was used, the reviewer should request that the laboratory recalculate the result using the proper quantitation ion. In addition, the reviewer should evaluate other sample chromatograms and check library reference retention times on quantitation lists to determine whether the false negative result is an isolated occurrence or whether additional data may be affected.

7. TCL compounds may be identified in more than one fraction. Verify that quantitation is made from the proper fraction.

8. Library searches should not be performed on internal standards or surrogates.

9. TIC concentration should be estimated assuming a RRF of 1.0.

E. Action

1. All TIC results should be qualified as tentatively identified (N) with estimated concentrations (J) or (NJ).

2. General actions related to the review of TIC results are as follows:

    a. A non-TCL compound is not considered to be "tentatively identified" until the mass spectrum and retention time data have been reviewed according to the evaluation guidelines in XIII.D. The review should be documented on the Tentative Identification of Library Search Compound worksheet. The worksheet will be useful if a better library match for the unknown is retrieved in another Case, SAS, or SDG. It may also be used in writing a Special Analytical Service Statement of Work to identify the unknown, or if the sample is sent to an EPA research laboratc [illegible] .SC identification by multiple spectral techniques.

    b. If all contractually required peaks were not library searched, the desig[illegible] representative could request these data from the laboratory.

3. TIC results which are not sufficiently above the level in the blank should not be reported. (Dilutions and sample size must be taken into account when comparing the amounts present in blanks and samples.)

4. When a compound is not found in any blanks, but is a suspected artifact or common laboratory contaminant, the result may be qualified as unusable (R).

5. The reviewer may elect to report all similar isomers as a total. (All alkanes may be summarized and reported as total hydrocarbons.)

6. The data reviewer should state the degree of confidence (high, medium, low) in the tentative identification after completing the review process.

7. The complete "Tentative Identification of Library Search Compound" worksheet should be attached to the final data review report.

APPENDIX

Equation 1:

$$RI = 100 \frac{RTunk - RTz}{RTz+1 - RTz} + 100Z$$

where: RTunk is the retention time of the unknown
RTz is the retention time of the preceeding retention index standard
RTz+1 is the retention time of the following retention index standard
Z = number of rings in the retention index standard
RI = Lee Retention Index

Retention Index Standards

naphthalene	z=2	RI=200.00
phenanthrene	z=3	RI=300.00
chrysene	z=4	RI=400.00
Benzo(g,h,i) perylene	z=5	RI=500.00

Note: when these compounds are not dound in the sample of interest, RT data for the deuterated internal standards or most recent calibration may be used. Retention time shifts and bias must be accounted for.

Equation 2

Number of rings-plus-double bonds (r+db):

$$(r+db) = C - \frac{H}{2} - \frac{X}{2} + \frac{N}{2} + 1$$

where: C = no. of carbons
H = no. of hydrogens
X = no. of halogens
N = no. of nitrogens

Note: oxygen and sulfur do not need to be accounted for. An aromatic ring counts as four rings and double bonds.

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3. Silverstein, R.M., Bassler, G.C., and Morrill, T.C., Spectrometric Identification of Organic Compounds 4th ed., Wiley, New York, 1981.

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Proposed Guidance for Tentatively Identified Compounds (SV)

A. **Review Items:** Form I SV-TIC, chromatograms, library search printout and spectra for three TIC candidates, and GC retention time data.

B. Objective

Chromatographic peaks in semivolatile analyses that are not TCL compounds, surrogates, or internal standards are potential tentatively identified compounds (TICs) or library search compounds (LSCs). TICs must be qualitatively identified by a library search of the National Institute of Standards and Technology (NIST) mass spectral library, and the identifications assessed by the data reviewer.

C. Criteria

For each sample, the laboratory must conduct a library search of the NIST mass spectral library and report the possible identity for the 20 largest semivolatile fraction peaks which are not surrogates, internal standards, or TCL compounds, but which have a peak area greater than 50 percent of the peak area of the nearest internal standard. TIC results are reported for each sample on the Organic Analysis Data Sheet (Form I SV-TIC).

Note: Since the SOW revision of October 1986, the CLP does not allow the laboratory to report as tentatively identified compounds any TCL compound which is properly reported in another fraction. (For example, late eluting volatile TCL compounds must not be reported as semivolatile TICs.)

D. Evaluation

1. Guidelines for Tentative Identification are as follows:

The interpretation of library search compounds (LSCs) is one of the aspects of data review which calls for the fullest exercise of professional judgement. The reviewer must be thoroughly familiar with the principles and practice of mass spectral interpretation and of gas chromatography. Because the interpretation process is labor-intensive, it is important to document the process involved in arriving at a tentative identification.

Worksheets for "Tentative Identification of Library Search Compounds" are provided in Appendix B for the semivolatile GC/MS fractions to assist in generating the information needed to make a reasonable identification of the TICs.

The process involved in tentatively identifying a library search compound may be summarized as follows:

a) Identify all samples in the related group (Case, SAS or SDG) in which the unknown compound occurs. Calculation of retention indices (RI) and comparison of RI and mass spectra across samples is extremely helpful in identifying unknowns that occur repeatedly in related samples. Use one worksheet per unknown for all samples in which it occurs. Retention indices are calculated according to the following example:

$$RI = 100 \quad \frac{RTunk - RTz}{RTz+1 - RTz} + 100Z$$

where: RTunk is the retention time of the unknown
RTz is the retention time of the preceeding retention index standard
RTz+1 is the retention time of the following retention index standard
Z = number of rings in the retention index standard
RI = Lee Retention Index

<u>Retention Index Standards</u>

naphthalene	z=2	RI=200.00
phenanthrene	z=3	RI=300.00
chrysene	z=4	RI=400.00
Benzo(g,h,i) perylene	z=5	RI=500.00

<u>Note:</u> when these compounds are not dound in the sample of interest, RT data for the deuterated internal standards or most recent calibration may be used. Retention time shifts and bias must be accounted for.

b) Inspect the library search spectrum retrieved for each unknown, to determine if detailed mass spectral interpretation is necessary. Often, it is obvious that the correct match is among the spectra retrieved for the unknown from the several samples in which it is found. It may only be necessary to check the unknown's RI versus a reference list of SV (generated under similar conditions and after accounting for bias in the sample) to arrive at a satisfactory tentative identification. Some references are provided. If a reference RI is not available, then a comparison of the unknown's RI or boiling point to the RI or boiling point of a closely related compound may also provide a satisfactory tentative identification. Within a compound class, retention time increases with increasing boiling point.

c) In the event that serious ambiguity still exists after examining the library spectra and RI data, a full mass spectral interpretation can narrow down the possibilities. While a full discussion [illegible] manual mass spectral interpretation is beyond the scope of this document, several key points may be mentioned as important objects:

- o Determine a likely molecular weight. Depending on the unknown, the MW may or may not be apparent due to the extent of fragmentation. The MW of the retrieved library spectra, interpreted in light of the RI, may be helpful if the molecular ion is not present.
- o Determine the isotope ratios (M+1)/M, (M+2)/M, (M+4)/M, etc. (where M is the molecular ion) and determine a short list of possible molecular formulas. Isotope ratios will also reveal the presence of S, Cl, and Br.

- o Calculate the total number of rings-plus-double-bonds in the unknown by applying the follwing equation to the likely molecular formulas, to determine the degree of unsaturation.

  Number of rings-plus-double bonds (r+db):

  $$(r+db) = C - \frac{H}{2} - \frac{X}{2} + \frac{N}{2} + 1$$

  where: C = no. of carbons
  H = no. of hydrogens
  X = no. of halogens
  N = no. of nitrogens

  Note: oxygen and sulfur do not need to be accounted for. An aromatic ring counts as four rings and double bonds.

- o Calculate the mass losses represented by major peaks in the unknown spectrum, and relate these to the fragmentation of neutral moieties from the molecular ion or other daughter ions.

- o Using the information gathered on molecular weight, molecular formula, degree of unsaturation, and mass losses in the unknown spectrum, combined with the RI data, give as precise a description of the unknown as possible, including an exact identification if it is justified.

d) In the event that the unknown spectrum is not that of a pure compound, mass spectral interpretation may not be possible. However, in some instances, a mixed spectrum may be recognized as two compounds having very similar retention indices (for example, ortho-terphenyl, RI=317.43 and nonadecane, RI=317.20). This particular coelution would result in an unknown spectrum having a polycyclic aromatic pattern at m/z 230, the MW of terphenyl, with an hydrocarbon type pattern at m/z 43,57,71, etc. Target compounds, surrogates and internal standards may also be responsible for extra ions in an unknown spectrum, and may be treated similarly.

2. Check the raw data to verify that the laboratory has generated a library search spectrum for all required peaks in the chromatograms for samples and blanks.

3. Blank chromatograms should be examined to verify that TIC peaks present in samples are not found in blanks. When a low-level non-TCL compound that is a common artifact or laboratory contaminant is detected in a sample, a thorough check of blank chromatograms may require looking for peaks which are less than 10 percent of the internal standard peak area or height, but present in the blank chromatogram at similar relative retention time.

4. All mass spectra for every sample and blank must be examined.

5. The reviewer should be aware of common laboratory artifacts/contaminants and their sources (e.g., aldol condensation products, solvent preservatives, and reagent contaminants). These may be present in blanks and not reported as sample TICs.

   Examples:

   a. Common laboratory contaminants: CO_2 (m/z 44), siloxanes (m/z 73), diethyl ether, hexane, certain freons (1,1,2-trichloro-1,2,2-trifluoroethane or fluorotrichloromethane), and phthalates at levels less than 100 ug/L or 4000 ug/Kg.

   b. Solvent preservatives such as cyclohexene which is a methylene chloride preservative. Related by-products include cyclohexanone, cyclohexenone, cyclohexanol, cyclohexenol, chlorocyclohexene, and chlorocyclohexanol.

   c. Aldol condensation reaction products of acetone include: 4-hydroxy-4-methyl-2-pentanone, 4-methyl-2-penten-2-one, and 5,5-dimethyl-2(5H)-furanone.

6. Occasionally, a TCL compound may be identified in the proper analytical fraction by non-target library search procedures, even though it was not found on the quantitation list. If the total area quantitation method was used, the reviewer should request that the laboratory recalculate the result using the proper quantitation ion. In addition, the reviewer should evaluate other sample chromatograms and check library reference retention times on quantitation lists to determine whether the false negative result is an isolated occurrence or whether additional data may be affected.

7. TCL compounds may be identified in more than one fraction. Verify that quantitation is made from the proper fraction.

8. Library searches should not be performed on internal standards or surrogates.

9. TIC concentration should be estimated assuming a RRF of 1.0.

E. Action

1. All TIC results should be qualified as tentatively identified (N) with estimated concentrations (J) or (NJ).

2. General actions related to the review of TIC results are as follows:

   a. A non-TCL compound is not considered to be "tentatively identified" until the mass spectrum and retention time data have been reviewed as per section XIII D. The review should be documented on the Tentative Identification of Library Search Compound worksheet. The worksheet will be useful if a better library match for the unknown is retrieved in another Case, SAS, or SDG. It may also be used in writing a Special Analytical Service Statement of Work to identify the unknown, or if the sample is sent to an EPA research laboratory for LSC identification by multiple spectral techniques.

b. If all contractually required peaks were not library searched, the designated representative could request these data from the laboratory.

3. TIC results which are not sufficiently above the level in the blank should not be reported. (Dilutions and sample size must be taken into account when comparing the amounts present in blanks and samples.)

4. When a compound is not found in any blanks, but is a suspected artifact or common laboratory contaminant, the result may be qualified as unusable (R).

5. The reviewer may elect to report all similar isomers as a total. (All alkanes may be summarized and reported as total hydrocarbons.)

6. The data reviewer should state the degree of confidence (high, medium, low) in the tentative identification after completing the review process.

7. The complete "Tentative Identification of Library Search Compound" worksheet should be attached to the final data review report.

APPENDIX E

GLOSSARY OF TERMS

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GLOSSARY OF TERMS

APO	Administrative Project Officer
BFB	Bromofluorobenzene - volatile instrument performance check compound
BNA	Base/Neutral/Acid Compounds - compounds analyzed by semivolatile technique
Case	A finite, usually predetermined number of samples collected over a given time period for a particular site. A Case consists of one or more Sample Delivery Group(s).
CCS	Contract Compliance Screening - process in which SMO inspects analytical data for contractual compliance and provides results to the Regions, laboratories and EMSL/LV.
CF	Calibration Factor
CRQL	Contract Required Quantitation Limit
CSF	Complete SDG File
DFTPP	Decafluorotriphenylphosphine - semivolatile instrument performance check compound
DPO	Deputy Project Officer
EICP	Extracted Ion Current Profile
GC/EC	Gas Chromatography/Electron Capture Detector
GC/MS	Gas Chromatograph/Mass Spectrometer
GPC	Gel Permeation Chromatography - A sample clean-up technique that separates compounds by size and molecular weight. Generally used to remove oily materials from sample extracts.
IS	Internal Standards - Compounds added to every VOA and BNA standard, blank, matrix spike duplicate, and sample extract at a known concentration, prior to instrumental analysis. Internal standards are used as the basis for quantitation of the target compounds.
LCS	Laboratory Control Sample
MS/MSD	Matrix Spike/Matrix Spike Duplicate
m/z	The ratio of mass (m) to charge (z) of ions measured by GC/MS
OADS	Organic Analysis Data Sheet (Form I)
ORDA	Organic Regional Data Assessment - from earlier version of the Functional Guielines
NIST	National Institute of Standards and Technology

PCB	Polychlorinated biphenyl (Arochlor is a trademark)
PE	Sample Performance Evaluation Sample
QA	Quality Assurance - Total program for assuring the reliability of data.
QC	Quality Control - Routine application of procedures for controlling the monitoring process.
RIC	Reconstructed Ion Chromatogram
RPD	Relative Percent Difference (between matrix spike and matrix spike duplicate)
RRF	Relative Response Factor
$\overline{\mathrm{RRF}}$	Average Relative Response Factor
RRT	Relative Retention Time (with relation to internal standard)
RSD	Relative Standard Deviation
RT	Retention Time
SDG	Sample Delivery Group - Defined by one of the following, whichever occurs first: ▪ Case of field samples ▪ Each 20 field samples within a Case ▪ Each 14-day calendar period during which field samples in a Case are received, beginning with receipt of the first sample in the SDG. (For VOA contracts, the calendar period is 7-day.)
SMC	System Monitoring Compound - formerly surrogates for volatile analysis.
SMO	Sample Management Office
SOP	Standard Operating Procedure
SOW	Statement of Work
SV	Semivolatile analysis - Method based on analysis by GC/MS for BNA organic compounds.
TCL	Target Compound List
TIC	Tentatively Identified Compound - A compound tentatively identified from search of the NIST mass spectral library that is not on the TCL.
TPO	Technical Project Officer

VOA	Volatile Organic Analysis - Method based on the purge and trap technique for organic compound analysis.
VTSR	Validated Time of Sample Receipt - Time of sample receipt at the laboratory as recorded on the shipper's delivery receipt and Sample Traffic Report.

LABORATORY DATA VALIDATION

FUNCTIONAL GUIDELINES FOR EVALUATING INORGANICS ANALYSES

Prepared for the

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INTRODUCTION

This document is designed to offer guidance in laboratory data evaluation and validation. In some aspects, it is equivalent to a Standard Operating Procedure (SOP). In other, more subjective areas, only general guidance is offered due to the complexities and uniqueness of data relative to specific samples. These Guidelines have been updated to include all requirements in the 7/87 Statement of Work (SOW) for Inorganics, Amendment 1 and December 1987 Revisions.

Those areas where specific SOPs are possible are primarily areas in which definitive performance requirements are established. These requirements are concerned with specifications that are not sample dependent; they specify performance requirements on matters that should be fully under a laboratory's control. These specific areas include blanks, calibration standards, calibration verification standards, laboratory control standards, and interference check standards. In particular, mistakes such as calculation and transcription errors must be rectified by resubmission of corrected data sheets.

This document is intended for technical review. Some areas of overlap between technical review and Contract Compliance Screening (CCS) exist; however, determining contract compliance is not intended to be a goal of these guidelines. It is assumed that the CCS is available and can be utilized to assist in the data review procedure.

At times, there may be an urgent need to use data which do not meet all contract requirements and technical criteria. Use of these data does not constitute either a new requirement standard or full acceptance of the data. Any decision to utilize data for which performance criteria have not been met is strictly to facilitate the progress of projects requiring the availability of the data. A contract laboratory submitting data which are out of specification may be required to rerun or resubmit data even if the previously submitted data have been utilized due to urgent program needs; data which do not meet specified requirements are never fully acceptable. The only exception to this requirement is in the area of requirements for individual sample analysis; if the nature of the sample itself limits the attainment of specifications, appropriate allowances must be made. The overriding concern of the Agency is to obtain data which are technically valid and legally defensible.

All data reviews must have, as a cover sheet, the Inorganic Regional Data Assessment (IRDA) form. (A copy is attached at the end of this document.) If mandatory actions are required, they should be specifically noted on this form. In addition, this form is to be used to summarize overall deficiencies requiring attention, as well as general laboratory performance and any discernible trends in the quality of the data. (This form is not a replacement for the data review.) Sufficient supplementary documentation must accompany the form to clearly identify the problems associated with a Case. The form and any attachments must be submitted to the Contract Laboratory Program Quality Assurance Coordinator (CLP QAC), the Regional Deputy Project Officer (DPO), and the Environmental Monitoring Systems Laboratory in Las Vegas (EMSL/LV).

It is the responsibility of the data reviewer to notify the Regional DPO concerning problems and shortcomings with regard to laboratory data. If there is an urgent requirement, the DPO may be contacted by telephone to expedite corrective action. It is recommended that all items for DPO action be presented at one time. In any case, the Inorganic Regional Data Assessment form must be completed and submitted.

PRELIMINARY REVIEW

In order to use this document effectively, the reviewer should have a general overview of the Case at hand. The exact number of samples, their assigned numbers, their matrix, and the number of laboratories involved in their analysis are essential information. Background information on the site is helpful but often this information is very difficult to locate. The site project officer is the best source for answers or further direction.

CCS is a source of a large quantity of summarized information. It can be used to alert the reviewer of problems in the Case or what may be sample-specific problems. This information may be utilized in data validation. If CCS is unavailable, those criteria affecting data validity must be addressed by the data reviewer.

Cases routinely have unique samples which require special attention by the reviewer. Field blanks, field duplicates, and performance audit samples need to be identified. The sampling records should provide:

1. Project Officer for site
2. Complete list of samples with notations on
    - a) sample matrix
    - b) blanks*
    - c) field duplicates*
    - d) field spikes*
    - e) QC audit sample*
    - f) shipping dates
    - g) labs involved

* If applicable

The chain-of-custody record includes sample descriptions and date of sampling. Although sampling date is not addressed by contract requirements, the reviewer must take into account lag time between sampling and shipping while assessing sample holding times.

INORGANICS PROCEDURE

The requirements to be checked in validation are listed below. ("CCS" indicates that the contractual requirements for these items will also be checked by CCS; CCS requirements are not always the same as the data review criteria.)

I. Holding Times (CCS - Lab holding times only)

II. Calibration

- o Initial (CCS)
- o Initial and Continuing Calibration Verification (CCS)

III. Blanks (CCS)

IV. ICP Interference Check Sample (CCS)

V. Laboratory Control Sample (CCS)

VI. Duplicate Sample (CCS)

VII. Matrix Spike Sample (CCS)

VIII. Furnace Atomic Absorption QC (CCS)

IX. ICP Serial Dilution (CCS)

X. Sample Result Verification (CCS - 10%)

XI. Field Duplicates

XII. Overall Assessment of Data for a Case

I. HOLDING TIMES

A. Objective

The objective is to ascertain the validity of results based on the holding time of the sample from time of collection to time of analysis.

Note: The holding time is based on the date of collection, rather than verified time of sample receipt, and date of digestion/distillation. It is a technical evaluation rather than a contractual requirement.

B. Criteria

Technical requirements for sample holding times have only been established for water matrices. The following holding time and preservation requirements were established under 40 CFR 136 (Clean Water Act) and are found in Volume 49, Number 209 of the Federal Register, page 43260, issued on October 26, 1984.

METALS:	6 months; preserved to pH < 2
MERCURY:	28 days; preserved to pH < 2
CYANIDE:	14 days; preserved to pH > 12

C. Evaluation Procedure

Actual holding times are established by comparing the sampling date on the EPA Sample Traffic Report with the dates of analysis found in the laboratory raw data (digestion logs and instrument run logs). Examine the digestion and/or distillation logs to determine if samples were preserved at the proper pH.

Analyte Holding Time (Days) = Analysis Date - Sampling Date

D. Action

1. If 40 CFR 136 criteria for holding times and preservation are not met, qualify all results > Instrument Detection Limit (IDL) as estimated (J) and results < IDL as estimated (UJ).

2. If holding times are exceeded, the reviewer must use professional judgment to determine the reliability of the data and the effects of additional storage on the sample results. The expected bias would be low and the reviewer may determine that results < IDL are unusable (R).

3. Due to limited information concerning holding times for soil samples, it is left to the discretion of the data reviewer whether to apply water holding time criteria to soil samples. If the data are qualified when water holding time criteria are applied to soil samples, it must be clearly documented in the review.

II. CALIBRATION

A. Objective

Compliance requirements for satisfactory instrument calibration are established to ensure that the instrument is capable of producing acceptable quantitative data. Initial calibration demonstrates that the instrument is capable of acceptable performance at the beginning of the analysis run, and continuing calibration verification documents that the initial calibration is still valid.

B. Criteria

1. Initial Calibration

Instruments must be calibrated daily and each time the instrument is set up.

a. ICP Analysis

A blank and at least one standard must be used in establishing the analytical curve.

b. Atomic Absorption Analysis (AA)

1) A blank and at least three standards, one of which must be at the Contract Required Detection Limit (CRDL), must be used in establishing the analytical curve.

2) The correlation coefficient must be ≥0.995.

Note: The correlation coefficient of 0.995 is a technical criterion and not contractual.

c. Mercury Analysis

1) A blank and at least four standards must be used in establishing the analytical curve.

2) The correlation coefficient must be ≥0.995.

d. Cyanide Analysis

1) A blank and at least three standards must be used in establishing the analytical curve.

2) A midrange standard must be distilled.

3) A correlation coefficient ≥0.995 is required for photometric determination.

2. Initial and Continuing Calibration Verification (ICV and CCV)

a. Analysis results must fall within the control limits of 90 -110 percent Recovery (%R) of the true value for all analytes except mercury and cyanide.

b. Analysis results for mercury must fall within the control limits of 80-120%R.

c. Analysis results for cyanide must fall within the control limits of 85-115%R.

C. Evaluation Procedure

1. Verify that the instrument was calibrated daily and each time the instrument was set up using the correct number of standards and blank.

2. Verify that the correlation coefficient is ≥0.995

3. Check the distillation log and verify that the midrange CN standard was distilled.

4. Recalculate one or more of the ICV and CCV %R per type of analysis (ICP, GFAA, etc.) using the following equation and verify that the recalculated value agrees with the laboratory reported values on Form IIA. Due to possible rounding discrepancies, allow results to fall within 1% of the contract windows (e.g., 89-111%).

$$\%R = \frac{\text{Found}}{\text{True}} \times 100$$

Where,

Found = concentration (in ug/L) of each analyte <u>measured</u> in the analysis of the ICV or CCV solution

True = concentration (in ug/L) of each analyte in the ICV or CCV source

D. Action

1. If the minimum number of standards as defined in section B were not used for initial calibration, or if the instrument was not calibrated daily and each time the instrument was set up, qualify the data as unusable (R).

2. If the correlation coefficient is <0.995, qualify results > IDL as estimated (J), and results < IDL as estimated (UJ).

Note: For critical samples, further evaluation of the calibration curve may be warranted to determine if qualification is necessary.

3. If the midrange CN standard was not distilled, qualify all associated results as estimated (J).

4. If the ICV or CCV %R falls outside the acceptance windows, use professional judgment to qualify all associated data. If possible, indicate the bias in the review. The following guidelines are recommended:

   a. If the ICV or CCV %R falls outside the acceptance windows but within the ranges of 75-89% or 111-125% (CN, 70-84% or 116-130%; Hg, 65-79% or 121-135%), qualify results > IDL as estimated (J).

   b. If the ICV or CCV %R is within the range of 111-125% (CN, 116-130%; Hg, 121-135%), results < IDL are acceptable.

   c. If the ICV or CCV %R is 75-89% (CN, 70-84%; Hg, 65-79%), qualify results < IDL as estimated (UJ).

   d. If the ICV or CCV %R is <75%, (CN, <70%; Hg, <65%), qualify all positive results as unusable (R).

   e. If the ICV or CCV %R is >125%, (CN >130%; Hg >135%), qualify results > IDL as unusable (R); results < IDL are acceptable.

III. BLANKS

A. Objective

The assessment of blank analysis results is to determine the existence and magnitude of contamination problems. The criteria for evaluation of blanks applies to any blank associated with the samples. If problems with any blank exist, all data associated with the Case must be carefully evaluated to determine whether or not there is an inherent variability in the data for the Case, or if the problem is an isolated occurrence not affecting other data.

B. Criteria

No contaminants should be in the blank(s).

C. Evaluation Procedures

Review the results reported on the Blank Summary (Form III) as well as the raw data (ICP printouts, strip charts, printer tapes, bench sheets, etc.) for all blanks and verify that the results were accurately reported.

D. Action

Action in the case of unsuitable blank results depends on the circumstances and origin of the blank. Sample results > IDL but <5 times the amount in any blank should be qualified as (U).

Any blank with a negative result whose absolute value is > IDL must be carefully evaluated to determine its effect on the sample data.

Note: The blank analyses may not involve the same weights, volumes, or dilution factors as the associated samples. In particular, soil sample results reported on Form I will not be on the same basis (units, dilution) as the calibration blank data reported on Form III. The reviewer may find it easier to work from the raw data when applying 5X criteria to soil sample data/calibration blank data.

In instances where more than one blank is associated with a given sample, qualification should be based upon a comparison with the associated blank having the highest concentration of a contaminant. The results must not be corrected by subtracting any blank value.

IV. ICP INTERFERENCE CHECK SAMPLE (ICS)

A. Objective

The ICP Interference Check Sample verifies the contract laboratory's interelement and background correction factors.

B. Criteria

1. An ICS must be run at the beginning and end of each sample analysis run (or a minimum of twice per 8 hour working shift, whichever is more frequent).
2. Results for the ICS solution AB analysis must fall within the control limits of ± 20% of the true value.

C. Evaluation Procedure

1. Recalculate from the raw data (ICP printout) one or more of the recoveries using the following equation (%R) and verify that the recalculated value agrees with the laboratory reported values on Form IV.

$$\text{ICS \%R} = \frac{\text{Found Solution AB}}{\text{True Solution AB}} \times 100$$

Where,

Found Solution AB = concentration (in ug/L) of each analyte measured in the analysis of solution AB

True Solution AB = concentration (in ug/L) of each analyte in solution AB

2. Check ICS raw data for results with an absolute value > IDL for those analytes which are not present in the ICS solution.

D. Action

1. For samples with concentrations of Al, Ca, Fe, and Mg which are comparable to or greater than their respective levels in the Interference Check Sample:

   a. If the ICS recovery for an element is >120% and the sample results are < IDL, this data is acceptable for use.

   b. If the ICS recovery for an element is >120% and the sample results are > IDL, qualify the affected data as estimated (J).

   c. If the ICS recovery for an element falls between 50 and 79% and the sample results are > IDL, qualify the affected data as estimated (J).

   d. If sample results are < IDL, and the ICS recovery for that analyte falls within the range of 50-79%, the possibility of false negatives may exist. Qualify the data for these samples as estimated (UJ).

   e. If ICS recovery results for an element fall <50%, qualify the affected data as unusable (R).

   Note: If possible, indicate the bias for the estimated results in the review.

2. If results > IDL are observed for elements which are not present in the EPA provided ICS solution, the possibility of false positives exists. An evaluation of the associated sample data for the affected elements should be made. For samples with comparable or higher levels of interferents and with analyte concentrations that approximate those levels found in the ICS (false positives), qualify sample results > IDL as estimated (J).

3. If negative results are observed for elements that are not present in the EPA ICS solutions, and their absolute value is > IDL, the possibility of false negatives in the samples may exist. If the absolute value of the negative results is > IDL, an evaluation of the associated sample data should be made. For samples with comparable or higher levels of interferents, qualify results for the affected analytes < IDL as estimated (UJ).

4. In general, the sample data can be accepted if the concentrations of Al, Ca, Fe and Mg in the sample are found to be less than or equal to their respective concentrations in the ICS. If these elements are present at concentrations greater than the level in the ICS, or other elements are present in the sample at >10 mg/L, the reviewer should investigate the possibility of other interference effects by using Table 2 given on page D-22 of the 7/87 SOW. These analyte concentration equivalents presented in the Table should be

considered only as estimated values, since the exact value of any analytical system is instrument specific. Therefore, estimate the concentration produced by an interfering element. If the estimate is >2X CRDL and also greater than 10% of the reported concentration of the affected element, qualify the affected results as estimated (J).

V. LABORATORY CONTROL SAMPLE (LCS)

A. Objective

The laboratory control sample serves as a monitor of the overall performance of all steps in the analysis, including the sample preparation.

B. Criteria

1. All aqueous LCS results must fall within the control limits of 80-120%R, except Sb and Ag which have no control limits.

2. All solid LCS results must fall within the control limits established by the EPA. This information is available from EMSL/LV.

C. Evaluation Procedure

1. Review Form VII and verify that results fall within the control limits.

2. Check the raw data (ICP printout, strip charts, bench sheets) to verify the reported recoveries on Form VII. Recalculate one or more of the recoveries (%R) using the following equation:

$$\text{LCS \%R} = \frac{\text{LCS Found}}{\text{LCS True}} \times 100$$

Where,

LCS Found = concentration (in ug/L for aqueous; mg/kg for solid) of each analyte measured in the analysis of LCS solution

LCS True = concentration (in ug/L for aqueous; mg/kg for solid) of each analyte in the LCS source

D. Action

1. Aqueous LCS

    a. If the LCS recovery for any analyte falls within the range of 50 - 79% or >120%, qualify results > IDL as estimated (J).

    b. If results are < IDL and the LCS recovery is greater than 120%, the data are acceptable.

    c. If results are < IDL and the LCS recovery falls within the range of 50-79%, qualify the data for the affected analytes as estimated (UJ).

d. If LCS recovery results are <50%, qualify the data for these samples as unusable (R).

2. Solid LCS

a. If the solid LCS recovery for any analyte falls outside the EPA control limits, qualify all sample results > IDL as estimated (J).

b. If the LCS results are higher than the control limits and the sample results are < IDL, the data are acceptable.

c. If the LCS results are lower than the control limits, qualify all sample results < IDL as estimated (UJ).

VI. DUPLICATE SAMPLE ANALYSIS

A. Objective

Duplicate analyses are indicators of laboratory precision based on each sample matrix.

B. Criteria

1. Samples identified as field blanks cannot be used for duplicate sample analysis.

2. A control limit of ± 20% (35% for soil) for the Relative Percent Difference (RPD) shall be used for sample values >5X CRDL.

3. A control limit of ±CRDL (±2X CRDL for soil) shall be used for sample values <5X CRDL, including the case when only one of the duplicate sample values is <5X CRDL.

C. Evaluation Procedure

1. Review Form VI and verify that results fall within the control limits.

2. Check the raw data and recalculate one or more RPD using the following equation to verify that results have been correctly reported on Form VI.

$$RPD = \frac{|S-D|}{(S+D)/2} \times 100$$

Where,

S = First Sample Value (original)
D = Second Sample Value (duplicate)

3. Verify that the field blank was not used for duplicate analysis.

D. Action

1. If duplicate analysis results for a particular analyte fall outside the appropriate control windows, qualify the results for that analyte in all associated samples of the same matrix as estimated (J).

2. If the field blank was used for duplicate analysis, all other QC data must be carefully checked and professional judgment exercised when evaluating the data.

   Note: This information must be included on the IRDA form.

VII. MATRIX SPIKE SAMPLE ANALYSIS

A. Objective

The matrix spike sample analysis provides information about the effect of each sample matrix on the digestion and measurement methodology.

B. Criteria

1. Samples identified as field blanks cannot be used for spiked sample analysis.

2. Spike recovery (%R) must be within the limits of 75-125%. However, spike recovery limits do not apply when sample concentration exceeds the spike concentration by a factor of 4 or more.

C. Evaluation Procedure

1. Review Form V and verify that results fall within the specified limits.

2. Check raw data and recalculate one or more %R using the following equation to verify that results were correctly reported on Form V.

$$\%R = \frac{(SSR-SR)}{SA} \times 100$$

Where,

SSR = Spiked Sample Result
SR = Sample Result
SA = Spike Added

3. Verify that the field blank was not used for spike analysis.

D. Action

1. If the spike recovery is >125% and the reported sample results are < IDL, the data is acceptable for use.

2. If the spike recovery is >125% or <75% and the sample results are > IDL, qualify the data for these samples as estimated (J).

3. If the spike recovery falls within the range of 30-74% and the sample results are < IDL, qualify the data for these samples as estimated (UJ).

4. If spike recovery results fall <30% and the sample results are < IDL, qualify the data for these samples as unusable (R).

5. If the field blank was used for matrix spike analysis, all other QC data must be carefully checked and professional judgment exercised when evaluating the data.

   Note: This information must be included on the IRDA form.

Note: If the matrix spike recovery does not meet criteria (except in Ag), a post digestion spike is required for all methods except furnace, but this data is not used to qualify sample results. However, this information must be included in the IRDA report.

VIII. FURNACE ATOMIC ABSORPTION QC

A. Objective

Duplicate injections and furnace post digestion spikes establish the precision and accuracy of the individual analytical determinations.

B. Criteria

1. For sample concentrations > CRDL, duplicate injections must agree within ±20% Relative Standard Deviation (RSD), (or Coefficient of Variation (CV)), otherwise the sample must be rerun once (at least two additional injections).

2. Spike recovery must be ≥85% and ≤115%.

3. The Furnace Atomic Absorption Scheme must be followed as described in the 7/87 SOW, p. E-15.

C. Evaluation Procedure

1. Check raw data to verify that duplicate injections agree within ±20% RSD (or CV) for sample concentrations > CRDL.

2. Review Furnace AA raw data to verify that the Furnace Atomic Absorption Scheme has been followed.

D. Action

1. If duplicate injections are outside the ±20% RSD (or CV) limits and the sample has not been rerun once as required, qualify the data as estimated (J).

2. If the rerun sample results do not agree within ±20% RSD (or CV), qualify the data as estimated (J).

3. If the post digestion spike recovery is <40%, qualify results > IDL as estimated (J).

4. If the post digestion spike recovery is ≥10%, but <40%, qualify results < IDL as estimated (UJ).

5. If the post digestion spike recovery is <10%, qualify results < IDL as unusable (R).

6. If sample absorbance is <50% of the post digestion spike absorbance then:

   a. If the furnace post digestion spike recovery is not within 85-115%, qualify the sample results > IDL as estimated (J).

   b. If the furnace post digestion spike recovery is not within 85-115%, qualify the sample results < IDL as estimated (UJ).

7. If Method of Standard Additions (MSA) is required but has not been done, qualify the data as estimated (J).

8. If any of the samples run by MSA have not been spiked at the appropriate levels, qualify the data as estimated (J).

9. If the MSA correlation coefficient is <0.995, qualify the data as estimated (J).

IX. ICP SERIAL DILUTION

A. Objective

The serial dilution determines whether significant physical or chemical interferences exist due to sample matrix.

B. Criteria

If the analyte concentration is sufficiently high (concentration in the original sample is minimally a factor of 50 above the IDL), an analysis of a 5-fold dilution must agree within 10% Difference (%D) of the original results.

C. Evaluation Procedures

1. Check the raw data and recalculate the %D using the following equation to verify that the dilution analysis results agree with results reported on Form IX.

$$\%D = \frac{|I-S|}{I} \times 100$$

Where,

I = Initial Sample Result
S = Serial Dilution Result (Instrument Reading x 5)

2. Check the raw data for evidence of negative interference, i.e., results of the diluted sample are significantly higher than the original sample.

D. Action

1. When criteria are not met, qualify the associated data as estimated (J).

2. If evidence of negative interference is found, use professional judgment to qualify the data.

X. SAMPLE RESULT VERIFICATION

A. Objective

The objective is to ensure that the reported quantitation results are accurate.

B. Criteria

Analyte quantitation must be calculated according to the appropriate SOW.

C. Evaluation Procedures

The raw data should be examined to verify the correct calculation of sample results reported by the laboratory. Digestion and distillation logs, instrument printouts, strip charts, etc. should be compared to the reported sample results.

1. Examine the raw data for any anomalies (i.e., baseline shifts, negative absorbances, omissions, legibility, etc.).
2. Verify that there are no transcription or reduction errors (e.g., dilutions, percent solids, sample weights) on one or more samples.
3. Verify that results fall within the linear range of the ICP (Form XIII) and within the calibrated range for the non-ICP parameters.
4. Verify that sample results are >5X ICP IDL, if ICP analysis results are used for As, Tl, Se, or Pb.

Note: When the laboratory provides both ICP and furnace results for an analyte in a sample and the concentration is > ICP IDL, the results can assist in identifying quantitation problems.

D. Action

If there are any discrepancies found, the laboratory may be contacted by the designated representative to obtain additional information that could resolve any differences. If a discrepancy remains unresolved, the reviewer may determine qualification of the data is warranted.

XI. FIELD DUPLICATES

A. Objective

Field duplicate samples may be taken and analyzed as an indication of overall precision. These analyses measure both field and lab precision; therefore, the results may have more variability than lab duplicates which measure only lab performance. It is also expected that soil duplicate results will have a greater variance than water matrices due to difficulties associated with collecting identical field samples.

B. Criteria

There are no review criteria for field duplicate analyses comparability.

C. Evaluation Procedures

Samples which are field duplicates should be identified using EPA Sample Traffic Reports or sample field sheets. The reviewer should compare the results reported for each sample and calculate the Relative Percent Difference (RPD), if appropriate.

D. Action

Any evaluation of the field duplicates should be provided with the reviewer's comments.

XII. OVERALL ASSESSMENT OF DATA FOR A CASE

It is appropriate for the data reviewer to make professional judgments and express concerns and comments on the validity of the overall data for a Case. This is particularly appropriate when there are several QC criteria out of specification. The additive nature of QC factors out of specification is difficult to assess in an objective manner, but the reviewer has a responsibility to inform the user concerning data quality and data limitations in order to assist that user in avoiding inappropriate use of the data, while not precluding any consideration of the data at all. If qualifiers other than those used in this document are necessary to describe or qualify the data, it is necessary to thoroughly document/explain the additional qualifiers used. The data reviewer would be greatly assisted in this endeavor if the data quality objectives were provided. The cover form and supplementary documentation must be included with the review.

GLOSSARY A

Data Qualifier Definitions

For the purposes of this document the following code letters and associated definitions are provided.

U	- The material was analyzed for, but was not detected above the level of the associated value. The associated value is either the sample quantitation limit or the sample detection limit.
J	- The associated value is an estimated quantity.
R	- The data are unusable. (Note: Analyte may or may not be present.)
UJ	- The material was analyzed for, but was not detected. The associated value is an estimate and may be inaccurate or imprecise.

GLOSSARY B

Additional Terms

Associated Samples	Any sample related to a particular QC analysis. For example: - For ICV, all samples run under the same calibration curve. - For duplicate RPD, all SDG samples digested/distilled of the same matrix.
AA	Atomic Absorption
Calibration Curve	A plot of absorbance versus concentration of standards
Case	A finite, usually predetermined number of samples collected in a given time period for a particular site. A Case consists of one or more Sample Delivery Groups.
CCB	Continuing Calibration Blank - a deionized water sample run every ten samples designed to detect any carryover contamination.
CCS	Contract Compliance Screening - process in which SMO inspects analytical data for contractual compliance and provides EMSL/LV, laboratories, and the Regions with their findings.
CCV	Continuing Calibration Verification - a standard run every ten samples designed to test instrument performance.
CLP	Contract Laboratory Program
CRDL	Contract Required Detection Limit
CV	Coefficient of Variation
DPO	Deputy Project Officer
EMSL/LV	Environmental Monitoring System Laboratory/ Las Vegas (P.O. Box 15027, Las Vegas, Nevada 89114)
Field Blank	Field blanks are intended to identify contaminants that may have been introduced in the field. Examples are trip blanks, travel blanks, rinsate blanks, and decontamination blanks.

Field Duplicate	A duplicate sample generated in the field, not in the laboratory.
Holding Time	The time from sample collection to laboratory analysis.
ICB	Initial Calibration Blank - first blank standard run to confirm the calibration curve.
ICP	Inductively Coupled Plasma
ICS	Interference Check Sample
ICV	Initial Calibration Verification - first standard run to confirm the calibration curve.
Initial Calibration	The establishment of a calibration curve with the appropriate number of standards and concentration range. The calibration curve plots absorbance or emission versus concentration of standards.
IRDA	Inorganic Regional Data Assessment
LCS	Laboratory Control Sample - supplied by EPA
MS	Matrix Spike - introduction of a known concentration of analyte into a sample to provide information about the effect of the sample matrix on the digestion and measurement methodology.
MSA	Method of Standard Addition
Post digestion Spike	The addition of a known amount of standard after digestion. (Also identified as analytical spike, or spike, for furnace analyses.)
QAC	Quality Assurance Coordinator
RPD	Relative Percent Difference
RSCC	Regional Sample Control Center
RSD	Relative Standard Deviation
Serial Dilution	A sample run at a specific dilution to determine whether any significant chemical or physical interferences exist due to sample matrix effects. (ICP only)

SDG	Sample Delivery Group - defined by one of the following, whichever occurs first: - case of field samples - each twenty field samples in a Case - each 14-day calendar period during which field samples in a Case are received, beginning with receipt of the first sample in the SDG.
SMO	Sample Management Office
SOP	Standard Operating Procedure
SOW	Statement of Work

Region ______

INORGANIC REGIONAL DATA ASSESSMENT

CASE NO. ______________________

SITE ______________________

LABORATORY ______________________

NO. OF SAMPLES/
MATRIX ______________________

SDG#______________________

REVIEWER (IF NOT ESD) ______________

SOW#______________________

REVIEWER'S NAME ______________

DPO: ACTION ______ FYI __________

COMPLETION DATE ______________

DATA ASSESSMENT SUMMARY

		ICP	AA	Hg	CYANIDE
1.	HOLDING TIMES	______	______	______	______
2.	CALIBRATIONS	______	______	______	______
3.	BLANKS	______	______	______	______
4.	ICS	______			
5.	LCS	______	______		
6.	DUPLICATE ANALYSIS	______	______	______	______
7.	MATRIX SPIKE	______	______	______	______
8.	MSA		______		
9.	SERIAL DILUTION	______			
10.	SAMPLE VERIFICATION	______	______	______	______
11.	OTHER QC	______	______	______	______
12.	OVERALL ASSESSMENT	______	______	______	______

O = Data had no problems/or qualified due to minor problems.
M = Data qualified due to major problems.
Z = Data unacceptable.
X = Problems, but do not affect data.

ACTION ITEMS: ____

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AREAS OF CONCERN: ____

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NOTABLE PERFORMANCE: ____

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