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American Chemical Service, Inc. NPL Site

Pilot/Treatability Testing QAPP and Field Sampling Plans

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PILOT/TREATABILITY TESTING

Three pilot and treatability studies will be conducted as part of the Pre-Design Activities at the American Chemical Service (ACS) Site:

- An In-Situ Vapor Extraction (ISVE) pilot study on buried waste materials in the On-Site Area

- Treatability testing of Low Temperature Thermal Treatment (LTTT) on buried waste in the Off-site Containment Area (OFCA), including a bench-scale LTTT test and a pretreatment/material handling field test

- An ISVE/air sparging pilot study on contaminated soils in the On-site Containment Area (ONCA).

The objectives, conceptual study design and equipment, testing procedures, data evaluation methods, reporting, and schedule for the pilot/treatability tests are generally described in the following sections. Detailed pilot system design, well and monitoring point construction details, monitoring and sampling procedures, analytical methods, and quality assurance/quality control (QA/QC) procedures for the pilot/treatability testing are included in the first submittal of the Field Sampling Plan (FSP) and Quality Assurance Project Plan (QAPP) for the pilot/treatability studies, which have been submitted with this revision of Section 5 of the Pre-Design Work Plan.

5.1 ISVE PILOT STUDY ON BURIED WASTE IN THE ON-SITE AREA

5.1.1. Objectives

An ISVE pilot study will be conducted in the On-Site Area to evaluate the effectiveness of the technology for removing contaminants from the buried waste materials. The buried waste materials, as defined in the SOW, are materials or soil with total volatile organic compounds (VOCs) in excess of 10,000 ppm or with PCBs in excess of 10 ppm or lead in excess of 500 ppm (refer to the RI/FS documents for information on constituents and their concentration ranges). The buried waste materials also contain drums that may interfere with the ISVE system's performance. The objectives of the pilot study are to:

- Assess the effectiveness of ISVE on materials with high levels (i.e., greater than 10,000 ppm VOCs) of organic contaminants.

- Demonstrate whether ISVE has the potential to reduce the contaminant levels in the buried waste to the performance standards listed in Table 5-1, which were outlined in the Record of Decision (ROD), the Unilateral Administrative Order (UAO), and the Statement of Work for the Remedial Design and Remedial Action (SOW).

- Develop design parameters for full-scale implementation of ISVE in the On-Site Area.

With respect to the second objective of demonstrating that ISVE has the potential to meet the performance standards, it is important to recognize that this area of the site has a broad variety of contaminants with many being present at high concentrations. Since the study will only be conducted for a limited time frame, it is not realistic to expect that all contaminants will be reduced to the performance standards during the study period. In particular, some of the semi-volatile organics and PCBs are not readily removable via ISVE. The expectation for the study, therefore, is to demonstrate that significant reductions in the volatile organics occurred as a result of the ISVE and that quantifiable reductions in the semi-volatile and PCBs occurred as a result of volatilization and biodegradation. This information will then be used in conjunction with mathematical

modeling to demonstrate if ISVE has the potential to meet the performance standards and, if so, to estimate the time frame necessary to do so.

5.1.2. Pilot Study Design and Equipment

The ISVE pilot study which will be performed on buried waste materials in the On-site Area is designed to: (1) evaluate whether ISVE will be effective for treating the buried waste materials to the performance standards in Table 5-1, and (2) collect design information to be used for full-scale implementation of ISVE in the buried waste materials at the site.

Since the water table at the site is currently 2 to 3 feet below ground surface (bgs), dewatering will be required before the ISVE pilot study can begin. Specifically, dewatering is necessary for this aspect of project for the following reasons:

- The wastes are water saturated, and must be dewatered to permit air flow during ISVE pilot testing

- During full-scale operation, water that is collected from the ISVE systems will have to be treated, so it is important to minimize the volume of water (moisture) in the extracted air, and also to obtain data regarding the quantity and chemical characteristics of the water for treatment purposes.

Rather than delay the pilot study until completion of the design and installation of the full-scale dewatering system at the site, a small portion of the On-Site Area will be dewatered specifically to conduct the ISVE pilot test. A test cell constructed of either sheet pile or a soil-bentonite slurry will be installed to control the size of the area to be dewatered and to minimize the volume of water generated during dewatering for the pilot study. The groundwater extracted as part of the dewatering for the ISVE pilot study will be placed in portable tanks for transportation and disposal at an off-site licensed facility, or the water will be stored in the tanks until it can be treated in the full-scale Perimeter Groundwater Containment System (PGCS) treatment plant. The details of the dewatering system are included in Appendix A, Section A.3.3 of the Pilot/Treatability Testing QAPP and FSP.

5.1.2.1. Pilot Study Activities. The ISVE pilot study in the On-Site Area buried waste will include the following activities:

- Installation of a test cell and dewatering wells in the pilot test area and dewatering of the test cell
- Installation of two vapor extraction wells and four multi-level vapor monitoring probe nests within the dewatered area
- Construction of a pilot-scale ISVE system, including a blower, vapor treatment unit, and associated piping and instrumentation
- Pre-test soil/waste and vapor sampling
- Start-up, operation, and monitoring of the pilot-scale ISVE system
- Post-test soil/waste and vapor sampling
- Data evaluation.

The effectiveness of ISVE and the ability of the technology to treat the buried waste material to the performance standards will be evaluated by comparing contaminant concentrations in pre- and post-test soil/waste and soil gas samples, estimating the mass of contaminants removed from the test area, and using a mathematical model to predict cleanup times. Mass removal will also be calculated using contaminant concentrations in the extracted vapors, and a mass balance will be performed. In addition, the pilot study will determine whether biological activity in the subsurface is enhanced by increasing the oxygen supply in the subsurface during ISVE.

Design parameters that will be determined from the pilot test data will include: number of extraction wells, well spacing, vapor extraction pumping rate, and off-gas treatment requirements. However, it is important to emphasize that the presence of the test cell will significantly affect vapor flow paths and will therefore impact the design data generated from the pilot study (specifically radius of influence). Consequently, some of the full-scale ISVE system design criteria (such as well spacing) will need to be developed using mathematical models such as Hyperventilate or MODFLOW-based AIR3D and

experience from sites with similar lithology`. A more detailed discussion is included in Appendix A, Section A.3.12 of the Pilot/Treatability Testing QAPP and FSP.

5.1.2.2. Pilot Study Data Requirements. The following data will be obtained from the pilot-scale ISVE system:

- Concentrations of VOCs and SVOCs in extracted vapors as a function of time
- Pre- and post-test concentrations of VOCs and SVOCs in soils/waste materials treated by the ISVE pilot system
- Pre- and post-test microbial population counts and in-situ respiration rates
- Well head vacuum versus vapor extraction flow rate curves for shallow and deep extraction wells
- Soil/waste permeability to air flow (although this may not be an accurate measurement due to the presence of the test cell)
- Areas/radii of influence of the vapor extraction wells
- Physical characteristics of the buried wastes
- Concentrations of separator drain water (condensate) as a function of time
- Quantity of separator drain water (condensate) as a function of time.

5.1.2.3. Test Location. The ISVE pilot study in the On-Site Area will be located as shown in Figure 5-1. Based on the site history, RI data, geoprobe data collected in March and June of 1995, and the recent soil borings conducted at the site, the area selected is representative of the contamination and geology of the On-Site Waste Area.

5.1.2.4. Test Cell and Dewatering System Design. The test cell will be 30 feet wide by 30 feet long and will be constructed of interlocking steel sheet piles with sealable joints or a soil-bentonite slurry wall. The test cell will be keyed into the silty clay confining layer beneath the upper aquifer (approximately 20 to 25 feet bgs) to minimize flow of

groundwater into the test cell during dewatering and ISVE testing. A dewatering system will be installed to dewater the area surrounded by the test cell down to the top of the clay. The pilot test cell and dewatering system design details are being finalized concurrent with the preparation of this document and they will be included as part of the 100 percent design submittal for the full-scale barrier wall systems or they will be submitted to USEPA in a letter prior initiation of the tests. The test cell will be dewatered to a depth of at least 20 feet prior to installing the vapor extraction wells and vapor monitoring probes. The dewatering system details are described in Appendix A, Section A. 3.2 of the Pilot/Treatability Testing QAPP and FSP. If ISVE is deemed appropriate as part of the final remedy, the dewatering wells inside the test cell will be incorporated into the full-scale system.

5.1.2.5. Vapor Extraction Wells and Vapor Monitoring Probes. After the test cell has been dewatered, two vapor extraction wells and four multi-level vapor monitoring probe nests will be installed. The layout of the vapor extraction wells and vapor monitoring points is shown in Figure 5-1. The vapor monitoring probes will be installed at four locations. Each location will contain vapor monitoring points at three different depths. The vapor monitoring points will be placed so that they extend radially away from the extraction wells in three different directions to obtain data regarding heterogeneity in the test cell.

The vapor extraction wells will be 4 inches in diameter. One will be screened at a shallow depth interval (approximately 5 to 10 feet bgs), and the other will be screened at a deeper depth interval (approximately 10 to 20 feet bgs). The exact depth intervals for the vapor extraction wells will be determined in the field. Each of the four vapor probe nests will contain three vapor monitoring points at different depths. Each vapor monitoring probe will consist of a slotted 6-inch long, 1-inch diameter screen connected to 1/4-inch tubing. Figures showing the details of the vapor extraction wells and monitoring probes are included in Appendix A of the Pilot/Treatability Testing QAPP and FSP.

If ISVE is deemed appropriate, the pilot wells and probes will be incorporated into the full-scale system.

5.1.2.6. ISVE Equipment. The pilot-scale ISVE system will consist of a blower, a condensate collection system, a vapor treatment unit, a flow meter located at the vapor extraction well heads, a vacuum gauge at the vapor extraction well heads, vapor sampling ports at the vapor extraction well heads, on the combined discharge line (manifold), and at the outlet of the blower, and associated piping, valves, and fittings. The blower will be an intrinsically safe, explosion-proof regenerative blower. The vapor treatment unit will consist of a catalytic oxidizer capable of treating the extracted vapors to applicable air discharge limits limits (i.e., IDEM air quality standards and the 1×10^{-4} to 1×10^{-6} cumulative cancer risk range for nearby residences and site workers as stated on page 30 of the ROD). The need for and design details of additional emissions control such as scrubbers will be finalized prior to mobilization based upon input from system suppliers (regarding removal efficiencies) and based upon review of proposed approach by the IDEM Office of Air Management. The equipment will be leased for the pilot study, and therefore will not likely be incorporated into the full-scale system if ISVE is deemed appropriate for the site. More details regarding the equipment are included in Appendix A of the Pilot/Treatability Testing QAPP and FSP.

5.1.3. Test Procedures

5.1.3.1. Pre-Test Soil/Waste Sampling. Two soil/waste samples will be collected from each boring drilled to install the ISVE wells and monitoring probes. The locations are shown in Figure 5-1 and the sampling depth intervals are discussed in Appendix A, Section A.3.3 of the Pilot/Treatability Testing QAPP and FSP. The samples will be sent to an off-site laboratory and analyzed for moisture content, grain size distribution, total organic carbon (TOC), microbial population count, VOCs, SVOCs, pesticides/PCBs, metals, total kjeldahl nitrogen (TKN), and ortho-phosphate. Specific analytical methods and sampling and laboratory protocols are included in the QAPP. These analytical results will be compared to the results of post-treatment soil/waste analyses that will be conducted after the ISVE pilot testing is complete.

5.1.3.2. Pre-Test Vapor Sampling. The pressure in each vapor probe, the air temperature at the extraction well, and the ambient air pressure will be measured and recorded prior to conducting the ISVE testing. Soil gas samples will also be collected from each extraction well and each vapor monitoring probe prior to beginning operation of the ISVE system. The samples will be analyzed in the field for carbon dioxide (CO_2),

oxygen (O_2), and total volatile hydrocarbons (using a FID). The field measurements will be used to assess the initial contaminant concentrations in the vapor phase and to evaluate biological activity prior to conducting the ISVE test. The initial CO_2 and O_2 concentrations in the soil gas will be used as a baseline for comparison to in-situ respiration rates at the conclusion of the ISVE test. An explosimeter will also be used to measure for explosive conditions at each extraction well. Details regarding the vapor sampling are included in Appendix A of the Pilot/Treatability Testing QAPP and FSP.

5.1.3.3. ISVE System Start-up. The ISVE equipment will be started and visually inspected prior to conducting the ISVE testing to confirm that the system is operating properly. Specific items to be checked are included in Appendix A, Section A.3.6 of the Pilot/Treatability Testing QAPP and FSP. System shutdown contingencies are discussed in Appendix A, Section A.3.3 of the Pilot/Treatability Testing QAPP and FSP

5.1.3.4. Initial Vapor Sampling. Vapor sampling will be conducted at each vapor extraction well head and at each vapor monitoring probe at the beginning of the short term variable flow rate tests to determine the initial composition of the extracted vapors. The initial vapor samples will be collected at each extraction well head while the ISVE system is operating and after at approximately one bore volume has been purged. The vapor samples will be sent to an off-site laboratory for analysis of VOCs, SVOCs, methane, and ethane. Prior to taking the samples, an explosimeter will be used to monitor for explosive conditions in each well and vapor probe.

A weather station will also be installed at the site to provide data regarding temperature, barometric pressure, humidity, precipitation, and wind speed and direction.

5.1.3.5. Short Term Variable Flow Rate Tests. Short term variable flow rate tests will be conducted initially to determine the vacuum versus flow rate operating curve for each extraction well. Five flow rates will be tested in ascending order for each extraction well in the short-term tests (5 flow rates for each well, 10 tests total). Vacuum measurements will be made frequently at the well heads and vapor monitoring probes until steady state is reached for each flow rate. The duration of each test will depend on the length of time required to achieve steady state vacuum measurements at the vapor monitoring probes.

The short term tests are expected to take less than one week to complete. More specific test procedures are included in Appendix A, Section A.3.8 of the Pilot/Treatability Testing QAPP and FSP.

As stated previously, the presence of the test cell will impact the vapor flow paths and the time frame necessary for the vapor probes to reach steady state. The main purpose of these test, however, is to determine the appropriate flow rate to use in the long term test. The information needed to select a flow rate is essentially a curve showing vacuum at the well head versus flow rate. As the flow rate increases, the vacuum will also increase up to certain value. From that point on, increasing the flow rate will not increase the vacuum. The lowest flow rate achieving the maximum vacuum will be selected for use in the long term test. The optimum flow rate will be determined on each vapor extraction well separately. Since both wells will be operated during the long term test and there will only be one blower, it will be necessary to throttle the valves on each discharge line to achieve the desired flow rate in the respective well.

The data collected during the short term test will also be used to estimate the air permeability of the soil/waste in the test area. A more detailed discussion of the test, air permeability calculation, and limitations on the use of air permeability determined from the test is included in Appendix A, Section A.3.8 of the Pilot/Treatability Testing QAPP and FSP.

5.1.3.6. Long Term Constant Flow Rate Test. At the conclusion of the short term tests, a long term constant rate test will be conducted. Vapors will be extracted from both extraction wells simultaneously for the long term test. The vapor extraction flow rate for the long term test will be determined in the field based upon the results of the short term tests. The long term test will be conducted for a minimum of two months, and it is anticipated to run for approximately six months, however, the test duration will be no longer than the time it takes to implement treatment for source materials in the off-site area. The specific duration of the test will be approved by USEPA and IDEM based upon the results and the schedule of the remaining RD/RA activities at the site.

Specific test procedures are described in Appendix A, Section A.3.9 of the Pilot/Treatability Testing QAPP and FSP. In general, however, the test will essentially involve operating the blower and taking measurements or samples from the vapor monitoring probes and the extraction well manifold. The wells will not be sampled

individually during the long term test since it is desireable to have data on the combined vapor stream for design purposes. The presence of the test cell will again affect the vapor flow paths as well as the steady state vacuum at each probe. Consequently, a calculation of the radius of influence using this data may not produce a result that would be representative of the area once it is dewatered without a test cell. At a minimum, the data will be used to verify that the area of influence extends out to the vapor probes. Vacuum measurements collected during transient conditions may also be used to evaluate the level of heterogeneity and possibly to estimate the air permeability and radius of influence. The primary information that will be obtained from the long term study, however, is the contaminant loading data for the extracted vapors and the quantity and quality of the condensate. Given that offgas treatment will be a very costly component of a full-scale ISVE system, it is important to obtain sufficient data regarding these parameters.

5.1.3.7. Sampling and Monitoring. Field monitoring and sampling for both on-site and off-site analyses will be conducted during the long-term test. The specific measurements, analytes, sampling locations, and sampling frequencies are included in Appendix A, Section A.3.9 of the Pilot/Treatability Testing QAPP and FSP.

5.1.3.8. Post-Test Vapor Sampling. Post-test vapor sampling will be conducted after the ISVE system is shut down. Vapor samples will be collected from each extraction well and vapor monitoring probe and analyzed in the field for CO_2, O_2, and total hydrocarbons. The post-test vapor sampling will be conducted immediately after ISVE system shut down, and at 0.5, 1, 2, 4, 8, 12, 24, and 48 hours after shut down. These measurements will be compared to the pre-test values to determine whether biological activity was enhanced due to the ISVE operation. This measurement frequency may be adjusted in the field based upon the observed respiration rates. Within 48 hours after shutdown, vapor samples will also be collected from each well head and each vapor monitoring probe for analysis of VOCs, SVOCs, methane, and ethane at an off-site laboratory. Specific analytical methods and sampling and laboratory protocols are included in the Pilot/Treatability Testing QAPP.

5.1.3.9. Post-Test Soil/Waste Sampling. Post-test soil/waste sampling will be conducted after the vapor sampling is complete. Soil/waste samples will be collected from borings installed within two feet of each of the borings sampled for the pre-test soil/waste sampling. The details are discussed in Appendix A, Section A. 3.11 of the

Pilot/Treatability Testing QAPP and FSP. The post-test soil/waste samples will be sent to an off-site laboratory and analyzed for moisture content, TOC, TKN, microbial population count, VOCs, SVOCs, pesticides/PCBs, metals, ortho-phosphate, and grain size distribution. The specific analytical methods and sampling and laboratory protocols are included in Appendix A of the Pilot/Treatability Testing QAPP. The post-test soil/waste sampling results will be compared to the pre-test soil/waste sampling results to evaluate the effectiveness of ISVE in the pilot test area.

5.1.4. Data Evaluation

The data generated from the pilot study in the On-Site Area will be used to: (1) evaluate the effectiveness of ISVE at removing contaminants from the buried waste materials in the On-Site Area and to assess the potential to meet the performance standards, (2) evaluate the effectiveness of ISVE for enhancing naturally-occurring biodegradation of the contaminants, and (3) determine design parameters for full-scale implementation of ISVE in the On-Site Area. The presence of lateral and vertical heterogeneities in the buried waste material also will be identified based on the vacuum measurements in the vapor monitoring probes. A discussion of assessing heterogeneity as well as the overall data evaluation is provided in Appendix A, Section A.3.12 of the Pilot/Treatabilty Testing QAPP and FSP.

5.1.4.1. Contaminant Removal and Performance Standards. The mass removal of contaminants will be calculated and used to perform a mass balance. The effectiveness of ISVE for removing contaminants and the potential for the technology to meet the performance standards will be evaluated based upon:

- The mass of contaminants removed during the ISVE pilot study, calculated based upon extracted vapor concentrations and vapor extraction flow rates

- Comparison between pre-test and post-test soil/waste and soil gas sampling results

- Predicted cleanup time and ability to meet performance standards for buried wastes using mathematical modeling (such as Hyperventilate or another model as approved by U.S. EPA) and concentration reductions observed during the pilot testing.

Since the pilot study will be conducted for a limited time, it is unlikely that soil contamination will be reduced to the performance standards. Consequently, it will be necessary to utilize a mathematical model such as Hyperventilate in conjunction with the field data to demonstrate if ISVE has the potential to meet the performance standards. If the study results do not demonstrate that ISVE has the potential to meet the standards, USEPA may require the Respondents to abandon the technology in favor of low temperature thermal treatment (depending on the results of the thermal treatment treatability study). As stated in the SOW (page 11), ISVE may still be an appropriate technology to use in conjunction with low temperature thermal treatment. Additional discussion of the data evaluation is included in Appendix A, Section A.3.12 of the Pilot/Treatability Testing QAPP and FSP.

5.1.4.2. Bioremediation Enhancement. Although bioventing is not specifically authorized in the SOW, it will occur naturally at the site and is known to be accelerated with ISVE. Therefore, the effectiveness of ISVE for enhancing naturally-occurring biodegradation of the buried wastes will be qualitatively evaluated using the pre- and post-test vapor sampling results. If oxygen is the limiting factor in biodegradation, the pre-test soil vapor samples in the contaminated area should exhibit elevated CO_2 concentrations (in the range of 5% to 15%), depressed O_2 concentrations (<5%), and elevated hydrocarbon concentrations. Background soil vapor concentrations should be similar to atmospheric concentrations for CO_2 and O_2, with hydrocarbon concentrations generally less than 100 ppm. During the ISVE test, the O_2 concentration in the soil vapor in the test area should increase to approximately 20%. Using the O_2 data from the 48-hour post-vapor sampling test, an oxygen utilization rate will be calculated. If the utilization rate is within the range of 0.05% to 1% O_2/hour, this will be sufficient evidence that some microbial activity is occurring and that the addition of O_2 in this area will enhance biodegradation.

Ideally, the utilization rate from the post-test vapor sampling should be compared to a utilization rate from a background vapor monitoring probe. Unfortunately, the probe would need to be very shallow (and therefore would reflect atmospheric conditions) and would need to be located off of the site. It is not possible to locate a vapor monitoring probe that will not be affected by the ISVE system within the dewatered test cell. Consequently, a background vapor probe for this purpose is not believed to be of significant value.

In addition to the above, microbial plate counts in pre-test and post-test soil samples will be compared to see if the microbial population has noticeably increased. Analytical data for specific constituents required for biological growth will also be evaluated to determine if other factors could be limiting biodegradation. The analytical data regarding methane will provide an indication of anaerobic activity and the presence of ethane may indicate degradation of chlorinated hydrocarbons.

Specific test procedures are included in Appendix A and the SOPs in the Pilot/Treatability Testing QAPP and FSP.

5.1.4.3. Design Parameters. The design parameters for full-scale ISVE will be determined as follows:

- An ISVE blower operating curve will be generated for each extraction well by plotting vapor extraction flow rate per foot of well screen versus vacuum at the extraction well head. The operating curve will be used as a guide for selecting a blower for the full-scale system. It should be noted that the vacuum measurements generated during the pilot study will be greater than those experienced in a system without the test cell. Consequently, the operating curve from the pilot study may suggest a larger blower than is actually necessary. This will be taken into consideration in the final sizing.

- Extracted vapor concentrations at the vapor extraction wells, volumes of extracted condensate, and condensate composition will be used to design the offgas treatment system.

- The effective treatment radii of the vapor extraction wells and the air permeability of the soil/waste will be calculated based upon the vacuum measurements at the vapor monitoring probes and the vacuum and flow rate measurements at the vapor extraction wells. (A discussion of how the data will be used to evaluate air permeability is included in Appendix A and the SOP in the Pilot/Treatability Testing QAPP and FSP.) However, it is important to emphasize that the presence of the test cell walls will affect vapor flow paths which will impact the estimate of the radius of influence generated from the pilot study. Therefore, radius of influence and well spacing for the final full-

scale ISVE system will be established using a combination of modeling, typical radius of influence for sites with similar lithology, and professional judgement based on the pilot study results.

5.2 LTTT AND PRETREATMENT/MATERIAL HANDLING STUDY IN THE OFCA

5.2.1. Objectives

Since the ROD requires implementation of low temperature thermal to treat the waste in the Off-Site Containment Area, the data generated from the pilot/treatability study will be used to determine design parameters for full scale implementation of the technology. Specifically, pilot/treatability testing will be conducted to:

- Evaluate whether LTTT can meet the performance standards listed in Table 5-1
- Develop LTTT operating parameters needed to treat soils and buried wastes to the performance standards.

On-site LTTT is required for full-scale treatment of dewatered/excavated buried waste materials and source areas. Treatment residuals from LTTT meeting performance standards will be redeposited on site. Air emissions from the LTTT must be controlled to meet the IDEM air quality standards and the 1×10^{-4} to 1×10^{-6} cumulative cancer risk range for nearby residence and site workers as stated on page 30 of the ROD. Air monitoring will be conducted while the on-site pilot study activities are taking place. The specific monitoring methods, frequency, duration, and compounds to be analyzed are discussed in Section 5.2.2.4. and in Appendix C, Section C.5 of the Pilot/Treatability Testing QAPP and FSP.

Excavation and dewatering of buried waste is required prior to using LTTT at the ACS Site. Soil borings from previous site investigations indicate that the water table is between 4 and 5 feet below ground surface and that a large quantity and wide variety of contaminated debris are present at the site, particularly in the saturated zone. These materials have the potential for significant impact on the remedial design and implementation. Consequently, a pretreatment/material handling pilot study is being conducted in addition to the required LTTT treatability study to evaluate potential material handling issues. The material handling issues are associated with the debris and wet nature of the material to be screened after excavation.

5.2.2. Pretreatment/Materials Handling Study

Failure to separate the debris from the contaminated soil and waste could result in the following impacts on the LTTT unit:

- Jamming of mechanical equipment, resulting in excessive downtime
- Inadequate decontamination of organic debris
- Excessive offgas and water treatment requirements
- Potential for exceeding air emissions limitations for the LTTT unit.

The pretreatment/materials handling study will address the following technical issues to develop a workable design for excavation of buried waste and material handling prior to LTTT:

- Type and quantity of debris at the site

- Ability of conventional materials handling equipment to separate untreatable debris from materials that can be thermally treated

- Potential for fugitive VOC emissions during materials handling operations

The following text presents the general test plan. Specific details are included in Appendix C of the Pilot/Treatability Testing QAPP and FSP.

5.2.2.1. Test Location. The pretreatment/materials handling study will be conducted in the OFCA as shown in Figure 5-2. Five test pits will be excavated, and approximately 100 to 400 tons of material will be removed from each test pit. The material from each test pit will be stockpiled on a plastic liner adjacent to the pit and allowed to dewater by gravity draining. The ground surface under the liner will be sloped and bermed so that the water from the stockpile will drain back into the excavated pit. Dewatering of the wastes is essential for the following reasons:

- The wastes contain large amounts of debris and other materials (paper, wood, cloth, scrap metal, etc.). These materials are currently below the water table and must be dewatered prior to LTTT. Because the effectiveness and cost of

LTTT are very sensitive to water content, it is important to understand what the water content of the wastes will be after dewatering. If the water content is high, the volume of condensate, which may have to be transported off-site for treatment, will also be high. This could impact the type of LTTT unit that would be used at the site or the method of condensate or offgas treatment.

- It is also important to have the material handling test be conducted in a dewatered area because fugitive VOC and airborne particulate emissions, which must be controlled or collected and treated, will be highest after the wastes are dewatered.

Once the material has drained and dried sufficiently, it will be moved to a central area for screening.

5.2.2.2. Equipment. Full-scale material handling equipment will be required to determine the ability to separate the debris from the soil. The equipment will be specified to accomplish the following separations:

- Coarse screening (6-inch grizzly)
- Medium screening (2-inch vibrating power screen)
- Fine screening (3/4-inch vibrating power screen) (optional).

A high lift front end loader will be used to move material onto the grizzly.

5.2.2.3. Test Procedures. The classified waste streams generated by the operation will be collected and weighed to determine the quantity of soil and debris from each test pit. The material passing the screening equipment will be visually examined to determine its debris content. After sampling, all material will be segregated and replaced into the excavations. Debris will be evaluated as a characteristically hazardous waste under the debris rule in 40 CFR Part 268.45 (treatment standards for hazardous debris). This will include sampling the matrix the debris is found in and testing for the characteristics of hazardous waste: ignitability, corrosivity, reactivity, and toxicity (40 CFR 261.21, 261.22, 261.23, and 261.24).

5.2.2.4. Air Monitoring. Fugitive emissions, particularly VOCs, are likely to be encountered during the study. To minimize the potential for odor problems off site, no more than three pits will be open at any one time. Also, plastic sheeting will be available to cover stockpiles to minimize emissions if off-site odors become a problem. In addition to these measures, daily ambient air samples will be collected upwind and downwind of the material handling activities. An air sample will also be collected prior to the start of these activities to serve as an indication of background conditions. The specific details of air monitoring program are included in Appendix C, Section C.5 of the Pilot/Treatability Testing QAPP and FSP.

5.2.3. LTTT Treatability Study

The LTTT treatability study is designed to: (1) evaluate whether LTTT will meet the cleanup standards and (2) determine LTTT operating parameters needed to treat soils and buried wastes to the performance standards.

The data generated by the LTTT treatability study will specifically be used to achieve the following objectives:

- Determine the VOC reductions during feed preparation operations
- Identify the concentrations of organic contaminants in the "prepared" treatability test samples
- Prepare two composite soil samples (a "typical" and a "worst-case" condition) for thermal desorption testing based on analyses of the "prepared" treatability test samples
- Determine the operating parameters such as temperature and residence time required to treat the soil and waste to the performance standards
- Conduct a total metals balance in the feed and treated soils
- Evaluate the effect of thermal treatment on the leachability of metals in the soil

- Characterize the offgas from the thermally treated soil
- Determine soil geotechnical properties to provide data useful in assessing material handling requirements
- Evaluate the potential for dioxin/furan formation during thermal treatment.
- Conduct a carbon and chloride balance on the thermally treated soils
- Evaluate (if necessary) the effect of treatment conditions on small pieces of debris (wood, rubber, paper, plastic, etc.).

5.2.3.1. Test Procedures. LTTT treatability testing will consist of the following steps:

- Sample preparation
- Laboratory-scale tray tests
- Bench-scale Rotary Thermal Apparatus (RTA) tests.

Specific details of the test procedures are included in Appnedix D of the Pilot/Treatability Testing QAPP and FSP.

5.2.3.1.1. Sample Preparation. Selection of representative soil/waste feed samples for testing is critical for generating results that can be scaled up to full-scale design. Five primary characteristics of the waste feed material impact the effectiveness of a thermal process:

- Clay and silt content and types
- Contaminant types and concentrations
- Organic and heat content
- Moisture content
- Debris content and types of debris.

Five samples will be collected from the materials processed during the pretreatment/ material handling study and submitted to an off-site laboratory for analysis of contaminant concentrations. Two samples will be prepared for thermal treatability testing to represent the following waste matrices: (1) typical soil/waste and (2) worst case

soil/waste. If significant amounts of small, shreddable debris are processed during the material handling study, then a third sample representing this material may be prepared. The typical and worst case samples will either be selected from the five samples or prepared as composites by blending aliquots from the five soil samples. The shredded debris sample (if necessary) will be a composite of various types of small debris encountered during the pretreatment/material handling study.

After the test samples have been homogenized, they will be sampled and analyzed in an off-site laboratory for VOCs, SVOCs, pesticides/PCBs, total and TCLP metals, dioxins and furans, miscellaneous chemical properties (total chlorine/chloride, carbon, hydrogen, sulfur, nitrogen, phosphorus, sodium, potassium, moisture content, ash content, pH, total organic carbon, and heating value), and geochemical properties (Proctor density particle size distribution, soil classification, and Atterberg limits). Specific analytical methods and sampling and laboratory procedures are included in the Pilot/Treatability Testing QAPP and FSP.

5.2.3.1.2. Laboratory-Scale Tray Tests. The laboratory testing will consist of static tray testing in a muffle furnace. Results of the tray tests will be used to select the treatment conditions to be used for the RTA testing. A four by one test matrix will be conducted using four different temperatures at one specific residence time on each soil/waste sample (typical and worst case). The range of soil/waste treatment temperatures for the testing will be selected based on experience with similar contaminants and based on the results of a previous study conducted for the ACS Site, which are documented in a report entitled: Bench-scale Treatability Study - SoilTech Anaerobic Thermal Process, American Chemical Service NPL Site (Montgomery Watson, April 1993). Based on the previous studies, the anticipated treatment temperature will be between 800°F and 1000°F at a residence time of approximately 20 minutes. Treated samples will be sent to an off-site laboratory and analyzed for VOCs, SVOCs, pesticides/PCBs, and dioxins and furans. (Specific analytical methods and sampling and laboratory procedures are included in the Pilot/Treatability Testing QAPP and FSP.) The shredded debris matrix will be processed at one set of tray test conditions, and qualitative observations will be made.

5.2.3.1.3. RTA Bench Testing. The RTA bench tests will be conducted in a device capable of processing one kilogram of material through a time/temperature profile closely resembling the conditions existing in a full-scale LTTT unit. RTA offgas will be treated

using a heated filter and a set of water impingers. The impinger offgas will be split into three separate streams to measure SVOCs, VOCs, carbon monoxide, and total hydrocarbons emanating from the soil during thermal treatment. Grab samples of the carbon offgas will also be collected at selected temperatures and analyzed for methane, ethane, propane, hydrogen, and other light gases. The impinger water and XAD resin will be analyzed for pesticides/PCBs, VOCs, SVOCs, and dioxins and furans. Specific analytical methods and sampling and laboratory procedures are included in the Pilot/Treatability Testing QAPP and FSP.

One RTA test will be conducted for each soil/waste matrix using the temperature and residence time values selected in the laboratory tray tests. One RTA test also will be conducted on the shredded debris matrix (if necessary). Treated samples will be analyzed for VOCs, SVOCs, pesticides/PCBs, total and TCLP metals, dioxins and furans, and miscellaneous chemical properties.

5.2.3.2. Data Evaluation. Results of the analyses of treated soil samples will be compared to the performance standards for soils and buried wastes to determine whether LTTT can meet the performance standards and to determine the operating conditions required for full-scale treatment. It is important to mention, however, that the results of the LTTT tests may not be conclusive as to whether the performance standards can be met with this technology. The laboratory testing will be conducted under relatively ideal conditions compared to those associated with a full-scale remediation. Consequently, if the laboratory LTTT tests demonstrate that the performance standards cannot be met, this will be taken as an indication that the standards cannot be met with a full-scale system either. On the other hand, if the laboratory LTTT tests show that the standards can be achieved, this will not necessarily prove that the standards can be met with a full-scale system. Other factors such as the degree of difficulty associated with achieving the standards in the laboratory, how the test sample initial concentrations compare to what might be encountered in the field, and the results of the pretreatment material handling study will all need to be considered in assessing the potential for a full-scale system to meet the performance standards. A specific discussion of data evaluation is included in Appendices C and D of the Pilot/Treatability Testing QAPP and FSP.

5.3 ISVE/AIR SPARGING PILOT STUDY IN CONTAMINATED SOILS IN THE ONCA

5.3.1. Objectives

The SOW requires treatability testing to determine whether ISVE can achieve the performance standards (listed in Table 5-1) in contaminated soils at the site. Since the water table is 2 to 3 feet below ground surface in this area, and contamination is present in the saturated soils, the area would need to be dewatered to perform the ISVE tests. Without dewatering the area, extraction wells cannot induce air flow through the soil matrix. In addition, during full-scale operation, water that is collected in the ISVE system will be contaminated and will need to be treated. Consequently, it is important to reduce the volume of moisture in the soils.

Dewatering the area would require at least six months and would generate a significant quantity of water to be treated. A sheet pile or soil-bentonite slurry test cell similar to the one proposed for the On-Site Area could be installed to reduce the time period and volume of water to dewater, but it would also impact the usefulness of the data for establishing full-scale system design parameters. As an alternative to dewatering, air sparging will be used to volatilize contaminants from the saturated soils and into the vadose zone where they will be captured by the ISVE system. If the results of the air sparging pilot study show that air sparging is not viable, the Respondents will submit documentation of this finding to USEPA and they may request an alternate course of action (such as dewatering and ISVE).

If the performance standards can be met by ISVE/air sparging, design of a full-scale system is required. Therefore, the objectives of the pilot study are to:

- Demonstrate whether ISVE/air sparging has the potential to reduce contaminant levels in the soils to the performance standards listed in Table 5-1

- Develop design parameters for full-scale implementation of ISVE/air sparging in the ONCA.

5.3.2. Pilot Study Design and Equipment

The ISVE/air sparging pilot study is designed to obtain information regarding ISVE, air sparging, and ISVE in combination with air sparging. To accomplish this, a short term test with just the ISVE system operating in the unsaturated zone will be conducted, followed by a short term test with just the air sparging system operating in the saturated zone, and then a test with both the ISVE and air sparging systems operating together. The short term tests provide the opportunity to optimize each of the individual systems prior to combining them (which increases the number of variables).

Since the unsaturated zone in this portion of the site is only 2 to 3 feet deep, horizontal vapor extraction wells (i.e., trenches) will be used instead of the conventional vertical wells. To minimize short circuiting from the surface (which is a concern since the wells will be so shallow), a plastic sheet will placed on the ground surface over the entire test area. The main purpose of the ISVE system for this study will be to induce air flow in the unsaturated zone to enhance removal of contaminants and to collect the potentially contaminated air that will be emanating from the saturated zone when the air sparging system is in operation.

The air sparging system will consist of a blower and two air injection wells. The injection wells will be vertical wells extending down almost to the clay layer. Air will be pumped into the injection wells and released through screens located at the well bottoms. As the air travels upward through the groundwater, contaminants will transfer from the water phase to the air phase and thus be stripped from the saturated zone. When the air reaches the unsaturated zone, it will be recovered by the ISVE system. This technology is well suited for sites with primarily VOC contamination and with relatively permeable saturated soils which, based on the data presented in the RI Report, is the case for the ONCA.

The study is designed to obtain the necessary data to determine (1) if ISVE/air sparging will be effective in the soils at the site, (2) if the technology can reduce contaminant levels to the performance standards, (3) if there are constituents in the groundwater (such as iron, calcium and manganese) at levels that could negatively impact the performance of an air sparging system, and (4) develop design parameters for a full-scale system.

5.3.2.1. Pilot Study Activities. The ISVE/air sparging pilot study in the contaminated soils in the ONCA will include the following activities:

- Installation of three vapor extraction trenches, two air injection wells, four groundwater monitoring wells/vapor monitoring probes, and three piezometers in the test area
- Construction of a pilot-scale ISVE/sparging system, including two blowers, a vapor treatment unit, and associated piping and instrumentation
- Pre-test soil, vapor, and groundwater sampling
- Operation and monitoring of the ISVE system alone
- Operation and monitoring of the air sparging system alone
- Simultaneous ISVE and air sparging system operation
- Post-test in-situ respiration testing
- Post-test soil, vapor, and groundwater sampling
- Data evaluation.

The effectiveness of ISVE/air sparging will be evaluated by comparing VOC and SVOC concentrations in pre-and post-test soil, soil vapor, and groundwater samples and estimating the mass of contaminants removed from the test area. The contaminant concentrations in the extracted vapors and the total volume of air extracted will be used in a mass balance to calculate the total mass of contaminants removed by the ISVE/air sparging process. In addition, the pilot study will determine whether biological activity in the subsurface is enhanced by increasing the oxygen supply in the subsurface during ISVE and air sparging. This determination will be made as described in Section 5.2.4.3.

Design parameters that will be determined from the treatability study data will include: number of extraction and injection wells/trenches, well/trench spacing, vapor extraction and injection pumping rates, and offgas treatment requirements.

5.3.2.2. Pilot Study Data Requirements. The ISVE/air sparging pilot study in the contaminated soils in the ONCA will be conducted to obtain the following data:

- Concentrations of VOCs and SVOCs in extracted vapors as a function of time
- Relative quantities of contaminants removed by air sparging versus ISVE
- Pre- and post-test contaminant concentration in soils and groundwater in the test area
- Pre- and post-test microbial population counts and in-situ respiration rates
- Trench vacuum versus vapor extraction flow rate curve
- Soil permeability to air flow in the unsaturated zone (Test procedures are included in Appendix B and the SOPS in the Pilot/Treatability Testing QAPP and FSP)
- Areas/radii of influence of the vapor extraction trenches
- Air sparging injection well head pressure versus injection flow rate
- Areas/radii of influence of the air sparge points.

5.3.2.3. Test Location. The ISVE/air sparging pilot study in the contaminated soils will be located in the ONCA outside the proposed dewatering barrier wall as shown in Figure 5-3. The test location has lithology and contamination representative of the contaminated soils (outside the waste areas).

5.3.2.4. Vapor Extraction Trenches and Air Injection Wells. Three vapor extraction trenches (approximately 60 feet long each) will be installed in the test area as shown in Figure 5-3. Two air injection wells will be installed as shown in Figure 5-3. The test area is laid out so that the air injection wells and monitoring points are aligned with the direction of groundwater flow. Each air injection well will be 2 inches in diameter and will have a 5-foot screened length. The air injection wells will be screened just above the

silty clay confining layer to treat the groundwater and saturated soils above the clay layer. Figures showing the details of the injection wells are included in Appendix B of the Pilot/Treatability Testing QAPP and FSP.

If ISVE/air sparging is deemed appropriate for this site, the pilot equipment will be incorporated into the full scale system.

5.3.2.5. Monitoring Locations. Groundwater/vapor monitoring nests will be installed at four locations as shown in Figure 5-3. A figure showing the conceptual design of the monitoring nests is included in Appendix B of the Pilot/Treatability Testing QAPP and FSP. Each monitoring nest will consist of one groundwater monitoring well with a 10-foot screen and one vapor monitoring point in the unsaturated zone. If ISVE/air sparging is deemed appropriate for the site, the pilot study monitoring wells and probes will be incorporated into the full-scale system.

5.3.2.6. ISVE/Air Sparging Equipment. The pilot-scale ISVE/sparging system will consist of two blowers (one for vapor extraction, and a second for air injection), a condensate collection system, a vapor treatment unit, flow meters, vacuum gauges, vapor sampling ports, and associated piping, valves, and fittings. The ISVE blower will be intrinsically safe, explosion proof regenerative blower. The air injection blower will be a positive displacement blower classified for the general use area. The vapor treatment unit will be capable of treating the extracted vapors to the applicable air discharge limits (i.e., IDEM air quality standards and the 1×10^{-4} to 1×10^{-6} cumulative cancer risk range for nearby residence and site workers as stated on page 30 of the ROD). For this study, activated carbon cannisters will most likely be used for offgas treatment. The need for and design details of additional emissions control will be finalized prior to mobilization based upon input from system suppliers (regarding removal efficiencies) and based upon review of proposed approach by the IDEM Office of Air Management. More specific details of the equipment and a process schematic are included in Appendix B of the Pilot/Treatability Testing QAPP and FSP. Since most of this pilot scale equipment will be leased, it is not likely that it will be incorporated into the full-scale system (if implemented).

5.3.3. Test Procedures

5.3.3.1. Pre-Test Soil Sampling. Two soil samples will be collected from each soil boring drilled to construct the air injection wells and monitoring wells/probes. Each sample will be sent to an off-site laboratory and analyzed for moisture content, grain size distribution, TOC, microbial population count, VOCs, SVOCs, pesticides/PCBs, metals, TKN, and ortho-phosphate. One soil sample from each boring will be collected from the saturated zone, and the other will be collected from the unsaturated zone. These analytical results will be compared to the results of post-treatment soil analyses that will be conducted after the pilot testing is complete. Specific analytical methods and sampling and laboratory procedures to be used are included in the Pilot/Treatability Testing QAPP and FSP.

5.3.3.2. Pre-Test Groundwater Sampling. One groundwater sample will be collected from each of the four groundwater monitoring wells shown in Figure 5-3. The groundwater samples will be analyzed in the field for dissolved oxygen and redox potential. Samples will also be sent to an off-site laboratory for analysis of VOCs, SVOCs, pesticides/PCBs, PCP, metals, microbial plate count, and general water quality parameters. Specific analytical methods and sampling and laboratory procedures to be used are included in the Pilot/Treatability Testing QAPP and FSP.

5.3.3.3. Pre-Test Vapor Sampling. The pressure and temperature at each vapor probe and the ambient air pressure, temperature, and humidity will be measured and recorded prior to conducting pre-test vapor sampling. Soil gas samples will be collected from each vapor monitoring probe prior to beginning operation of the ISVE system. The samples will be analyzed in the field for CO_2, O_2, and total volatile hydrocarbons. The field measurements will be used to assess the initial contaminant concentrations in the vapor phase and to evaluate biological activity prior to conducting the treatability test. Samples will not be collected from the trenches because they will reflect atmospheric conditions and because they have not been purged yet. The initial CO_2 and O_2 concentrations in the soil vapor will be used as a baseline for comparison to in-situ respiration rates at the conclusion of the pilot study. Samples will not be collected for VOC and SVOC analyses since the probes have not been purged yet. These samples will be taken immediately after the system is started up (after at least one bore volume is purged) as discussed in Section 5.3.3.5.

5.3.3.4. Equipment Start-Up. The blowers and vapor treatment unit will be started and checked prior to testing to ensure that they are operating properly. The equipment checks will be conducted without extracting vapors from the extraction trenches or injecting air into the sparging points.

5.3.3.5. ISVE Test. The ISVE system will be operated without injecting air into the air sparging wells to evaluate the performance of ISVE alone. Vapor sampling will be conducted at the extraction trench manifold and at each vapor monitoring probe at the beginning of the ISVE tests to determine the initial composition of the extracted vapors. The vapor samples will be sent to an off-site laboratory for analysis for VOCs, SVOCs, methane, and ethane. Specific analytical methods and sampling and laboratory protocols are included in the Pilot/Treatability Testing QAPP and FSP.

Short term variable flow rate tests will be conducted initially to determine the vacuum versus flow rate operating curve for the ISVE trenches and blower. These short term tests will also be the air permeability tests. Five flow rates will be tested in ascending order. Vacuum measurements will be made frequently at the extraction trench manifold (on the combined discharge line from the extraction trenches) and at each vapor monitoring probe until steady state is reached for each flow rate. The duration of each test will depend on the length of time required until steady state vacuum measurements are achieved at the vapor monitoring probes. Specific details of the test procedure are included in Appendix B, Section B.3.7 of the Pilot/Treatability Testing QAPP and FSP.

A vapor extraction flow rate will be chosen based on the results of the variable flow rate tests, and this flow rate will be used to operate the ISVE system alone for approximately two weeks. Field monitoring and sampling for on-site and off-site analyses will be conducted according to the schedule in Appendix B, Section B.3.7.4 of the Pilot/Treatability Testing QAPP and FSP.

5.3.3.6. Air Sparging Test. Upon completion of the ISVE short term test, the air sparging system will be operated without extracting vapors from the vapor extraction trenches to evaluate the performance of air sparging alone.

Short term variable flow rate tests will be conducted initially to determine the injection well head pressure versus flow rate operating curve for the injection wells and blower.

Five flow rates will be tested in ascending order for each injection well. Pressure measurements will be made frequently at the vapor monitoring probes, monitoring wells, and piezometers until steady state is reached for each flow rate. The duration of each test will depend on the length of time required until steady state pressure measurements are achieved at the vapor monitoring probes. More specific details of the test procedures are included in Appendix B, Section B.3.8 of the Pilot/Treatability Testing QAPP and FSP.

An air injection flow rate will be chosen based on the results of the variable flow rate tests, and this flow rate will be used to operate the air sparging system alone for approximately two weeks. A flow rate will be selected based on data regarding the degree of mounding of the water table and radius of infuence. A more specific discussion is included in Appendix B, Section B.3.8 of the Pilot/Treatability Testing QAPP and FSP. Field monitoring and sampling for on-site and off-site analyses will be conducted according to the schedule in Appendix B, Section B.3.8.3 of the Pilot/Treatability Testing QAPP and FSP.

5.3.3.7. ISVE/Air Sparging Test. A constant flow rate test will be conducted using ISVE and air sparging together. The air injection and vapor extraction rates used for this test will be chosen on the basis of the results of the previous tests. The test will be conducted for a minimum of two weeks, but it may run for a longer time frame. The specific duration of the test will be approved by USEPA and IDEM on the basis of the results and the schedule of the remaining RD/RA activities at the site. Specific test procedures are included in Appendix B, Section B.3.9 of the Pilot/Treatability Testing QAPP and FSP.

Field monitoring and sampling for on-site and off-site analyses will be conducted according to the schedule in Appendix B, Section B.3.9 of the Pilot/Treatability Testing QAPP and FSP.

5.3.3.8. Post-Test Vapor Sampling. Post-test vapor sampling will be conducted after the ISVE/air sparging system is shut down. Vapor samples will be collected from the vapor monitoring probes and extraction trench manifold and analyzed in the field for CO_2, O_2, and total volatile hydrocarbons. The post-test vapor sampling will be conducted immediately after system shut down, and at 0.5, 1, 2, 4, 8, 12, 24, and 48 hours after shut down. These measurements will be compared to the pre-test values and used to evaluate whether biological activity was enhanced due to system operation. This measurement

frequency may be adjusted in the field based upon the observed respiration rates. Additionally, within 48 hours after completion of the long term test, vapor samples will be collected from the extraction trench manifold and each vapor monitoring point and submitted to an off-site laboratory for VOC, SVOC, methane, and ethane analyses. Specific test procedures and analytical methods and sampling and laboratory procedures are included in Appendix B; Section B.3.10 of the Pilot/Treatability Testing QAPP and FSP.

5.3.3.9. Post-Test Groundwater Sampling. Within 48 hours after completion of the long term test, one groundwater sample will be collected from each of the four groundwater monitoring wells. The groundwater samples will be analyzed in the field for dissolved oxygen, pH, conductivity, temperature, and redox potential. Samples will also be sent to an off-site laboratory for analysis for VOCs, SVOCs, pesticides/PCBs, PCP, metals, and general water quality parameters. Specific analytical methods and sampling and laboratory procedures are included in the Pilot/Treatability Testing QAPP and FSP.

Post-Test Soil Sampling. Within two weeks of completing the long term test, soil samples will be collected from soil borings installed within two feet of each of the soil borings sampled for the pre-test soil sampling. Two soil samples will be collected from each soil boring. The samples will be sent to an off-site laboratory and analyzed for moisture content, grain size distribution, TOC, microbial population count, VOCs, SVOCs, pesticides/PCBs, metals, TKN, and ortho-phosphate. One soil sample from each boring will be collected from the saturated zone, and the other will be collected from the unsaturated zone. The post-test soil sampling results will be compared to the pre-test soil sampling results to evaluate the effectiveness of ISVE/air sparging in the pilot test area. Specific analytical methods and sampling and laboratory procedures are included in the Pilot/Treatability Testing QAPP and FSP.

5.3.4. Data Evaluation

The data generated from the ISVE/air sparging pilot study in the contaminated soils in the ONCA will be used to: (1) evaluate the effectiveness of ISVE/air sparging for removing volatile contaminants from the contaminated soils and the potential for the technology to meet the performance standards, (2) evaluate the effectiveness of ISVE/air sparging for enhancing naturally-occurring biodegradation of the contaminants, and (3) determine design parameters for full-scale implementation of ISVE/air sparging in the contaminated soils.

5.3.4.1. Contaminant Removal and Performance Standards. The mass removal of contaminants will be calculated and used to perform a mass balance. The effectiveness of ISVE/air sparging for removing contaminants and the potential for the technology to meet the performance standards will be evaluated based upon:

- The mass of contaminants removed during the ISVE/air sparging pilot study, calculated based upon extracted vapor concentrations and vapor extraction flow rates

- Comparison between pre-test and post-test soil, vapor, and groundwater sampling results

- Predicted cleanup time and ability to meet performance standards using the concentration reductions observed during the pilot testing, extraction system offgas concentrations, estimated mass of contamination, and estimated flow rates for the full-scale system.

A more detailed discussion of the data evaluation is included in Appendix B of the Pilot/Treatability Testing QAPP and FSP.

5.3.4.2. Bioremediation Enhancement. The effectiveness of ISVE/air sparging for enhancing naturally-occurring biodegradation of the contaminated soil will be determined by evaluating the pre- and post-test vapor sampling results. If oxygen is the limiting factor in biodegradation, the pre-test soil vapor samples in the contaminated area should exhibit elevated CO_2 concentrations (in the range of 5% to 15%), depressed O_2 concentrations (<5%), and elevated hydrocarbon concentrations. Background soil vapor concentrations should be similar to atmospheric concentrations for CO_2 and O_2, with hydrocarbon concentrations generally less than 100 ppm. During the ISVE/air sparging test, the O_2 concentration in the soil vapor in the test area should increase to approximately 20%. Using the O_2 data from the 48-hour post-vapor sampling test, an oxygen utilization rate will be calculated. If the utilization rate is within the range of 0.05% to 1% O_2/hour, this will be sufficient evidence that some microbial activity is occurring in the unsaturated zone and that the addition of O_2 in this area will enhance biodegradation.

Ideally, the utilization rate from the post-test vapor sampling should be compared to a utilization rate from a background vapor monitoring probe. Unfortunately, the probe would need to be very shallow (and therefore would reflect atmospheric conditions) and would need to be located off of the site. Consequently, a background vapor probe for this purpose is not believed to be of significant value.

In addition to the above, microbial plate counts in pre-test and post-test soil and groundwater samples will be compared to see if the microbial population has noticeably increased. Analytical data for specific constituents required for biological growth will also be evaluated to determine if other factors could be limiting biodegradation. The analytical data will also provide information regarding the pre- and post-test conditions and their suitability for biological activity.

5.3.4.3. Design Parameters. The design parameters for full-scale ISVE will be determined as follows:

- An ISVE blower operating curve will be generated by plotting vapor extraction flow rate per foot of trench versus vacuum at the extraction trench. The operating curve will be used during full-scale ISVE system design to determine the vapor extraction flow rate and vacuum to be used for the full-scale system.

- An air injection blower operating curve will be generated by plotting air injection flow rate versus injection pressure. The operating curve will be used during full-scale air sparging system design to determine the air injection flow rate to be used for the full-scale system.

- Extracted vapor concentrations at the vapor extraction trench, volumes of extracted condensate, and condensate composition will be used to design the offgas treatment system.

- Extracted vapor concentrations at the exhaust of the blower will be used to verify removal efficiencies.

- The effective treatment areas of the vapor extraction trenches and air injection wells will be calculated based upon the measurements made during the pilot study. Effective treatment area estimates will be used to estimate sparge point and trench spacing during full-scale design.

5.4 TREATABILITY STUDY REPORTS

A treatability study report will be prepared for each pilot/treatability study at the conclusion of the testing. Each report will contain field and laboratory monitoring results, descriptions of the field procedures used, results of the data evaluation for each pilot/treatability test, recommendations, and preliminary design parameters. The pilot/treatability study reports will be submitted as separate reports. For the On-Site Containment Area, the report will be submitted and request from USEPA that a specific technology be utilized or recommend an alternate course of action.

5.5 SCHEDULE

The schedule for the pilot/treatability studies is shown on Figure 5-4. The dates shown are contingent upon agency review and approval times. The pilot ISVE tests will be operated for a minimum of two months, but it is anticipated that the ISVE test will run for six months. The ISVE/air sparging tests will take approximately 6 weeks to complete. Both tests may be operated for a longer period of time, but they will not be operated longer than the time it takes to implement the treatment for source materials in the off-site area. The specific duration of the tests may be modified by USEPA on the basis of the results and the schedule of the remaining RD/RA activities at the site.

The schedule for remedial design (Tasks 4 through 9) of the LTTT portion of the remedy is based on the deliverables identified in the SOW. The list of deliverables and sequencing does not match the requirements for a performance based procurement of services which is usually the preferred and most cost-effective approach for completing on-site thermal treatment remedies. The duration of the attached schedule could be reduced significantly by combining SOW deliverables and minimizing review and comment cycles.

TABLE 5-1

PERFORMANCE STANDARDS

Chemical	Remediation Criteria	Basis	Corresponding Risk	
			Carcinogenic	Non-Carcinogenic
Groundwater — Organic Compounds (µg/l)				
Acetone	192 to 2,300	HI	NA	0.08 to 1.0
Benzene	5.0	MCL	6.5×10^{-7}	NA
bis-(2-Chloroethyl) ether	21	Risk	1.0×10^{-6}	NA
bis-(2-Ethylhexyl) phthalate	5.8	Risk	1.0×10^{-6}	NA
Branched alkanes	18 to 210	HI	NA	0.08 to 1.0
2-Butanone	2,000 to 24,000	HI	NA	0.08 to 1.0
Chloromethane	8.4	Risk	1.0×10^{-6}	NA
cis--1,2-Dichloroethene	28 to 330	HI	NA	0.08 to 1.0
Cyclic ketones	5.8	Risk	1.0×10^{-6}	NA
1,1-Dichloroethane	183 to 2,200	HI	NA	0.08 to 1.0
1,4-Dichlorobenzene	3.3	Risk	1.0×10^{-6}	NA
Dimethyl ethyl benzenes	21 to 250	HI	NA	0.08 to 1.0
Ethylbenzene	33 to 390	HI	NA	0.08 to 1.0
Isophorone	19	Risk	1.0×10^{-6}	NA
4-Methyl-2-pentanone	53 to 640	HI	NA	0.08 to 1.0
4-Methylphenol	142 to 1,700	HI	NA	0.08 to 1.0
Methylene chloride	5.0	MCL	5.4×10^{-7}	NA
Non-cyclic acids	23 to 280	HI	NA	0.08 to 1.0
Pentachlorophenol	1.0	MCL	1.5×10^{-6}	NA
Polychlorinated biphenyls	0.06	Risk	1.0×10^{-6}	NA
Tetrachloroethene	5.0	MCL	6.2×10^{-7}	NA
Trichloroethene	5.0	MCL	2.1×10^{-7}	NA
Vinyl chloride	0.25	Risk	1.0×10^{-6}	NA
Groundwater—Metals (µg/l)				
Arsenic	8.8	Risk	1.0×10^{-6}	<0.1
Beryllium	0.02	Risk	1.0×10^{-6}	NA
Manganese	275 to 3,300	HI	NA	0.08 to 1.0
Thallium	0.2 to 2.4	HI	NA	0.08 to 1.0
Soil—Organic Compounds (mg/kg)				
2-Butanone	21 to 620	HI	NA	0.03 to 1.0
4,4'-DDD	0.12	Risk	1.0×10^{-6}	NA
4,4'-DDE	0.16	Risk	1.0×10^{-6}	NA
4,4'-DDT	0.088	Risk	1.0×10^{-6}	NA
4-Methyl-2-pentanone	21 to 630	HI	NA	0.03 to 1.0
Acetone	80 to 2,400	HI	NA	0.03 to 1.0
Aldrin	0.002	Risk	1.0×10^{-6}	NA
alpha-BHC	0.0047	Risk	1.0×10^{-6}	NA
Benzene	1.0	Risk	1.0×10^{-6}	NA
beta-BHC	0.016	Risk	1.0×10^{-6}	NA
bis(2-chloroethyl)ether	0.027	Risk	1.0×10^{-6}	NA

TABLE 5-1

PERFORMANCE STANDARDS
(CONTINUED)

Chemical	Remediation Criteria	Basis	Corresponding Risk	
			Carcinogenic	Non-Carcinogenic
Soil—Organic Compounds (mg/kg) (continued)				
bis-(2-Ethylhexyl)phthalate	1.1	Risk	1.0×10^{-6}	NA
Branched alkanes	26 to 770	HI	NA	0.03 to 1.0
Carbon tetrachloride	0.38	Risk	1.0×10^{-6}	NA
Chlorobenzene	5 to 150	HI	NA	0.03 to 1.0
Chloroethane	90 to 2,700	HI	NA	0.03 to 1.0
Chloroform	9.5	Risk	1.0×10^{-6}	NA
1,4-Dichlorobenzene	2.4	Risk	1.0×10^{-6}	NA
1,2-Dichloroethane	0.64	Risk	1.0×10^{-6}	NA
1,1-Dichloroethene	0.098	Risk	1.0×10^{-6}	NA
cis-1,2-Dichloroethene	8.3 to 250	HI	NA	0.03 to 1.0
1,2-Dichloropropane	0.42	Risk	1.0×10^{-6}	NA
2,4-Dinitrotoluene	0.044	Risk	1.0×10^{-6}	NA
2,6-Dinitrotoluene	0.044	Risk	1.0×10^{-6}	NA
Cyclic ketones	7.3	Risk	1.0×10^{-6}	NA
Cyclic polyaromatic hydrocarbons	0.0026	Risk	1.0×10^{-6}	NA
Di-n-butylphthalate	77 to 2,300	HI	NA	0.03 to 1.0
Diethyl benzenes	43 to 1,300	HI	NA	0.03 to 1.0
Dimethyl ethyl benzenes	43 to 1,300	HI	NA	0.03 to 1.0
Endosulfan I	0.02 to 0.63	HI	NA	0.03 to 1.0
Ethyl methyl benzenes	163 to 4,900	HI	NA	0.03 to 1.0
Ethylbenzene	43 to 1,300	HI	NA	0.03 to 1.0
gamma-BHC (Lindane)	0.046	Risk	1.0×10^{-6}	NA
Halogenated alkanes	77 to 2,300	HI	NA	0.03 to 1.0
Heptachlor epoxide	0.0033	Risk	1.0×10^{-6}	NA
Hexachlorobenzene	0.018	Risk	1.0×10^{-6}	NA
Hexachlorobutadiene	0.36	Risk	1.0×10^{-6}	NA
Isophorone	7.2	Risk	1.0×10^{-6}	NA
Methyl propyl benzenes	16 to 490	HI	NA	0.03 to 1.0
Methylated naphthalenes	3 to 85	HI	NA	0.03 to 1.0
Methylene chloride	6.2	Risk	1.0×10^{-6}	NA
n-chain Alkanes	25 to 760	HI	NA	0.03 to 1.0
n-Nitrosodiphenylamine	12.0	Risk	1.0×10^{-6}	NA
Naphthalene	3 to 82	HI	NA	0.03 to 1.0
Nitrogenated benzenes	0.2 to 6.2	HI	NA	0.03 to 1.0
Non-cylclic acids	33 to 1,000	HI	NA	0.03 to 1.0
Oxygenated benzenes	40 to 1,200	HI	NA	0.03 to 1.0
Pentachlorophenol	0.43	Risk	1.0×10^{-6}	NA
Propenyl benzenes	11 to 320	HI	NA	0.03 to 1.0
Styrene	1.7	Risk	1.0×10^{-6}	NA
1,2,4-Trichlorobenzene	0.5 to 16	HI	NA	0.03 to 1.0
1,1,1-Tetrachloroethane	77 to 2,300	HI	NA	0.03 to 1.0
1,1,2,2-Tetrachloroethane	0.28	Risk	1.0×10^{-6}	NA

TABLE 5-1

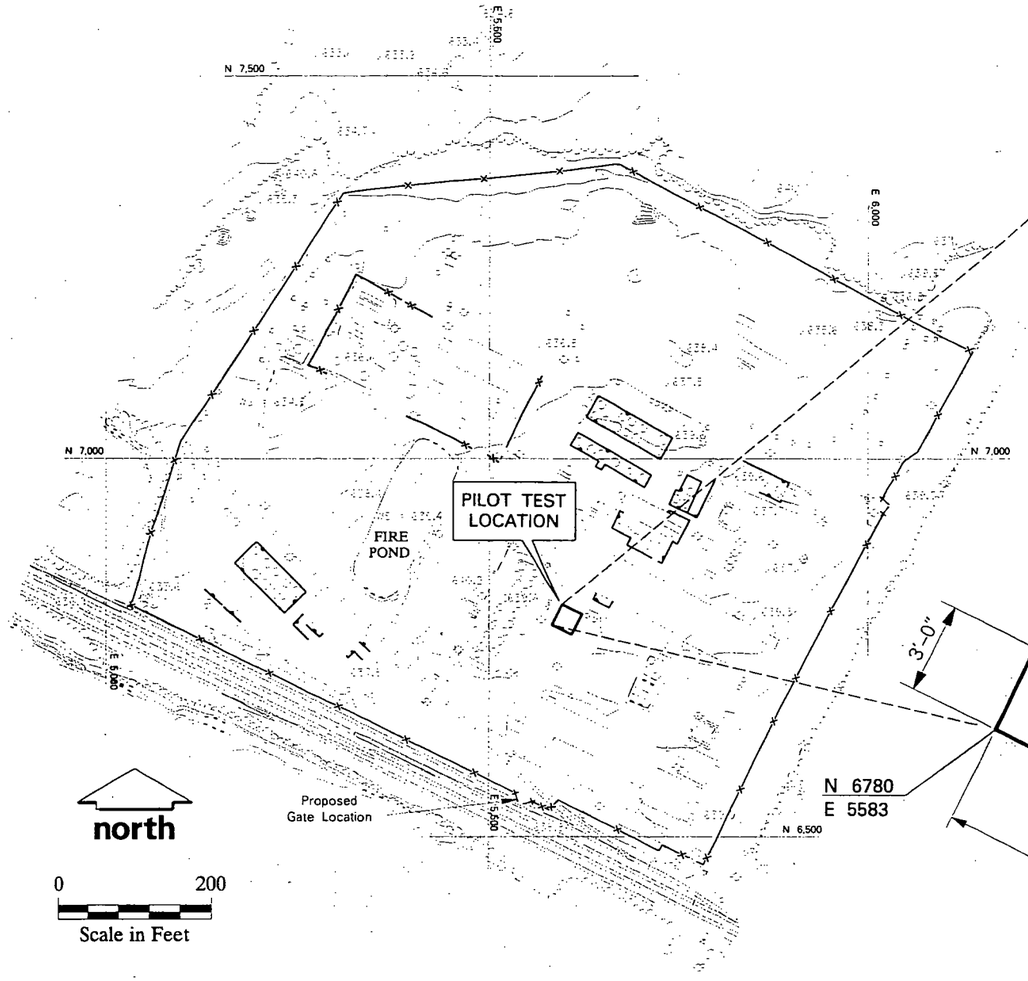
PERFORMANCE STANDARDS (CONTINUED)

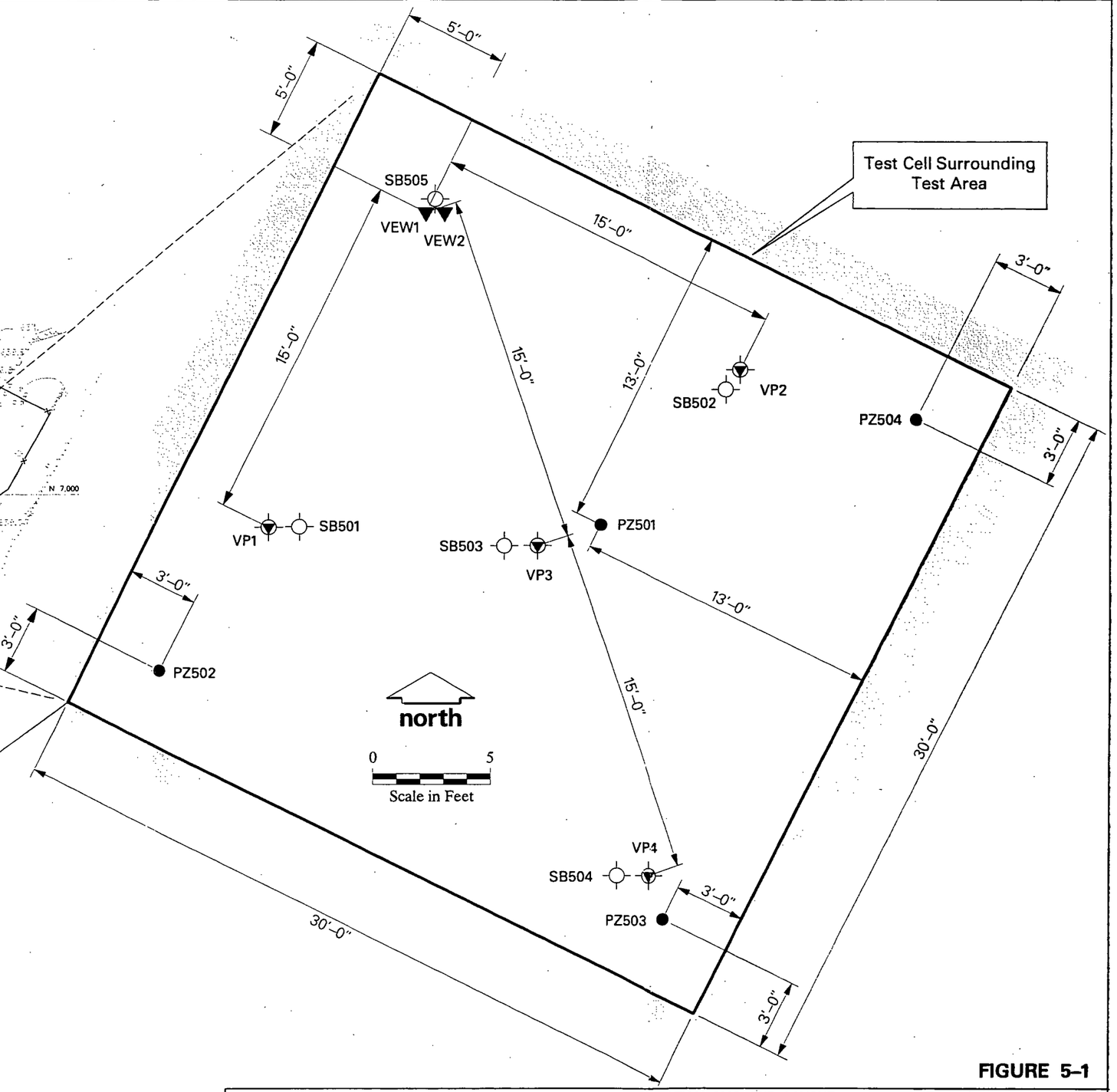
Chemical	Remediation Criteria	Basis	Corresponding Risk	
			Carcinogenic	Non-Carcinogenic
Soil—Organic Compounds (mg/kg) (continued)				
Tetrachloroethene	1.1	Risk	1.0×10^{-6}	NA
1,1,2-Trichloroethane	0.51	Risk	1.0×10^{-6}	NA
Toluene	167 to 5,000	HI	NA	0.03 to 1.0
Trichloroethene	5.3	Risk	1.0×10^{-6}	NA
Vinyl chloride	0.031	Risk	1.0×10^{-6}	NA
Xylenes (mixed)	867 to 26,000	HI	NA	0.03 to 1.0
Soil—Metals (mg/kg)				
Antimony	0.5 to 15	HI	NA	0.03 to 1.0
Barium	87 to 2,600	HI	NA	0.03 to 1.0
Cadmium	2 to 51	HI	NA	0.03 to 1.0
Hexavalent chromium	47 to 1,400	HI	NA	0.03 to 1.0

Source: Tables 7 and 8 from the Record of Decision for the American Chemical Service Site

Management Review
Other
Technical Review
Project Manager
Graphic Standards
Lead Professional
QUALITY CONTROL

This document has been developed for a specific application and may not be used without the written approval of Montgomery Watson.





LEGEND

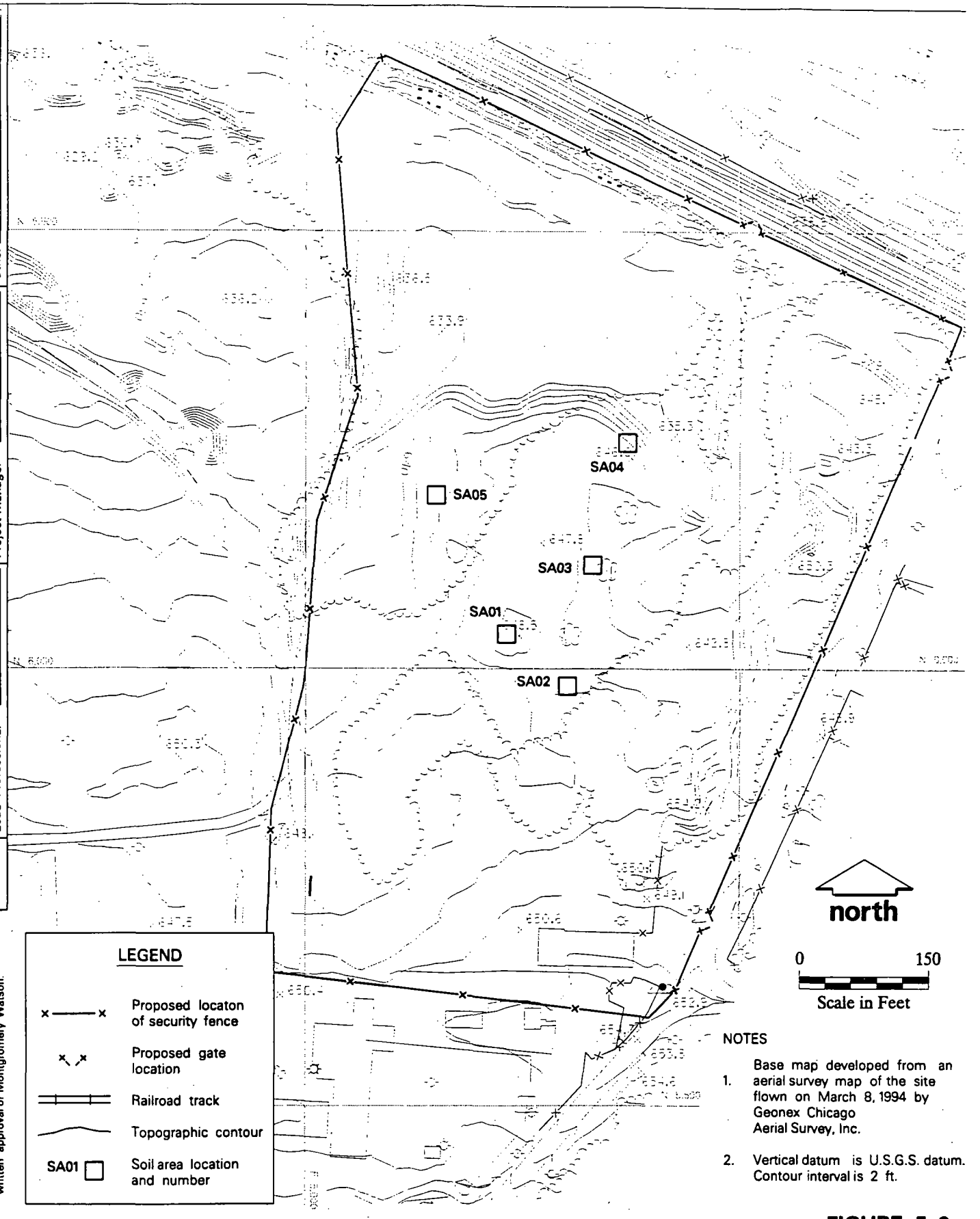
- Vapor extraction well
- Vapor monitoring probe nest
- Post-test soil borings
- Piezometer-groundwater extraction well

FIGURE 5-1

Developed By	Drawn By	**LOCATION MAP AND LAYOUT ISVE PILOT STUDY ON BURIED WASTE IN THE ON-SITE AREA**
Approved By	Date	PRE-DESIGN WORK PLAN AMERICAN CHEMICAL SERVICE, INC. NPL SITE GRIFFITH, INDIANA
Reference		
Revisions		

Drawing Number 4077.0191

MONTGOMERY WATSON



Management Review
Other

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LEGEND

- Proposed locaton of security fence
- Proposed gate location
- Railroad track
- Topographic contour
- SA01 — Soil area location and number

NOTES

1. Base map developed from an aerial survey map of the site flown on March 8, 1994 by Geonex Chicago Aerial Survey, Inc.
2. Vertical datum is U.S.G.S. datum. Contour interval is 2 ft.

FIGURE 5-2

Developed By	Drawn By	**LOCATION MAP PRETREATMENT/MATERIAL HANDLING STUDY IN THE OFF-SITE AREA TEST PIT**	Drawing Number 4077.0191
Approved By	Date		
Reference		PRE-DESIGN WORK PLAN AMERICAN CHEMICAL SERVICE, INC. NPL SITE GRIFFITH, INDIANA	**MONTGOMERY WATSON**
Revisions			

Management Review
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Project Manager

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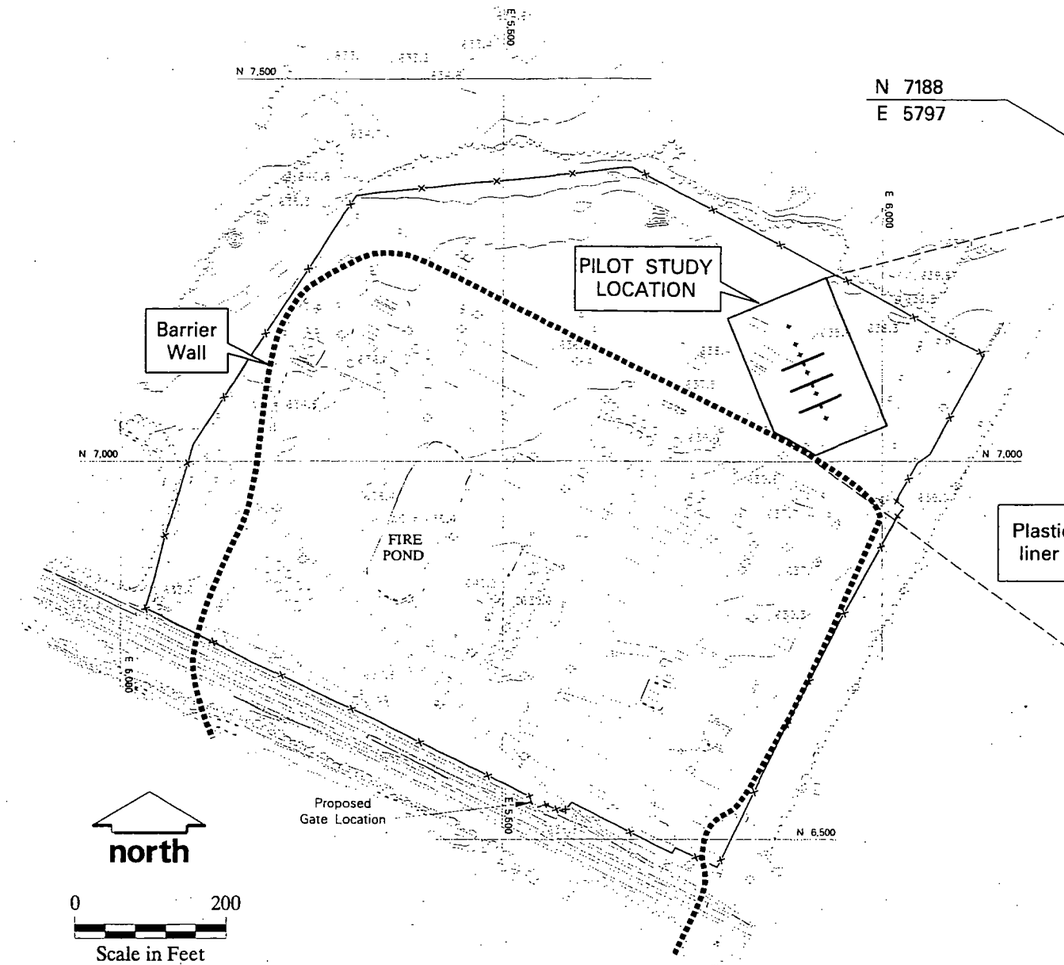

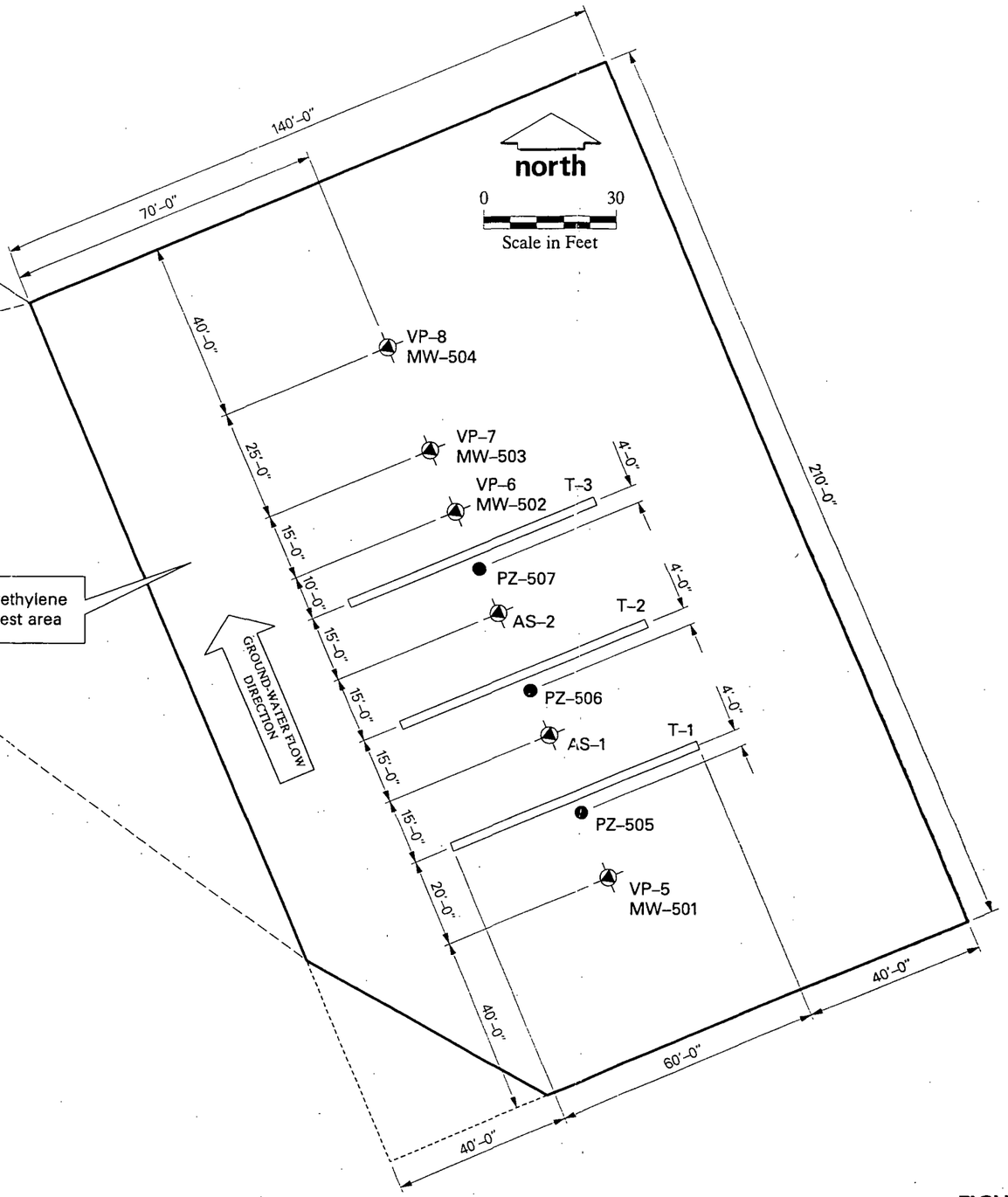

LEGEND

- Groundwater/vapor monitoring well
- Piezometer–groundwater extraction well

FIGURE 5–3

Developed By	Drawn By	**ISVE/AIR SPARGING PILOT STUDY LOCATION MAP AND LAYOUT ON SOILS IN THE ONCA**	Drawing Number 4077.0191
Approved By	Date 8/26/96		
Reference		PRE-DESIGN WORK PLAN AMERICAN CHEMICAL SERVICE, INC. NPL SITE GRIFFITH, INDIANA	**MONTGOMERY WATSON**
Revisions			

AMERICAN CHEMICAL SERVICES
SCHEDULE FOR PILOT AND TREATABILITY STUDIES

ID	Task Name	Duration
1		
2	Submittal of Work Plan, QAPP, FSPs	1 d
3	Agency Review for PMHS/LTTT	81 d
4	Partial approval for PMHS/LTTT	1 d
5	Agency Review for ISVE/air sparging	59 d
6	Revise QAPP/FSP	30 d
7	Agency Review	45 d
8	Agency Appproval of QAPP/FSP	1 d
9		
10	ISVE/AIR SPARGING	452 d
11	Task 1 ISVE pilot test on buried wastes	452 d
12	Task 1.1 Contracting	21 d
13	Task 1.2 Mobilization/setup	5 d
14	Task 1.3 Install test cell	14 d
15	Task 1.4 Install prezometers	2 d
16	Task 1.5 Dewater test cell	14 d
17	Task 1.6 Install ISVE and vapor points	5 d
18	Task 1.7 Construct ISVE system	14 d
19	Task 1.8 Pre-test vapor sampling	1 d
20	Task 1.9 Initial vapor sampling	1 d
21	Task 1.10 Short term variable flow rate tests	14 d
22	Task 1.11 Long term constant rate test	180 d
23	Task 1.12 Post-test vapor sampling	2 d
24	Task 1.13 Post-test soil/waste sampling	14 d
25	Task 1.14 Laboratory analysis	45 d
26	Task 1.15 Develop draft report (to agency)	35 d
27	Task 1.16 Agency review/comment report	60 d
28	Task 1.17 Revise/finalize report	60 d
29		
30	Task 2 ISVE/air sparging pilot test on contaminated soil	342 d
31	Task 2.1 Contracting	21 d
32	Task 2.2 Mobilization/setup	5 d
33	Task 2.3 Install trenches and piping	5 d
34	Task 2.4 Install wells and piezometers	5 d
35	Task 2.5 Construct ISVE/air sparging system	14 d
36	Task 2.6 Pre-test vapor sampling	1 d
37	Task 2.7 Initial vapor sampling	1 d
38	Task 2.8 Collect pre-test groundwater samples	2 d
39	Task 2.9 Variable rate ISVE test	14 d
40	Task 2.10 Constant rate ISVE test	14 d
41	Task 2.11 Short term variable rate air sparging test	14 d
42	Task 2.12 Constant rate air sparging test	14 d
43	Task 2.13 Long term constant rate ISVE/air sparge test	14 d
44	Task 2.14 Collect post-test vapors	2 d
45	Task 2.15 Collect post-test groundwater samples	2 d
46	Task 2.16 Collect post-test soil/waste samples	14 d
47	Task 2.17 Laboratory analysis	45 d
48	Task 2.18 Develop draft report (to agency)	35 d
49	Task 2.19 Agency review/comment report	60 d
50	Task 2.20 Revise/finalize report	60 d
51		
52	LOW TEMPERATURE THERMAL TREATMENT (LTTT)	302 d
53	Task 1 Pretreatment/Material Handling Study (PMHS)	186 d
54	Task 1.1 Contracting	15 d
55	Task 1.2 Mobilization/setup	2 d
56	Task 1.3 Background air sampling	2 d
57	Task 1.4 Test pit excavation/screening	10 d
58	Task 1.5 Decontamination/demobilization	2 d
59	Task 1.6 Develop draft report (to agency)	70 d
60	Task 1.7 Agency review/comment report	25 d
61	Task 1.8 Revise/finalize report	60 d
62		
63	Task 2 Thermal Treatability Study (TTS)	273 d
64	Task 2.1 Sample receipt/preparation	21 d
65	Task 2.2 Tray testing	39 d
66	Task 2.3 RTA testing	38 d
67	Task 2.4 Develop draft report (to agency)	70 d
68	Task 2.5 Agency review/comment report	45 d
69	Task 2.6 Revise/finalize report	60 d

Project: ACS PILOT AND TREATABILITY STUDIES
Project Number: 4077.0191

Task | Progress | Milestone | Summary | Rolled Up Task | Rolled Up Milestone | Rolled Up Progress

1 OF 2

FIGURE 5-4

Schedule is subject to USEPA approval of the work plan, QAPP, and FSP documents on the dates shown

DRAFT

QUALITY ASSURANCE PROJECT PLAN
AND
FIELD SAMPLING PLAN
FOR
PILOT/TREATABILITY TESTING

AMERICAN CHEMICAL SERVICE, INC.
NPL SITE

GRIFFITH, INDIANA

January 1997

PREPARED FOR:

ACS RD/RA EXECUTIVE COMMITTEE

PREPARED BY:

MONTGOMERY WATSON AMERICAS, INC.

QUALITY ASSURANCE PROJECT PLAN AND FIELD SAMPLING PLAN FOR PILOT/TREATABILITY TESTING

AMERICAN CHEMICAL SERVICES INC.
NPL SITE
GRIFFITH, INDIANA

JANUARY 1997

Prepared by: Montgomery Watson

Montgomery Watson Project Manager	Date
Montgomery Watson QA Officer	Date
IDEM Project Manager	Date
U.S. EPA Region 5 Remedial Project Manager	Date
U.S. EPA Region 5 Quality Assurance Reviewer	Date
IEA Quality Assurance Officer	Date
Quanterra Quality Assurance Officer	Date

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LIST OF ACRONYMS

1,1-DCE	1,1-Dichloroethene
2,4,-D	2,4-Dichlorophenoxyacetic acid
ACS	American Chemical Service
AD	Air Downwind
ARARs	Applicable or Relevant and Appropriate Requirements
ASTM	American Society of Testing and Materials
AU	Air Upwind
BFB	Bromofluorobenzene
C	Centigrade
CCC	Continuing Calibration Compounds
CLP	Contract Laboratory Program
COC	Chain of Custody
DQO	Data Quality Objective
EICP	Extracted ion current profile
FSP	Field Sampling Plan
GC	Gas Chromatograph
GC/MS	Gas Chromatography/Mass Spectroscopy
GWMP	Ground Water Management Plan
ICB	Initial Calibration Blanks
ICV	Initial Calibration Verification
IDEM	Indiana Department of Environmental Management
IEA	Industrial Environmental Analytical
ISVE	In-Situ Soil Vapor Extraction
LCS	Laboratory Control Sample
LCS	Laboratory Control Sample
LIMS	Laboratory Information management Systems
LTTT	Low Temperature Thermal Treatment
MDL	Method Detection Limit
mg/l	Milligrams per Liter
ml	Milliliter
Montgomery Watson	Montgomery Watson Americas, Inc.
MS	Matrix Spike
MSD	Matrix Spike Duplicate
NIST	National Institute of Standards and Technology
NPL	Nation Priorities List
OFCA	Off-Site Containment Area
ONCA	On-site Containment Area
PARCC	Precision Accuracy Representativeness, Completeness, and Comparability
PCE	Tetrachloroethene
PE	Performance Evaluation
ppm	Part Per Million
PQL	Practical Quantitation Level
PRP	Potentially Responsible Party
QA	Quality Assurance
QAO	Quality Assurance Officer

QAPP	Quality Assurance Project Plan
QC	Quality Control
Quanterra	Quanterra Environmental Services
RCRA	Resource Conservation and Recovery Act
RD/RA	Remedial Design/Remedial Action
RF	Response Factor
RI/FS	Remedial Investigation/Feasibility Study
ROD	Record of Decision
RPD	Relative Percent Difference
RPM	Remedial Project Manager
RSD	Relative Standard Deviation
SBP	Still Bottoms Pond
SD	Spike Duplicate
SD	Standard Deviation
SOP	Standard Operating Procedure
SPCC	System Performance Calibration Compounds
SRM	Standard Reference Material
TCE	Trichloroethene
TSS	Technical Support Section
U.S. EPA	U.S. Environmental Protection Agency
UAO	Unilateral Administrative Order
µg/l	Micrograms per Liter

1.0 PROJECT DESCRIPTION

1.1 INTRODUCTION

This Quality Assurance Project Plan (QAPP) presents the project organization, objectives, and functional activities, and the project-specific quality assurance (QA) and quality control (QC) procedures for the following pilot and treatability studies that will be conducted at the American Chemical Service (ACS), Inc. National Priorities List (NPL) Site in Griffith, Indiana:

- In-Situ Vapor Extraction (ISVE) pilot study on buried wastes in the On-Site Area
- ISVE/Air Sparging pilot study on contaminated soils in the On-Site Containment Area (ONCA)
- Low Temperature Thermal Treatment (LTTT) treatability study on buried waste in the Off-Site Containment Area (OFCA), including a bench-scale LTTT and a pre-treatment/material handling pilot test.

These studies are being conducted as part of a Remedial Design/Remedial Action (RD/RA) to assess whether full-scale remediation using these technologies can meet the cleanup criteria specified in the Record of Decision (ROD; U.S. EPA, 1992) as listed in Table 1-1 and to provide data for full-scale design.

The objective of this QAPP is to define the project data quality objectives (DQOs) for each treatability study and to describe the field and analytical procedures that will be used to ensure that the resulting data are of sufficient quality to evaluate the effectiveness of each technology and to assess whether these technologies can meet the performance criteria specified in the ROD (Table 1-1).

All procedures detailed this QAPP are in accordance with applicable professional technical standards, United States Environmental Protection Agency (U.S. EPA) Region V requirements, Indiana Department of Environmental Management (IDEM)

requirements, and the project-specific DQOs. This QAPP was prepared by Montgomery Watson Americas, Inc. (Montgomery Watson) in accordance with the *Region V Superfund Model QAPP* (May 1996, Revision 1) and the *Guidance for Conducting Treatability Studies Under CERCLA* (U.S. EPA, 1994). The analytical and data validation protocol described in this QAPP is based on the *EPA Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW-846, 3rd Edition*. (U.S. EPA, 1992), *EPA Methods for Chemical Analysis of Water and Wastes, EPA Manual 600/4-79-020* (U.S. EPA, 1983), and *the USEPA Contact Laboratory Program National Functional Guidelines for Organic and Inorganic Date Review* (U.S. EPA, 1994).

1.2 SITE DESCRIPTION

1.2.1. Site Size and Borders

The ACS Site is located at 420 South Colfax Avenue in the City of Griffith, Indiana, in the northwest corner of the state. The Site is bordered on the east and northeast by Colfax Avenue. The Chesapeake and Ohio Railway bisects the Site in a northwest-southeast direction, between the fenced On-Site Area (north) and the Off-Site Area (south). On the west and northwest, south of the Chesapeake and Ohio Railway, the Site is bordered by the abandoned Erie and Lackawanna Railway, and the Griffith Municipal Landfill. North of the Chesapeake and Ohio Railway, the Site is bordered on the west by wetland areas. The northern boundary of the Site is formed by the Grand Trunk Railway.

The Site comprises approximately 30 acres of land including the ONCA, the Still Bottoms Pond/Treatment Lagoon (SBP Area), the OFCA, and the Kapica/Pazmey Area[1]. The Griffith Municipal Landfill is also located within the boundaries of the Site as defined by U.S. EPA. However, the landfill is not addressed by these treatability studies and is not part of the remedy. The landfill, which is an active solid waste disposal facility that has operated since the 1950s, currently is going through closure.

[1] The terms On-Site and Off-Site are used to denote particular portions of the ACS Site: both areas are within the CERCLA Site. The Off-Site Containment area is designated as off-site only because it is adjacent to, rather than within the boundaries of the property where ACS currently conducts its chemical formulation operations. However, ACS owns the property and as noted, for CERCLA purposes, both of these areas are considered on-site.

1.2.2. Operational History

The Site contains an active chemical processing facility and several former land disposal areas. The chemical processing facility began operation in May 1955 as a solvent recovery facility. Solvent recovery remained the primary on-Site operation until the late 1960s, when operations changed to the manufacture of small quantities of specialty chemicals. These manufacturing operations included treating rope with fungicide, bromination, and treating ski lift cable.

In 1961, ACS sold a two-acre parcel to Mr. John Kapica, and in 1962 Mr. Kapica began operating a drum reclaiming/reconditioning business at the location. Kapica Drum was sold to Pazmey Corporation in February 1980. Kapica and Pazmey operated from 1980 to 1987. The Pazmey Corporation property was sold to Darija Djurovic in March 1987.

ACS' solvent operations included recovery of spent solvent mixtures containing alcohols, ketones, esters, chlorinated solvents, aromatics, aliphatics, and glycols. In the early years of operation, spent solvents were stored in 55-gallon drums at various locations at the Site. Solvent was recovered in batch evaporation units, which were charged by pumping material directly from 55-gallon drums into the evaporation vessels. Until 1966, still bottoms from the evaporation vessels were disposed in the Still Bottom Pond. ACS installed its first incinerator in 1966 and installed a second incinerator in 1969. The incinerators were used to burn still bottoms and non-reclaimable materials generated at the Site, and wastes from off site. The incinerator units were dismantled in 1977.

From 1970 to 1975, spent solvents reclaimed at the Site were similar to those handled in the 1960s. However, during this period an increasing percentage of shipments were received at the Site in bulk tanker trucks. In addition, the batch manufacturing processes were expanded during this period. A lard oil process, tallow and animal rendering, was used to manufacture a lubricant product. This process, as well as a soldering flux operation, was discontinued prior to 1990. In 1971, the additive manufacturing area was built to produce various detergents, lubricants, and chemical additives, in addition to soldering flux, various amines, methanol, formaldehyde, sodium hydroxide, and maleic anhydride. An epoxidation plant was constructed in 1974, and a bromination operation

using hexane was added in 1975. The epoxidation plant used toluene or benzene as a reaction carrier at various times up until 1990.

Some time between 1975 and 1990, the solvent distillation units were replaced with new units, although the types of solvent wastes reclaimed remained essentially the same. Spent and reclaimed solvent recovery tank farms were constructed during this period, and the majority of the spent solvent waste streams were shipped in bulk tanker trucks, although drummed wastes were still processed. A hazardous waste drum unloading dock and storage area was built in the early 1970s, with spill containment curbing and a sump area added at a later date. In September 1990, ACS ceased accepting hazardous waste shipments and filed for closure. On March 31, 1993 ACS completed closure and terminated its interim Resource Conservation and Recovery Act (RCRA) status.

ACS currently operates a chemical production facility at the Site. The operations include chemical reaction processes, custom blending, and product distribution. The facility encompasses 8.5 acres with a process building, tank farms, loading and unloading areas, a laboratory, offices, and support utility buildings. The company operates 24 hours per day, five to six days per week. The operating production facility is secured by a continuous fence with a single, controlled access gate.

1.2.3. Land Disposal History

When ACS began operations in 1955, the still bottoms from the solvent recovery operations were disposed of in the Still Bottoms Pond/Treatment Lagoon area. In 1972, the pond and lagoons were drained, and were used to landfill drums partially filled with sludge materials.

The OFCA was used to landfill wastes including materials excavated from the Still Bottoms/Treatment Lagoon between 1958 and 1975. The waste types disposed in the OFCA over the course of ACS' operations also included general refuse, drums, still bottoms and incinerator ash. According to the ACS, Inc. owner/operator, drums placed in the OFCA were crushed or punctured as part of the disposal process.

During the mid-1960s, an estimated 400 drums of sludge and semi-solids were landfilled in the ONCA. No intact drums were observed in test pits excavated in 1993. Residual wastes and rinse waters from the Kapica/Pazmey drum reconditioning operation were discharged to the ground in the Kapica/Pazmey Area.

1.2.4. Administrative History

In February 1980, the U.S. EPA performed a Preliminary Assessment of the ACS Site, and collected samples in the Off-Site Containment Area and at the Griffith Municipal Landfill in May 1980. The U.S. EPA performed a site inspection on September 9, 1980. In July 1982, U.S. EPA contractors installed four monitoring wells near the Off-Site Containment Area and the Griffith Landfill. Based on information developed during these investigative efforts, U.S. EPA assigned a hazard ranking system score of 34.98 to the ACS Site in June 1983.

In 1986, a group of approximately 125 Potentially Responsible Parties (PRPs) formed a Steering Committee to conduct the Remedial Investigation/Feasibility Study (RI/FS) pursuant to an agreement with the U.S. EPA. The PRPs signed a Consent Order to perform the RI/FS in June 1988. Following U.S. EPA approval of the RI/FS Work Plan, the field investigation for Phase I of the RI began in July 1989. Phase II RI field work began in March 1990, and in December 1990, the Phase III RI field work was initiated. The RI report was completed in June 1991. Warzyn (now Montgomery Watson Americas, Inc.) completed the FS report in June 1992.

In June 1992, the U.S. EPA published notice of its Proposed Plan for Remedial Action for the ACS Site. The remedy presented in that Proposed Plan was described by U.S. EPA as a modification of Remedial Alternative 6B from the FS. The U.S. EPA issued the ROD for the Site in September 1992. The Unilateral Administrative Order (UAO) was issued on September 30, 1994. The Respondents provided notice to the U.S. EPA of their intent to comply with the UAO, and have complied with the schedule in the UAO for developing planning documents and performing other required tasks to date.

1.3 PAST DATA COLLECTION ACTIVITIES

For information regarding past data collection activities at the Site, refer to the RI report (Warzyn, 1991).

1.4 CURRENT STATUS

The Site is currently in the RD/RA stage, and the purpose of the field activities discussed in this QAPP is to determine the effectiveness of selected technologies and to establish design parameters.

1.5 PROJECT OBJECTIVES AND SCOPE

Three pilot studies and one treatability study will be conducted as part of the Pre-Design Activities. These studies include:

1. ISVE pilot study on buried waste material in the On-Site Area

2. ISVE/air sparging pilot study on contaminated soil in the ONCA

3. Treatability testing of LTTT on buried waste in the OFCA, including a bench-scale LTTT and a pre-treatment/material handling pilot study.

The overall objective is to evaluate the effectiveness of these three treatment alternatives in removing contaminants from the subsurface. Table 1-2 defines the specific objectives for each pilot and treatability study and their respective sub-tasks, the data quality objectives, data that will be collected, and the uses of these data.

1.6 SAMPLE NETWORK DESIGN AND RATIONALE

The specific sample locations, types and frequencies of samples, and test designs are provided in test procedures presented in Appendix A—ISVE Pilot Study on Buried Wastes in the On-Site Area, Appendix B—ISVE/Air Sparging Pilot Study on

Contaminated Soils in the ONCA, Appendix C—Material Handling Pilot Study in the OFCA, and Appendix D—LTTT Treatability Study on Buried Waste in the OFCA.

1.7 PROJECT SCHEDULE

The project schedule is shown in Figure 1-1. The schedule for remedial design (Tasks 4 through 9) of the LTTT portion of the remedy is based on the deliverables identified in the Scope of Work for Remedial Design and Remedial Action prepared by the U.S. EPA. The list of deliverables and sequencing does not match the requirements for a performance-based procurement of services which is usually the preferred and most cost-effective manner for completing on-site thermal treatment remedies. The duration of the attached schedule could be reduced significantly by combining SOW deliverables and minimizing review and comment cycles.

TABLE 1-1

CLEANUP CRITERIA FOR AMERICAN CHEMICAL SERVICE

Chemical Compound	Remediation Criteria	Basis	Corresponding Risk	
			Carcinogenic	Non-Carcinogenic
Groundwater—Organic Compounds (µg/l)				
Acetone	192 to 2,300	HI	NA	0.08 to 1.0
Benzene	5.0	MCL	6.5×10^{-7}	NA
bis-(2-Chloroethyl) ether	21	Risk	1.0×10^{-6}	NA
bis-(2-Ethylhexyl) phthalate	5.8	Risk	1.0×10^{-6}	NA
Branched alkanes	18 to 210	HI	NA	0.08 to 1.0
2-Butanone	2,000 to 24,000	HI	NA	0.08 to 1.0
Chloromethane	8.4	Risk	1.0×10^{-6}	NA
cis--1,2-Dichloroethene	28 to 330	HI	NA	0.08 to 1.0
Cyclic ketones	5.8	Risk	1.0×10^{-6}	NA
1,1-Dichloroethane	183 to 2,200	HI	NA	0.08 to 1.0
1,4-Dichlorobenzene	3.3	Risk	1.0×10^{-6}	NA
Dimethyl ethyl benzenes	21 to 250	HI	NA	0.08 to 1.0
Ethylbenzene	33 to 390	HI	NA	0.08 to 1.0
Isophorone	19	Risk	1.0×10^{-6}	NA
4-Methyl-2-pentanone	53 to 640	HI	NA	0.08 to 1.0
4-Methylphenol	142 to 1,700	HI	NA	0.08 to 1.0
Methylene chloride	5.0	MCL	5.4×10^{-7}	NA
Non-cyclic acids	23 to 280	HI	NA	0.08 to 1.0
Pentachlorophenol	1.0	MCL	1.5×10^{-6}	NA
Polychlorinated biphenyls	0.06	Risk	1.0×10^{-6}	NA
Tetrachloroethene	5.0	MCL	6.2×10^{-7}	NA
Trichloroethene	5.0	MCL	2.1×10^{-7}	NA
Vinyl chloride	0.25	Risk	1.0×10^{-6}	NA
Groundwater—Metals (µg/l)				
Arsenic	8.8	Risk	1.0×10^{-6}	<0.1
Beryllium	0.02	Risk	1.0×10^{-6}	NA
Manganese	275 to 3,300	HI	NA	0.08 to 1.0
Thallium	0.2 to 2.4	HI	NA	0.08 to 1.0
Soil—Organic Compounds (mg/kg)				
4,4'-DDD	0.12	Risk	1.0×10^{-6}	NA
4,4'-DDE	0.16	Risk	1.0×10^{-6}	NA
4,4'-DDT	0.088	Risk	1.0×10^{-6}	NA

TABLE 1-1

CLEANUP CRITERIA FOR AMERICAN CHEMICAL SERVICE

Chemical	Remediation Criteria	Basis	Corresponding Risk	
			Carcinogenic	Non-Carcinogenic
Soil—Organic Compounds (mg/kg), (continued)				
4-Methyl-2-pentanone	21 to 630	HI	NA	0.03 to 1.0
Acetone	80 to 2,400	HI	NA	0.03 to 1.0
Aldrin	0.002	Risk	1.0×10^{-6}	NA
alpha-BHC	0.0047	Risk	1.0×10^{-6}	NA
Benzene	1.0	Risk	1.0×10^{-6}	NA
beta-BHC	0.016	Risk	1.0×10^{-6}	NA
bis(2-Chloroethyl) ether	0.027	Risk	1.0×10^{-6}	NA
bis-(2-Ethylhexyl)phthalate	1.1	Risk	1.0×10^{-6}	NA
Branched alkanes	26 to 770	HI	NA	0.03 to 1.0
2-Butanone	21 to 620	HI	NA	0.03 to 1.0
Carbon tetrachloride	0.38	Risk	1.0×10^{-6}	NA
Chlorobenzene	5 to 150	HI	NA	0.03 to 1.0
Chloroethane	90 to 2,700	HI	NA	0.03 to 1.0
Chloroform	9.5	Risk	1.0×10^{-6}	NA
1,4-Dichlorobenzene	2.4	Risk	1.0×10^{-6}	NA
1,2-Dichloroethane	0.64	Risk	1.0×10^{-6}	NA
1,1-Dichloroethene	0.098	Risk	1.0×10^{-6}	NA
cis-1,2-Dichloroethene	8.3 to 250	HI	NA	0.03 to 1.0
1,2-Dichloropropane	0.42	Risk	1.0×10^{-6}	NA
2,4-Dinitrotoluene	0.044	Risk	1.0×10^{-6}	NA
2,6-Dinitrotoluene	0.044	Risk	1.0×10^{-6}	NA
Cyclic ketones	7.3	Risk	1.0×10^{-6}	NA
Cyclic polyaromatic hydrocarbons	0.0026	Risk	1.0×10^{-6}	NA
Di-n-butylphthalate	77 to 2,300	HI	NA	0.03 to 1.0
Diethyl benzenes	43 to 1,300	HI	NA	0.03 to 1.0
Dimethyl ethyl benzenes	43 to 1,300	HI	NA	0.03 to 1.0
Endosulfan I	0.02 to 0.63	HI	NA	0.03 to 1.0
Ethyl methyl benzenes	163 to 4,900	HI	NA	0.03 to 1.0
Ethylbenzene	43 to 1,300	HI	NA	0.03 to 1.0
gamma-BHC (Lindane)	0.046	Risk	1.0×10^{-6}	NA
Halogenated alkanes	77 to 2,300	HI	NA	0.03 to 1.0
Heptachlor epoxide	0.0033	Risk	1.0×10^{-6}	NA

TABLE 1-1

CLEANUP CRITERIA FOR AMERICAN CHEMICAL SERVICE

Chemical	Remediation Criteria	Basis	Corresponding Risk	
			Carcinogenic	Non-Carcinogenic
Soil—Organic Compounds (mg/kg), (continued)				
Hexachlorobenzene	0.018	Risk	1.0×10^{-6}	NA
Hexachlorobutadiene	0.36	Risk	1.0×10^{-6}	NA
Isophorone	7.2	Risk	1.0×10^{-6}	NA
Methyl propyl benzenes	16 to 490	HI	NA	0.03 to 1.0
Methylated naphthalenes	3 to 85	HI	NA	0.03 to 1.0
Methylene chloride	6.2	Risk	1.0×10^{-6}	NA
n-chain Alkanes	25 to 760	HI	NA	0.03 to 1.0
n-Nitrosodiphenylamine	12.0	Risk	1.0×10^{-6}	NA
Naphthalene	3 to 82	HI	NA	0.03 to 1.0
Nitrogenated benzenes	0.2 to 6.2	HI	NA	0.03 to 1.0
Non-cylclic acids	33 to 1,000	HI	NA	0.03 to 1.0
Oxygenated benzenes	40 to 1,200	HI	NA	0.03 to 1.0
Pentachlorophenol	0.43	Risk	1.0×10^{-6}	NA
Propenyl benzenes	11 to 320	HI	NA	0.03 to 1.0
Styrene	1.7	Risk	1.0×10^{-6}	NA
1,2,4-Trichlorobenzene	0.5 to 16	HI	NA	0.03 to 1.0
1,1,1-Tetrachloroethane	77 to 2,300	HI	NA	0.03 to 1.0
1,1,2,2-Tetrachloroethane	0.28	Risk	1.0×10^{-6}	NA
Tetrachloroethene	1.1	Risk	1.0×10^{-6}	NA
1,1,2-Trichloroethane	0.51	Risk	1.0×10^{-6}	NA
Toluene	167 to 5,000	HI	NA	0.03 to 1.0
Trichloroethene	5.3	Risk	1.0×10^{-6}	NA
Vinyl chloride	0.031	Risk	1.0×10^{-6}	NA
Xylenes (mixed)	867 to 26,000	HI	NA	0.03 to 1.0
Soil—Metals (mg/kg)				
Antimony	0.5 to 15	HI	NA	0.03 to 1.0
Barium	87 to 2,600	HI	NA	0.03 to 1.0
Cadmium	2 to 51	HI	NA	0.03 to 1.0
Hexavalent chromium	47 to 1,400	HI	NA	0.03 to 1.0

HI Hazard index
NA Not applicable
mg/kg milligrams per killogram
µg/l micrograms per liter

(a) Corresponding risk indicates the risk from compounds at the concentration specified in the Remediation Criteria as presented in the ROD (1992).

TABLE 1-2

DATA QUALITY OBJECTIVES FOR THE ACS ISVE AND ISVE/AIR SPARGING PILOT STUDIES AND THE LOW TEMPERATURE THERMAL DESORPTION TREATABILITY STUDY

Pilot/Treatability Study	Data Quality Objectives	Task	Task Description	Data (Method)	Data Type	Data Uses
On-Site Area ISVE Pilot Study	An ISVE pilot study will be conducted in a 30 by 30 foot dewatered test cell in the On-Site Area to assess whether ISVE will remove sufficient VOC and SVOC contaminant mass to meet the performance standards specified in the ACS ROD, UAO, and SOW.	Pre-Test: System Installation and Soil Sampling	The ISVE test cell will be constructed by enclosing an approximately 30x30x25 foot area with a physical barrier (either sheet pile or slurry wall). One 2-inch piezometer, PZ501, and three 1-inch piezometers, PZ502 - PZ504, will be installed in the test cell. During drilling, each piezometer boring will be continuously logged for lithologies. A pump will be installed in Piezometer PZ501 to dewater the test cell. Piezometers PZ502 - PZ504 will be used to monitor ground-water elevation during the pilot study. Purge water from Piezometer PZ501 will be stored in a tank on site and sampled prior to disposal or on-site treatment.	USCS lithologies Groundwater elevation (feet above NGVD) SW-846 8260A (VOCs) purge water SW-846 8270B (SVOCs) purge water SW-846 8081 (Pesticides/PCBs) purge water SW-846 8151 (Pentachlorophenol) purge water Metals[a] (SW-846 6010A) purge water	A A B B B B B	D D D D D D D
			Two SVE well borings, VEW1 and VEW2, will be drilled to approximately 11 feet and 21 feet bgs, respectively. Each boring will be continuously sampled for lithologies. One soil/waste sample from each boring will be collected during drilling to assess vertical contaminant distribution and provide pre-test baseline data for technology evaluation. One soil sample will be collected from 5 feet bgs to assess shallow soil contamination and the other will be collected from 15 feet bgs to assess deeper soil contamination. A pump will be installed in SVE well VEW2 and along with Piezometer PZ501 will be used to dewater the site. Purge water from VEW2 will be combined with effluent from PZ501.	USCS lithologies VOCs (SW-846 8260A) SVOCs (SW-846 8270B) Pesticides/PCBs (SW-846 8081) Metals[b] (SW-846 6010A) Hexavalent Chrome (SW-846 7196) TOC (SW-846 9060) TKN (EPA/600 351.2) o-Phosphate (EPA/600 365.2) pH (field measurement) Heterotrophic Plate Count Soil particle size distribution (ASTM D-422) Moisture content	C C C C C C B B B A A A A	TA, D TA, D TA, D D D D BA BA BA BA BA D D
			Four soil vapor probe borings (VP1 - VP4) will be drilled to approximately 18 feet bgs. The borings will be continuously sampled for lithologies. Two soil waste samples per boring will be collected during drilling to assess vertical contaminant distribution and provide pre-test baseline data for technology evaluation. One soil sample will be collected from 5 feet bgs and the other from 15 feet bgs. Three nested soil vapor probes will be installed in each boring at depths of 7, 12, and 17 feet bgs. The letters A, B, and C will be used to identify the relative depth of each probe.	USCS lithologies VOCs (SW-846 8260A) SVOCs (SW-846 8270B) Pesticides/PCBs (SW-846 8081) Metals[b] (SW-846 6010A) Hexavalent Chrome (SW-846 7196) TOC (SW-846 9060) TKN (EPA/600 351.2) o-Phosphate (EPA/600 365.2) pH (field measurement) Heterotrophic plate count Soil particle size distribution (ASTM D-422) Moisture content	A C C C C C B B B A A A A	D TA, D TA, D TA, D D D BA BA BA BA BA D D
		Meteorological Measurements	A weather station will be set up at the test site to monitor weather conditions to assess the impact on meter calibration and soil vapor data.	Ambient air temperature Relative humidity Barometric pressure Wind speed and direction Precipitation	A A A A A	TA, D TA, D TA, D TA, D TA, D
		Pre-Test: Soil Vapor Sampling	Soil vapor samples will be collected from both extraction wells and each soil vapor probe prior to system start up to provide baseline data for biological activity evaluation.	Carbon dioxide Oxygen Total volatile hydrocarbons	A A A	D, BA D, BA D, BA
		Pre-Test: Initial Soil Vapor Sampling	Soil vapor samples will be collected from each SVE well and soil vapor probe after the first soil vapor purge volume has been extracted to provide baseline data for technology evaluation.	SVOCs (TO-13) VOCs (TO-14) Ethane and Methane (TO-3)	B B B	TA, D TA, D TA, D
		Short-Term Variable Flow Rate Test	A short term test will be conducted to determine system operation parameters. During the short term test vacuum measurements and flow rate measurements will be collected until the vacuum stabilizes.	Vacuum (inches of mercury) Flow rate (ft^3/minute)	A A	D D

TABLE 1-2

DATA QUALITY OBJECTIVES FOR THE ACS ISVE AND ISVE/AIR SPARGING PILOT STUDIES AND THE LOW TEMPERATURE THERMAL DESORPTION TREATABILITY STUDY

Pilot/Treatability Study	Data Quality Objectives	Task	Task Description	Data (Method)	Data Type	Data Uses
On-Site Area ISVE Pilot Study (con't)		Long-Term Constant Flow Rate Test:	A long-term constant flow rate test will be conducted based on the results of the short-term test. During this test the system operation parameters will be monitored, soil vapor samples for TVH will be collected from the extraction well manifold and the vapor treatment off-gas system at 8-hour intervals during the first week of operation, 12-hour intervals during the second week of operation, and daily for the remainder of the pilot study. Soil vapor samples will be collected from the sampling port at the manifold and off-gas samples will be collected from the vapor treatment system for contaminant analysis every two days during the first week of operation, two times per week during the second week of operation, weekly during the third through the sixth week of operation, and monthly thereafter.	Vacuum (inches of mercury) Flow rate (ft^3/minute) Total volatile hydrocarbons Condensate volume VOCs (TO-14) SVOCs (TO-13) Groundwater elevation (feet above NGVD)	A A A A B B A	D, BA D, BA D, BA D TA, D TA, D D
		Condensate Sampling	Condensate samples will be collected from the air/water separator on a bi-weekly basis for the duration of the ISVE system operation to evaluate the type and concentration of contaminants in the condensate.	SW-846 8260A (VOCs) SW-846 8270B (SVOC) SW-846 8081 (Pesticides/PCBs) SW-846 8151 (Pentachlorophenol) Metals[a] (SW-846 6010A)	B B B B B	D D D D D
		Post Test: Soil Vapor Sampling	After the long-term ISVE test is complete, a 48 hour test will be conducted to assess equilibration of the test area. Soil vapor samples will be collected at each vapor probe and each SVE extraction well immediately after shut down, 1/2 hour, and at 1, 2, 4, 8, 12, 24, and 48 hours after shut down.	Carbon dioxide Oxygen Total volatile hydrocarbons	A A A	D, BA D, BA D, BA
			Within 48 hours after the long-term term constant rate test, post-test soil vapor samples will be collected from each soil vapor probe and each extraction well.	SVOCs (TO-13) VOCs (TO-14) Ethane and Methane (TO-3)	B B	TA, D TA, D
		Post-Test: Soil Sampling	Within two weeks of the conclusion of the constant - rate test, five soil borings, SB501 - SB505, will be drilled and sampled for technology evaluation. A boring will be located next to each soil vapor probe and one next to the SVE wells.	VOCs (SW-846 8260A) SVOC (SW-846 8270B) Pesticides/PCBs (SW-846 8081) Metals (SW-846 6010A)[b]	C C C C	TA, D TA, D TA, D D
			Two soil waste samples per boring will be collected from the same depth intervals from which the pre-test soil samples were collected.	Hexavalent Chrome (SW-846 7196) TOC (SW-846 9060) TKN (EPA/600 351.2) o-Phosphate (EPA/600 365.2) pH (field measurement) Heterotrophic plate count Soil particle size distribution (ASTM D-422)	B B B B A A A	D BA BA BA BA BA D, BA
ONCA ISVE/Air Sparging Pilot Study	An ISVE/air sparging pilot study will be conducted in the ONCA to assess whether this technology will remove sufficient contaminant mass to meet the performance standards specified in the ACS ROD, UAO, and SOW.	Pre-Test: System Installation and Soil Sampling	Four monitoring well (MW501 - MW504) and two air injection well (AS1 and AS2) borings will be drilled to approximately 20 feet bgs. Each boring will be continuously logged for lithologies and two soil samples per boring will be collected to assess vertical contaminant distribution and to provide baseline data for technology evaluation. One soil sample will be collected 1 foot bgs (above the water table) and the other will be collected 10 feet bgs (below the water table). In addition to the monitoring well, one soil vapor probe (VP5 - VP8) will be installed in each monitoring well boring. Horizontal SVE wells will be installed in 60 foot x18 inch x 2 foot trenches. Soil samples will not be collected during trench excavation.	USCS lithologies VOCs (SW-846 8260A) SVOCs (SW-846 8270B) Pesticides/PCBs (SW-846 8081) Metals[b] (SW-846 6010A) Hexavalent Chrome (SW-846 7196) TOC (SW-846 9060) TKN (EPA/600 351.2) ortho-Phosphate (EPA/600 365.2) pH (field measurement) Heterotrophic plate count Soil particle size distribution (ASTM D-422) Groundwater elevation (feet above NGVD) Moisture Content	A C C C C C B B B B A A A A	D TA, D TA, D TA, D D D BA BA BA BA BA D D D

TABLE 1-2

DATA QUALITY OBJECTIVES FOR THE ACS ISVE AND ISVE/AIR SPARGING PILOT STUDIES
AND THE LOW TEMPERATURE THERMAL DESORPTION TREATABILITY STUDY

Pilot/Treatability Study	Data Quality Objectives	Task	Task Description	Data (Method)	Data Type	Data Uses
ONCA ISVE/Air Sparging Pilot Study (con't)		Pre-Test: System Installation and Soil Sampling (con't)	Three piezometers, P505- P507 will be installed to monitor groundwater elevation near the horizontal SVE wells during the pilot test. During drilling, each piezometer boring will be continuously sampled for lithologies.	USCS lithologies	A	D
				Ground-water elevation (feet above NGVD)	A	D
		Meteorological Measurements	A weather station will be set up at the test site to monitor weather conditions to assess the impact on meter calibration and soil vapor data.	Ambient air temperature	A	TA, D
				Relative humidity	A	TA, D
				Barometric pressure	A	TA, D
				Wind speed and direction	A	TA, D
				Precipitation	A	TA, D
		Pre-Test Groundwater Sampling	After the monitoring wells are installed and developed groundwater samples will be collected from each well to provide baseline data for technology evaluation.	VOCs (SW-846 8260A)	C	TA, D
				SVOCs (SW-846 8270B)	C	TA, D
				Pesticides/PCBs (SW-846 8081)	C	TA, D
				Pentachlorophenol (SW-846 8151)	C	TA, D
				Metals (SW-846 6010A)[a]	C	D
				Cations[c] (SW-846 6010A)	B	BA
				Hexavalent Chrome (SW-846 7196)	C	D
				TOC (415.1)	B	BA
				Chloride (EPA/600 325.2)	B	BA
				Fluoride (EPA/600 340.2)	B	BA
				Sulfate (EPA/600 375.4)	B	BA
				Sulfide (EPA/600 376.1)	B	BA
				Nitrate/Nitrite (EPA/600 353.2)	B	BA
				Ammonia (EPA /600 350.3)	B	BA
				ortho-Phosphate (EPA/600 365.2)	B	BA
				Heterotrophic Plate Count	A	BA
		ISVE Pre-Test Soil Vapor Sampling	Soil vapor samples will be collected from the extraction trench manifold and soil vapor probes prior to start up of the ISVE system to provide baseline data for biological activity evaluation.	Carbon dioxide	A	D, BA
				Oxygen	A	D, BA
				Total volatile hydrocarbons	A	D, BA
		ISVE Initial Vapor Sampling	Soil vapor samples will be collected from the sampling port on the extraction trench manifold and from each soil vapor probe after the first soil vapor purge volume has been extracted to provide baseline data.	SVOCs (TO-13)	B	TA, D
				VOCs (TO-14)	B	TA, D
				Ethane and Methane	B	TA, D
		ISVE Short-Term Variable Flow Rate Test	A short-term variable flow rate test will be conducted to determine system operation parameters. During the short-term test system operation parameters will be monitored.	Vacuum (inches of mercury)	A	D, BA
				Flow rate (ft^3/minute)	A	D, BA
		ISVE Two-Week Constant Rate Test	A two-week constant flow rate test will be conducted using the operation parameters from the short-term test. During this test, groundwater elevations will be measured in the piezometers and monitoring wells, soil vapor samples for system parameter assessment will be collected every 8 hours the first week and every 12 hours the second week, condensate samples for contaminant concentration assessment will be collected on a weekly basis, soil vapor samples for VOCs and SVOCs analysis will be collected from the extraction trench manifold every two days the first week of the test and twice the second week of the test, and off-gas samples for emissions evaluation will be collected from the vapor treatment unit on a bi-weekly basis.	Vacuum (inches of mercury)	A	D
				Flow rate (ft^3/meter)	A	D
				Groundwater elevation (feet above NGVD)	A	D
				Total volatile hydrocarbons	A	D, BA
				Condensate volume	A	D
				VOCs (SW-846 8260A)	C	D
				SVOC (SW-846 8270B)	C	D
				Pesticides/PCBs (SW-846 8081)	C	D
				Pentachlorophenol (SW-846 8151)	C	D
				Metals (SW-846 6010A)[a]	C	D
				SVOCs (TO-13)	B	D, VE
				VOCs (TO-14)	B	D, VE
		Air Sparging Short-Term Variable Flow Rate Test	A short-term variable flow rate test will be conducted to determine system operation parameters. This test will be conducted until the system stabilizes at each flow step. Flow rates will be measured at Air Sparge Wells AS1 and AS2 and the blower.	Pressure (inches of mercury)	B	D
				Flow rate (ft^3/minute)	B	D

TABLE 1-2

DATA QUALITY OBJECTIVES FOR THE ACS ISVE AND ISVE/AIR SPARGING PILOT STUDIES AND THE LOW TEMPERATURE THERMAL DESORPTION TREATABILITY STUDY

Pilot/Treatability Study	Data Quality Objectives	Task	Task Description	Data (Method)	Data Type	Data Uses
ONCA ISVE/Air Sparging Pilot Study (con't)		Air Sparging Two-Week Constant Rate Test	A two-week constant flow rate test will be conducted using the operation parameters from the short-term test. During this test, groundwater elevations will be measured in the piezometers and monitoring wells and system operation parameters will be monitored.	Vacuum (inches of mercury)	A	D
				Flow rate (ft^3/meter)	A	D
				Groundwater elevation (feet above NGVD)	A	D
				Total volatile hydrocarbons	A	D, BA
		ISVE/Air Sparging Two Week Constant Rate Test	The two week constant-rate test will be based on the operation parameters from the short-term tests. During this test, groundwater elevation will be monitored; samples from the extraction trench manifold and off-gas from the vapor treatment system will be collected for operating parameters every 8 hours the first week of the test and every 12 hours during the second week of the test; and soil vapor samples from the extraction trench manifold and off-gas from the vapor treatment system will be collected for VOC and SVOC analysis every two days the first week of the test and twice during the second week of the test.	Vacuum (inches of mercury)	A	D
				Flow rate (ft^3/minute)	A	D
				Groundwater elevation (feet above NGVD)	A	D
				Total volatile hydrocarbons	A	D, BA
				SVOCs (TO-13)	B	D, VE
				VOCs (TO-14)	B	D, VE
		ISVE/Air Sparging Condensate Sampling	Condensate samples will be collected from the air/water separator on a weekly basis for the duration of the ISVE/air sparging system operation.	SW-846 8260A (VOCs)	B	D
				SW-846 8270B (SVOC)	B	D
				SW-846 8081 (Pesticides/PCBs)	B	D
				SW-846 8151 (Pentachlorophenol)	B	D
				Metals (SW-846 6010A)[a]	B	D
		Post-Test: Soil Vapor Sampling	After the 2-week constant rate ISVE/air sparging test, a 48 hour test will be conducted to assess post-test equilibration of the study area. Soil vapor samples will be collected at each vapor probe and the extraction trench manifold for biological activity evaluation at shutdown, 1/2 hour and 1, 2, 4, 8, 12, 24, and 48 hours after shut down.	Carbon dioxide	A	D, BA
				Oxygen	A	D, BA
				Total volatile hydrocarbons	A	D, BA
			Within 48 hours of the equilibration test, post-test soil vapor samples will be collected from each soil vapor probe and the extraction trench manifold for VOC, SVOC, ethane, and methane analysis.	SVOCs (TO-13)	B	TA, D
				VOCs (TO-14)	B	TA, D
				Ethane and Methane (TO-3)	B	TA, D
		Post-Test: Groundwater Sampling	Within 48 hours of the equilibration test, groundwater samples will be collected from each monitoring well for technology evaluation.	VOCs (SW-846 8260A)	C	TA, D
				SVOCs (SW-846 8270B)	C	TA, D
				Pesticides/PCBs (SW-846 8081)	C	TA, D
				Pentachlorophenol (SW-846 8151)	C	TA, D
				Metals (SW-846 6010A)[a]	C	D
				Cations (SW-846 6010A)	B	BA
				Hexavalent Chrome (SW-846 7196)	C	D
				TOC (415.1)	B	BA
				Chloride (EPA/600 325.2)	B	BA
				Fluoride (EPA/600 340.2)	B	BA
				Sulfate (EPA/600 375.4)	B	BA
				Sulfide (EPA/600 376.1)	B	BA
				Nitrate/Nitrite (EPA/600 353.2)	B	BA
				Ammonia (EPA /600 350.3)	B	BA
				ortho-Phosphate (EPA/600 365.2)	B	BA
				Heterotrophic Plate Count	A	BA

TABLE 1-2

DATA QUALITY OBJECTIVES FOR THE ACS ISVE AND ISVE/AIR SPARGING PILOT STUDIES AND THE LOW TEMPERATURE THERMAL DESORPTION TREATABILITY STUDY

Pilot/Treatability Study	Data Quality Objectives	Task	Task Description	Data (Method)	Data Type	Data Uses
ONCA ISVE/Air Sparging Pilot Study (con't)		Post Test: Soil Sampling	Within two weeks after the long-term test has ended, six soil borings, SB506 - SB511, will be drilled and sampled for technology evaluation. One boring will be located next to each monitoring well and air sparging well. Two soil samples per boring will be collected from the same depth intervals from which the pre-test soil samples were collected.	VOCs (SW-846 8260A)	C	TA, D
				SVOC (SW-846 8270B)	C	TA, D
				Pesticides/PCBs (SW-846 8081)	C	TA, D
				Metals (SW-846 6010A)[b]	C	D
				Hexavalent Chrome (SW-846 7196)	C	BA
				TOC (SW-846 9060)	B	BA
				TKN (EPA/600 351.2)	B	BA
				ortho-Phosphate (EPA/600 365.2)	B	BA
				pH (field measurement)	B	BA
				Heterotrophic Plate Count	A	BA
				Soil particle size distribution (ASTM D-422)	A	D
				Moisture Content	A	D
OFCA Pre-Treatment Materials Handling Treatability Study	The pre-treatment materials handling treatability study will be conducted to assess the type and quantity of debris in soils representative of the ACS site, to assess the ability of conventional materials handling equipment to effectively separate debris from soils to be thermally treated, determine the magnitude of the VOC fugitive emissions during excavation activities, and to collect soil samples for thermal treatability testing.	Soil Excavation	Five 20 x 20 test pits will be excavated. Material removed from each test pit will be screened for oversized reject material and soil. The oversized reject material will be weighed and the percentage of debris types will be estimated. The soil will be weighed and sampled for low temperature thermal treatment treatability testing.	Total tonnage per test pit	A	TA, D
				Total tonnage of soil per grid	A	TA, D
				Total tonnage of debris per grid	A	TA, D
				Percent debris per grid	A	TA, D
				Percent debris type per grid (i.e., metal)	A	TA, D
				Screening throughput in tons/hour	A	TA, D
				PCB Field Screening	A	TA
		Air Monitoring	During excavation and screening, ambient air samples will be collected upwind and downwind of material handling at eight-hour intervals on a daily basis. Meteorological conditions will be monitored with an on-site weather station.	VOCs (TO -14)	B	TA, D
				Ambient air temperatures	A	Information
				Humidity	A	Information
				Barometric pressure	A	Information
				Wind speed and direction	A	Information
OFCA Low Temperature Thermal Treatment (LTTT) Treatability Study	An LTTT treatability study will be conducted on the soils collected from the pre-treatment materials handling treatability study to assess whether this technology will remove sufficient VOC and SVOC contaminant mass to meet the performance standards specified in the ACS ROD, UAO, and SOW.	Pre-Treatment: Soil Analysis	Samples from soil collected during the pre-treatment materials handling study will be analyzed for VOCs. These samples will be collected as the soil is received by the thermal treatment laboratory to provide baseline VOC concentrations for technology evaluation and air emission assessment.	VOCs (SW-846 8260A)	C	TA, D
			After the VOC samples are collected, the soil will be prepared for treatment and soil samples will be collected to provide data for composite sample preparation.	VOCs (SW-846 8260A)	C	TA, D
				SVOCs (SW-846 8270B)	C	TA, D
				Pesticides/PCBs (SW-846 8081)	C	TA, D
			Based on the results of the prepared sample analyses, the soil will be batched to form two composite feed samples that represent worst-case and typical conditions. Soil from the composite feed will be analyzed to provide pre-treatment baseline data for technology evaluation.	VOCs (SW-846 8260A)	C	TA, D
				SVOCs (SW-846 8270B)	C	TA, D
				Pesticides/PCBs (SW-846 8081)	C	TA, D
				Dioxins/Furans (SW-846 8290)	B	TA, D
				Metals (SW-846 6010A)[d]	C	TA, D
				TCLP Metals (SW-846 1311/6010A/7000)	B	TA, D
				Ultimate/Proximate (ASTM D5373/D4239/E-422/D-5142)	A	TA, D
				Leachable chlorides (GLI G-40/ME-4)	A	TA, D
				Ash Fusability (ASTM D1857)	A	Information
				Soil pH (SW-846 9045)	A	Information
				Heating value (ASTMD-1989)	A	Information
				Particle size distribution (ASTM D-422)	A	Information
				Atterberg limits (ASTM D-4318)	A	Information
				Proctor density (ASTM D-698)	A	Information
				Soil classification (ASTM D-2487)	A	Information
		Tray Test	Aliquots of the composite soil samples will be thermally treated at temperatures from 700°F to 1,100°F. Post-treatment soil samples will be analyzed to determine operation criteria for the Rotary Thermal Apparatus Testing.	VOCs (SW-846 8260A)	C	TA, D
				SVOCs (SW-846 8270B)	C	TA, D
				Pesticides/PCBs (SW-846 8081)	C	TA, D
				Dioxins/Furans (SW-846 8290)	B	TA, D

TABLE 1-2

DATA QUALITY OBJECTIVES FOR THE ACS ISVE AND ISVE/AIR SPARGING PILOT STUDIES AND THE LOW TEMPERATURE THERMAL DESORPTION TREATABILITY STUDY

Pilot/Treatability Study	Data Quality Objectives	Task	Task Description	Data (Method)	Data Type	Data Uses
OFCA Low Temperature Thermal Treatment (LTTT) Treatability Study (con't)		Rotary Thermal Apparatus Testing	The composite soil samples will be thermally treated based upon the results of the Tray Tests. Post-treatment soil samples will be analyzed to provide data for technology evaluation.	VOCs (SW-846 8260A)	C	TA, D
				SVOCs (SW-846 8270B)	C	TA, D
				Pesticides/PCBs (SW-846 8081)	C	TA, D
				Dioxins/Furans (SW-846 8290)	B	TA, D
				Metals (SW-846 6010A)[d]	C	TA, D
				TCLP Metals (SW-846 1311/6010A/7000)	B	TA, D
				Ultimate/Proximate (ASTM D5373/D4239/E-422/D-5142)	A	TA, D
				Leachable chlorides (GLI G-40/ME-4)	A	TA, D
				Soil pH (SW-846 9045)	A	Information
			Off-gas purge samples will also be analyzed during the rotary thermal apparatus testing to provide data for technology evaluation. These samples consist of off-gas solubilized in water and in methanol and sorbed to XAD™ resin and to Tenex™ resin.	VOCs (SW-846 8260A)	B	Information
				SVOCs (SW-846 8270B)	B	Information
				Pesticides/PCBs (SW-846 8081)	B	Information
				Dioxins/Furans (SW-846 8290)	B	Information
				Acidity (EPA/600 310.1)	A	Information
				Water pH (EPA 150.1, SW-846 9040/9045)	A	Information
				Total sulfur (EPA/600 375.2/375.4/300)	A	Information
				Total chloride (EPA/600325.2/325.3)	A	Information

EPA Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW-846, 3rd Edition (EPA, 1992).
EPA Methods for Chemical Analysis of Water and Wastes, EPA Manual, 600/4-79-020 (USEPA, 1983, with additions).
Compendium of Methods for the Determination of Toxic Organic Compounds in Ambient Air, April 1994.

(a) Arsenic, beryllium, thallium, manganese
(b) Antimony, cadmium, barium, lead, phosphorous, potassium
(c) Calcium, iron, magnesium, phosphorous, potassium, sodium
(d) Beryllium, thallium, sodium, potassium, phosphorous

ACS American Chemical Service
BA Biodegradation assessment
bgs Below ground surface
ISVE In-situ soil vapor extraction
LTTT Low temperature thermal treatment
NGVD National geodetic vertical datum
OFCA Off-Site Containment Area
ONCA On-site Containment Area
PCB Polychlorinated biphenyl
ROD Record of Decision
SOW Statement of Work
SVE Soil vapor extraction
SVOC Semi-volatile organic compound
TA Technology assessment
TCLP Toxicity characteristic leaching procedure
TKN Total Kjeldahl nitrogen
TOC Total organic compound
UAO Unilateral Administrative Order
USCS Unified soil classification system
VE Air emission assessment
VOC Volatile organic compound

RCRA metals: Arsenic, barium, cadmium, chromium, lead, mercury, selenium, silver

A Field data, microbial data, and soil physical and chemical characteristic data
B SW-846 or EPA/600 methodologies and associated QC with standard data packages
C SW-846 methodologies and associated QC with CLP-like data packages
D Design

AMERICAN CHEMICAL SERVICES
SCHEDULE FOR PILOT AND TREATABILITY STUDIES

ID	Task Name	Duration
1		
2	Submittal of Work Plan, QAPP, FSPs	1 d
3	Agency Review for PMHS/LTTT	81 d
4	Partial approval for PMHS/LTTT	1 d
5	Agency Review for ISVE/air sparging	59 d
6	Revise QAPP/FSP	30 d
7	Agency Review	45 d
8	Agency Appproval of QAPP/FSP	1 d
9		
10	ISVE/AIR SPARGING	452 d
11	Task 1 ISVE pilot test on buried wastes	452 d
12	Task 1.1 Contracting	21 d
13	Task 1.2 Mobilization/setup	5 d
14	Task 1.3 Install test cell	14 d
15	Task 1.4 Install piezometers	2 d
16	Task 1.5 Dewater test cell	14 d
17	Task 1.6 Install ISVE and vapor points	5 d
18	Task 1.7 Construct ISVE system	14 d
19	Task 1.8 Pre-test vapor sampling	1 d
20	Task 1.9 Initial vapor sampling	1 d
21	Task 1.10 Short term variable flow rate tests	14 d
22	Task 1.11 Long term constant rate test	180 d
23	Task 1.12 Post-test vapor sampling	2 d
24	Task 1.13 Post-test soil/waste sampling	14 d
25	Task 1.14 Laboratory analysis	45 d
26	Task 1.15 Develop draft report (to agency)	35 d
27	Task 1.16 Agency review/comment report	60 d
28	Task 1.17 Revise/finalize report	60 d
29		
30	Task 2 ISVE/air sparging pilot test on contaminated soil	342 d
31	Task 2.1 Contracting	21 d
32	Task 2.2 Mobilization/setup	5 d
33	Task 2.3 Install trenches and piping	5 d
34	Task 2.4 Install wells and piezometers	5 d
35	Task 2.5 Construct ISVE/air sparging system	14 d
36	Task 2.6 Pre-test vapor sampling	1 d
37	Task 2.7 Initial vapor sampling	1 d
38	Task 2.8 Collect pre-test groundwater samples	2 d
39	Task 2.9 Variable rate ISVE test	14 d
40	Task 2.10 Constant rate ISVE test	14 d
41	Task 2.11 Short term variable rate air sparging test	14 d
42	Task 2.12 Constant rate air sparging test	14 d
43	Task 2.13 Long term constant rate ISVE/air sparge test	14 d
44	Task 2.14 Collect post-test vapors	2 d
45	Task 2.15 Collect post-test groundwater samples	2 d
46	Task 2.16 Collect post-test soil/waste samples	14 d
47	Task 2.17 Laboratory analysis	45 d
48	Task 2.18 Develop draft report (to agency)	35 d
49	Task 2.19 Agency review/comment report	60 d
50	Task 2.20 Revise/finalize report	60 d
51		
52	LOW TEMPERATURE THERMAL TREATMENT (LTTT)	302 d
53	Task 1 Pretreatment/Material Handling Study (PMHS)	186 d
54	Task 1.1 Contracting	15 d
55	Task 1.2 Mobilization/setup	2 d
56	Task 1.3 Background air sampling	2 d
57	Task 1.4 Test pit excavation/screening	10 d
58	Task 1.5 Decontamination/demobilation	2 d
59	Task 1.6 Develop draft report (to agency)	70 d
60	Task 1.7 Agency review/comment report	25 d
61	Task 1.8 Revise/finalize report	60 d
62		
63	Task 2 Thermal Treatability Study (TTS)	273 d
64	Task 2.1 Sample receipt/preparation	21 d
65	Task 2.2 Tray testing	30 d
66	Task 2.3 RTA testing	38 d
67	Task 2.4 Develop draft report (to agency)	70 d
68	Task 2.5 Agency review/comment report	45 d
69	Task 2.6 Revise/finalize report	60 d

Project: ACS PILOT AND TREATABILITY STUDIES
Project Number: 4677-0141

Task | Progress | Milestone | Summary | Rolled Up Task | Rolled Up Milestone | Rolled Up Progress

1 OF 2

FIGURE 1-1

Schedule is subject to USEPA approval of the work plan, QAPP, and FSP documents on the dates shown

AMERICAN CHEMICAL SERVICES
SCHEDULE FOR PILOT AND TREATABILITY STUDIES

ID	Task Name	Duration
1		
2	Submittal of Work Plan, QAPP, FSPs	1 d
3	Agency Review for PMHS/LTTT	81 d
4	Partial approval for PMHS/LTTT	1 d
5	Agency Review for ISVE/air sparging	59 d
6	Revise QAPP/FSP	30 d
7	Agency Review	45 d
8	Agency Appproval of QAPP/FSP	1 d
9		
10	ISVE/AIR SPARGING	452 d
11	Task 1 ISVE pilot test on buried wastes	452 d
12	Task 1.1 Contracting	21 d
13	Task 1.2 Mobilization/setup	5 d
14	Task 1.3 Install test cell	14 d
15	Task 1.4 Install piezometers	2 d
16	Task 1.5 Dewater test cell	14 d
17	Task 1.6 Install ISVE and vapor points	5 d
18	Task 1.7 Construct ISVE system	14 d
19	Task 1.8 Pre-test vapor sampling	1 d
20	Task 1.9 Initial vapor sampling	1 d
21	Task 1.10 Short term variable flow rate tests	14 d
22	Task 1.11 Long term constant rate test	180 d
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24	Task 1.13 Post-test soil/waste sampling	14 d
25	Task 1.14 Laboratory analysis	45 d
26	Task 1.15 Develop draft report (to agency)	35 d
27	Task 1.16 Agency review/comment report	60 d
28	Task 1.17 Revise/finalize report	60 d
29		
30	Task 2 ISVE/air sparging pilot test on contaminated soil	342 d
31	Task 2.1 Contracting	21 d
32	Task 2.2 Mobilization/setup	5 d
33	Task 2.3 Install trenches and piping	5 d
34	Task 2.4 Install wells and piezometers	5 d
35	Task 2.5 Construct ISVE/air sparging system	14 d
36	Task 2.6 Pre-test vapor sampling	1 d
37	Task 2.7 Initial vapor sampling	1 d
38	Task 2.8 Collect pre-test groundwater samples	2 d
39	Task 2.9 Variable rate ISVE test	14 d
40	Task 2.10 Constant rate ISVE test	14 d
41	Task 2.11 Short term variable rate air sparging test	14 d
42	Task 2.12 Constant rate air sparging test	14 d
43	Task 2.13 Long term constant rate ISVE/air sparge test	14 d
44	Task 2.14 Collect post-test vapors	2 d
45	Task 2.15 Collect post-test groundwater samples	2 d
46	Task 2.16 Collect post-test soil/waste samples	14 d
47	Task 2.17 Laboratory analysis	45 d
48	Task 2.18 Develop draft report (to agency)	35 d
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52	LOW TEMPERATURE THERMAL TREATMENT (LTTT)	302 d
53	Task 1 Pretreatment/Material Handling Study (PMHS)	186 d
54	Task 1.1 Contracting	15 d
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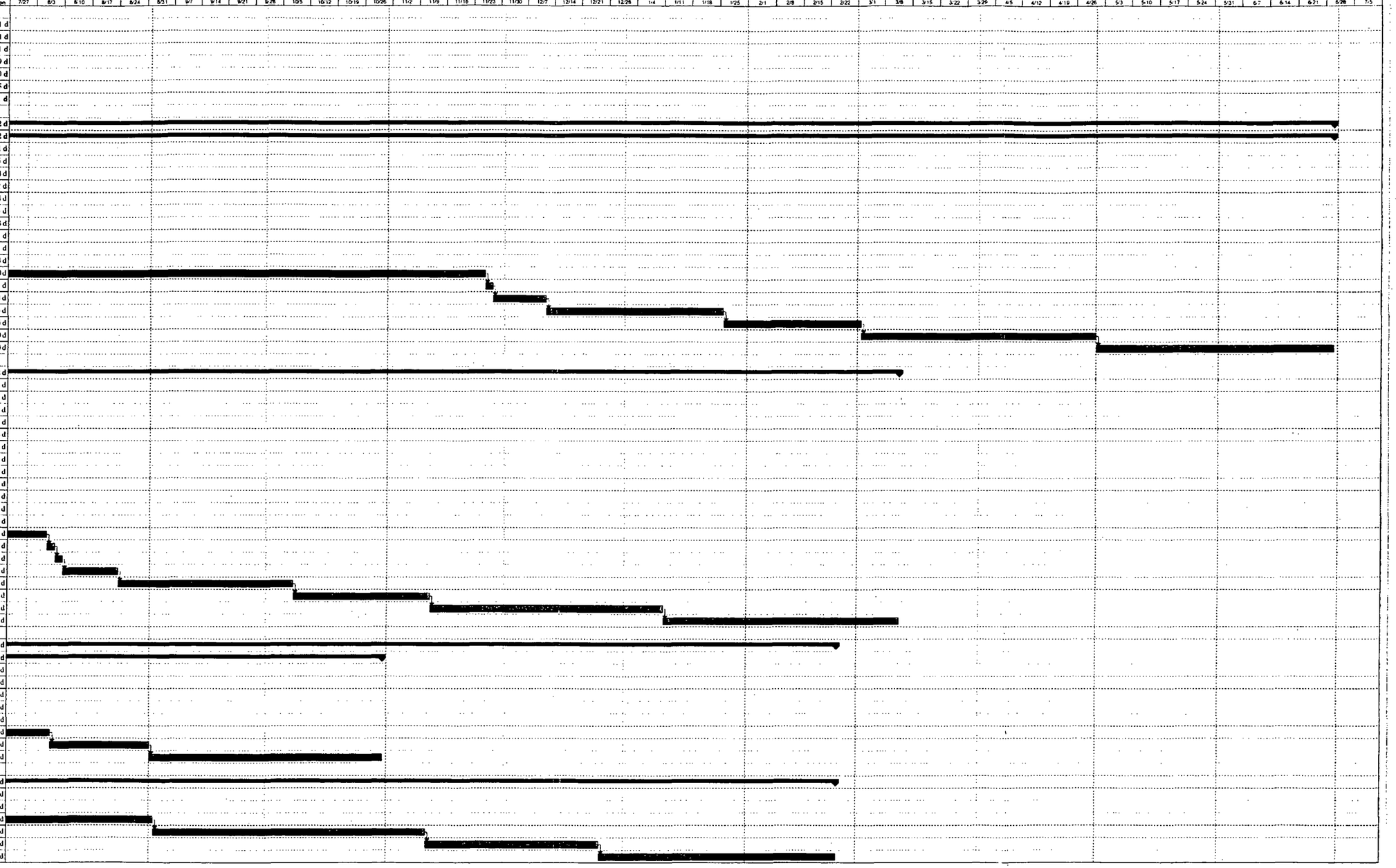
66	Task 2.3 RTA testing	38 d
67	Task 2.4 Develop draft report (to agency)	70 d
68	Task 2.5 Agency review/comment report	45 d
69	Task 2.6 Revise/finalize report	60 d



Project: ACS PILOT AND TREATABILITY STUDIES
Project Number: 4077.0191

Task | Progress | Milestone | Summary | Rolled Up Task | Rolled Up Milestone | Rolled Up Progress

FIGURE 1-1

Schedule is subject to USEPA approval of the work plan, QAPP, and FSP documents on the dates shown

2.0 PROJECT ORGANIZATION AND RESPONSIBILITY

At the direction of the ACS PRP Executive Committee and the approval of the U.S. EPA, Montgomery Watson has overall responsibility for the implementation and analysis of the ISVE, ISVE/Air Sparging, Pre-Treatment Materials Handling, pilot studies, and the LTTT treatability study. Montgomery Watson's responsibilities include preparing the QAPP and FSP, and conducting the pilot and treatability studies. Under the direction of the ACS PRP Executive Committee, Montgomery Watson also is responsible for project management. The various quality assurance and management responsibilities of key project personnel are defined below. A site-specific project organization chart is provided as Figure 2-1.

2.1 MANAGEMENT RESPONSIBILITIES

2.1.1. U.S. EPA Region V Project Manager

U.S. EPA Region V is the lead agency and is responsible for providing oversight of the ACS remedial design, which includes the ISVE, ISVE/Air Sparging, and Pre-Treatment Materials Handling, pilot studies, and the LTTT treatability study. The U.S. EPA Remedial Project Manager is Ms. Sheri Bianchin. Ms. Bianchin has overall responsibility for this project and also is responsible for coordinating communication between the U.S. EPA, IDEM, the ACS PRPs, and Montgomery Watson.

2.1.2. IDEM Project Manager

The IDEM Project Manager is Ms. Holly Gredja. Ms. Gredja is responsible for ensuring that the State of Indiana's environmental requirements are met.

2.1.3. ACS PRPs

The ACS PRPs are responsible for implementing the project and have the authority to obtain the resources necessary to meet the project objectives and requirements. The ACS PRPs are represented by Mr. Ron Frehner and Mr. Mark Travers. Mr. Frehner's primary function is to ensure that technical and scheduling objectives are successfully achieved.

Mr. Traver's primary function is to provide technical input and to manage the financial aspects of the project.

2.1.4. Montgomery Watson Project Manager

The Montgomery Watson Project Manager is Mr. Peter Vagt, Ph.D. Dr. Vagt has overall responsibility for ensuring that the U.S. EPA project objectives and Montgomery Watson's quality standards are met. In addition, he is responsible for technical quality control and project oversight, and provides the ACS PRPs with access to corporate management. Dr. Vagt's responsibilities include:

- Defining project objectives and developing a detailed work plan schedule
- Establishing project policy and procedures to address the specific needs of the project as a whole, as well as the objectives of each task
- Acquiring and applying technical and corporate resources as needed to ensure performance within budget and schedule constraints
- Orienting all field leaders and support staff concerning the project's special considerations
- Monitoring and directing the field leaders
- Developing and meeting ongoing project and/or task staffing requirements, including mechanisms to review and evaluate each task product
- Reviewing work performed on each task to ensure its quality, responsiveness, and timeliness
- Reviewing and analyzing overall task performance with respect to planned requirements and authorizations

- Approving all external reports (deliverables) before their submittal to the client and U.S. EPA

- Preparing and ensuring the quality of design and construction submittals

- Representing the project team at meetings and public hearings.

2.1.5. Focus Environmental Project Manager

The Pre-Treatment Materials Handling Pilot Study and LTTT treatability study will be managed by Focus Environmental of Knoxville, Tennessee under direct contract to Montgomery Watson. The Focus Environmental Project Manager is Mr. Paul Sadler. Mr. Sadler will provide technical quality control and project oversight for these studies. Mr. Sadler's responsibilities include:

- Coordinating with the Montgomery Watson Project Manager and other field personnel

- Defining the project objectives and developing a detailed work plan schedule

- Establishing project policies and procedures to address the specific needs of the project as a whole, as well as the objectives of each task

- Acquiring and applying technical and corporate resources as needed to ensure performance within budget and schedule constraints

- Orienting all field leaders and support staff concerning the special project considerations

- Developing and meeting ongoing project and/or task staffing requirements, including mechanisms to review and evaluate each task product

- Reviewing the work performed on each task to ensure its quality, responsiveness, and timeliness

- Reviewing and analyzing overall task performance with respect to planned requirements and authorizations

- Approving all reports (deliverables) before their submittal to Montgomery Watson

- Preparation of quality report submittals

- Representing the project team at meetings.

2.2 QUALITY ASSURANCE RESPONSIBILITIES

2.2.1. U.S. EPA Region V, Superfund Division, Technical Support Section (TSS) Quality Assurance Coordinator

The U.S. EPA Region V Quality Assurance Coordinator has the responsibility to review and approve this QAPP. Additional project responsibilities include:

- Conducting external Performance and System Audits of the laboratories and field activities

- Reviewing and evaluating analytical field and laboratory procedures.

2.2.2. Montgomery Watson Quality Assurance Officer

The Montgomery Watson Quality Assurance Officer (QAO) is Mr. Jack Kelly. Mr. Kelly has over 40 years of experience in the performance and management of environmental investigations at numerous sites, including CERCLA sites. As the QAO, Mr. Kelly will remain independent of direct project involvement and day-to-day operations. He is responsible for ensuring that the QA program defined in this QAPP is implemented. In addition, he will ensure that the project follows U.S. EPA, IDEM, and Montgomery Watson's policies and requirements. Mr. Kelly has direct access to

corporate executive staff as necessary to resolve any QA disputes that may arise. His specific responsibilities include:

- Conducting field QA audits
- Reviewing and approving QA plans and procedures
- Providing technical assistance to project staff for QA issues.

2.3 FIELD RESPONSIBILITIES

2.3.1. Montgomery Watson Field Team Leader

The Montgomery Watson field team leader is Mr. Dave Pieczynski. Mr. Pieczynski supports the Montgomery Watson Project Manager and is responsible for leading and coordinating the day-to-day activities of the project staff under his supervision. His responsibilities include:

- Day-to-day coordination with the project manager on technical issues in specific areas of expertise
- Development and implementation of field-related work plans, assurance of schedule compliance, and adherence to management-developed study requirements
- Coordination and management of field staff
- Implementation of QC for technical data provided by the field staff including field measurement data
- Adherence to work schedules provided by the project manager
- Generation, review, and approval of text and graphics required for field team efforts
- Coordination and oversight of technical efforts of subcontractors

- Identification of problems at the field-team level, discussion of resolutions with the project manager, and communication between the field team and upper management

- Participation in the preparation of project reports.

2.3.2. Montgomery Watson Technical Staff

The technical staff for this project will be selected from Montgomery Watson's pool of corporate resources. The technical staff will gather and analyze data, and prepare various task reports. All of the designated technical staff will be experienced professionals who possess the degree of specialization and technical competence required to effectively and efficiently perform the required work.

2.4 LABORATORY RESPONSIBILITIES

Analytical services for the pilot and treatability studies will be as follows:

- Analytical services for all soil, groundwater, and condensate chemical analysis (Data Types B and C) except for dioxin/furan analysis will be provided by:

  Industrial Environmental Analytical (IEA)
  300 Weston Parkway
  Cary, North Carolina 27513

- Analytical services for all dioxin/furan analysis will be provided by:

  Quanterra Environmental Services (formerly Enseco)
  880 Riverside Parkway
  West Sacramento, California 95605

- Analytical services for all air samples analysis (Data Type B) will be provided by:

  Quanterra Environmental Services
  1721 South Grand Avenue
  Santa Ana, California 92705

- Analytical services for Type A geotechnical parameters (soil particle size distribution) will be provided by:

  Montgomery Watson Soils Laboratory
  505 Science Drive, Suite C
  Madison, Wisconsin 53711

- Analytical services for all soil physical and chemical characteristic analysis for the LTTT will be provided by:

  Galbraith Laboratory
  2323 Sycamore Drive
  Knoxville, Tennessee 37921-1750

As described in Table 1-2, three data types (A, B, and C) will be collected for the pilot and treatability studies. Data Type A includes all data from methods that have limited QC or data validation requirements and data that will be used for informational purposes. Included in Data Type A are all field data, microbial population count data, and physical and chemical soil characteristic data from American Society of Testing and Materials (ASTM) methods. Data Type B includes all samples analyzed using EPA SW-846, EPA/600, or standard air methods with the respective associated QC. Standard data packages will be provided with Level B data and the data will be validated based on the results of QC sample analysis and other criteria (i.e., holding times). Data Type C includes samples analyzed using EPA SW-846 methods and the respective associated QC; however, the data reports will be consistent with Contract Laboratory Program (CLP) protocol (CLP-like) and the data will be validated in accordance with functional guidelines for data validation (U.S. EPA, 1994). The following sections focus on the laboratories that are providing Type B and C data.

2.4.1. IEA Laboratories

The IEA organization is a network of six integrated environmental laboratories located throughout the eastern United States, with corporate headquarters in Cary, North Carolina. The corporate Quality Assurance Program serves as an operational charter for the organization. It defines the purpose, organizational structure, and operating principles of the network and presents an overview of the key elements of the quality assurance

program. The QA program applies to the generation of analytical data at each of the IEA network lab locations utilized for environmental monitoring and assessment programs. In some cases more rigorous requirements may be applied at a particular laboratory based on its site QA program. Quality assurance at IEA is monitored at both the corporate and laboratory levels. IEA's network quality assurance program is led by the president of IEA. The QA program at each network laboratory is directed by the QA manager who monitors the day-to-day quality assurance activities of that facility, and reports directly to the laboratory's director and indirectly to the president. All scientists, analysts, and supervisory personnel play a vital role in ensuring the project-specific and general quality of the environmental analyses they perform.

Project Manager. Mr. Bill Scott is the IEA Project Manager for this project. Mr. Scott will serve as the primary IEA contact for Montgomery Watson and Focus Environmental, and will ensure that project requirements are met by the laboratory. He will be responsible for scheduling sample analyses and ensuring that results adhere to the contract and QAPP requirements. He will monitor the progress and timeliness of the work, review work orders and laboratory reports, and process any changes in the scope of work. Mr. Scott also will ensure that project-specific corrective action is taken when necessary to address problems identified by the QC results or QA audit findings. Mr. Scott will be responsible for approving final analytical reports prior to their submittal to Montgomery Watson.

IEA Quality Assurance Officer. Ms. Linda Mitchell is IEA-NC's Quality Assurance Manager. She is responsible for developing and administering the laboratory's standard operating procedures (SOPs), and developing and implementing the analytical portion of this QAPP. Her role includes preparing written documents that define QA/QC procedures, reviewing and approving laboratory QC procedures, supervising sample control operations, and oversight of interlaboratory testing programs and laboratory certifications. She also will be responsible for spot-checking data sets to ensure that appropriate QC measures have been taken and will evaluate the effectiveness of the laboratory QA/QC program through audits. She will report unacceptable findings to the Project Manager and Laboratory Manager for corrective action. Ms. Mitchell will sign the title page of this QAPP.

Sample Custodian. The IEA sample custodian reports to the laboratory and is responsible for:

- Receiving and inspecting the samples
- Recording information regarding sample condition and signing the appropriate forms
- Verifying the chain-of-custody and documenting any discrepancies
- Notifying the Project Manager and Laboratory Manager of sample receipt and inspection
- Assigning a unique identification number and customer number to each sample and logging it into the sample receiving log book.
- Transferring samples to the appropriate laboratory sections
- Controlling and monitoring access and storage of samples and extracts.

2.4.2. Quanterra Environmental Services (Quanterra)

The QA efforts at Quanterra are administered by the Director of QA, who reports directly to Quanterra's Chief Operating Officer. The QA Director has responsibility for establishing QC policies and procedures for all Quanterra facilities. Each laboratory facility has a QA manager who monitors the day-to-day QA activities of that facility and reports to the QA Director and to the laboratory manager. All scientists, analysts, and supervisory personnel are responsible for ensuring the project-specific and general quality of the environmental analyses they perform.

Project Manager. Ms. Sharon Meves is the Quanterra Project Manager for this project. Ms. Meves will serve as the primary Quanterra contact person for Montgomery Watson and will ensure that project requirements are met by the laboratory. She will be responsible for scheduling sample analysis and ensuring that results adhere to the contract

and QAPP requirements. She will monitor the progress and timeliness of the work, review work orders and laboratory reports, and process any changes in the scope of work. Ms. Meves also will ensure that project-specific corrective action is taken when necessary to address problems identified by the QC results or QA audit findings. Ms. Meves will be responsible for approving final analytical reports prior to submission to Montgomery Watson.

Project Quality Assurance Officer. Ms. Sevda Aleckson is the Project Quality Assurance Officer. She is responsible for seeing that project QA activities are performed in accordance with the requirements of the laboratory's QAPP. Ms. Aleckson's responsibilities include preparing QA documents that define QA/QC procedures, reviewing and approving laboratory QC procedures, and oversight of interlaboratory testing programs and laboratory certifications. She also is responsible for monitoring method operation through periodic data reviews and technical systems audits. She will report unacceptable findings to the Project Manager and Laboratory Manager for corrective action. Ms. Aleckson will sign the title page of this QAPP.

Sample Custodian. The Quanterra sample custodian reports to the Laboratory Manager and is responsible for:

- Receiving and inspecting the samples
- Recording information regarding sample condition on and signing the appropriate forms
- Verifying the chain-of-custody and documenting any discrepancies
- Notifying the Project Manager and Laboratory Manager of sample receipt and inspection
- Assigning a unique identification number and customer number to each sample and logging it into the sample receiving log book.
- Transferring samples to the appropriate laboratory sections
- Controlling and monitoring access and storage of samples and extracts.

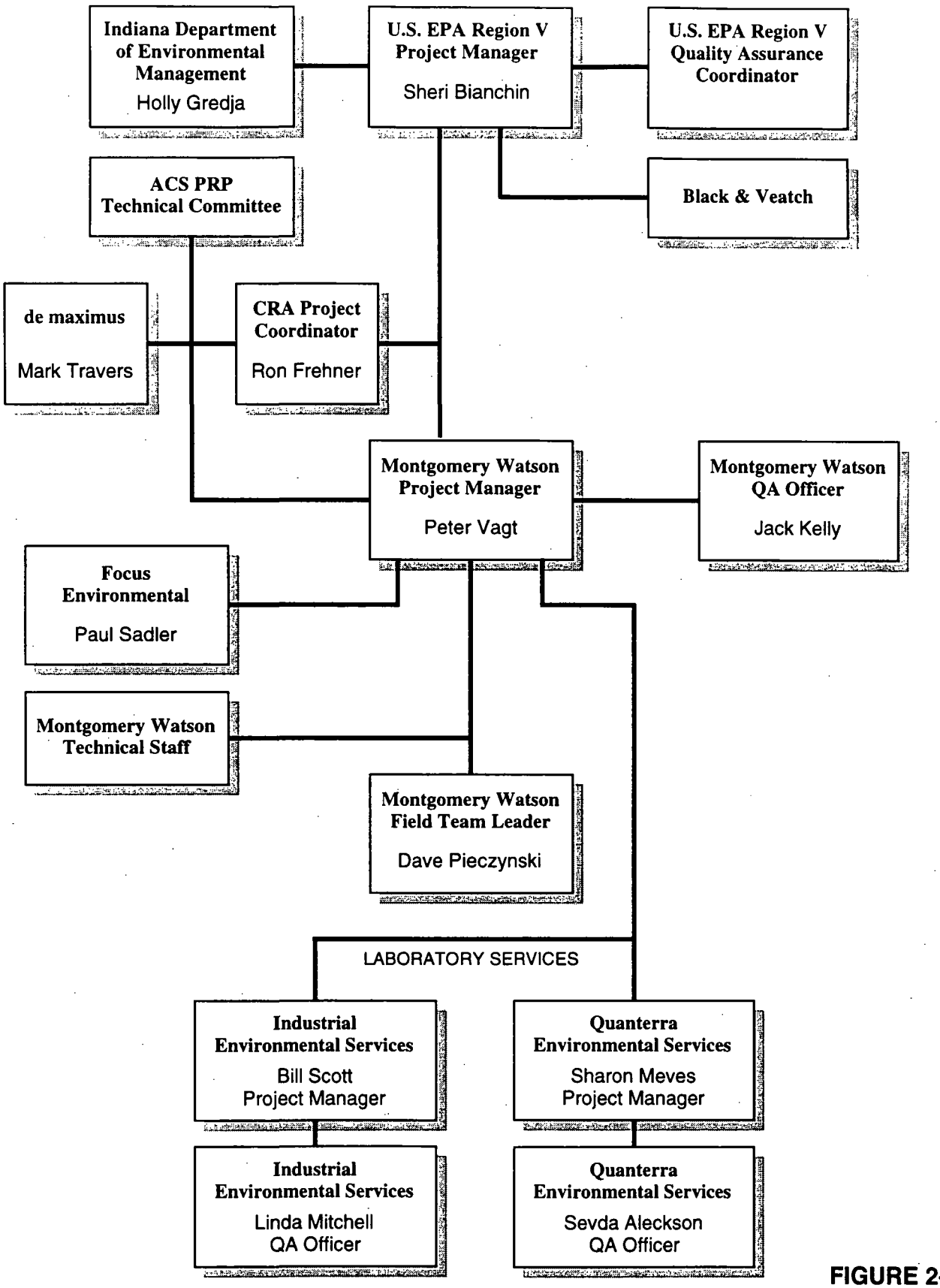


FIGURE 2-1

Developed By	Drawn By	**PROJECT ORGANIZATION CHART**	Drawing Number 4077.0191
Approved By	Date *8/17/96*	TREATABILITY STUDY AMERICAN CHEMICAL SERVICE, INC. NPL SITE GRIFFITH, INDIANA	**MONTGOMERY WATSON**
Reference			
Revisions			

QUALITY CONTROL | Graphic Standards ____ Lead Professional ____ | Technical Review ____ Project Manager ____ | Management Review ____ Other ____

This document has been developed for a specific application and may not be used without the written approval of Montgomery Watson.

3.0 QUALITY ASSURANCE OBJECTIVES FOR MEASUREMENT DATA

The overall QA objective for this project is to develop and implement procedures for field sampling, chain-of-custody, laboratory analysis, and data reporting that will provide results that meet the project DQOs and that are legally defensible. DQOs are qualitative and quantitative statements that specify the field and laboratory data quality necessary to support specific decisions or regulatory actions. The DQOs describe which data are needed, why the data are needed, and how the data will be used to meet the needs of the project. DQOs also establish numeric limits for the data to allow the data user (or reviewers) to determine whether the data collected are of sufficient quality for their intended use. The individual tasks for the pilot and treatability studies and their associated DQOs, the samples that will be collected for each task, the analyses types, the required data types, and the end use of the data are listed in Table 1-2. Specific procedures for sampling, chain-of-custody, laboratory instrument calibration, laboratory analysis, reporting of data, internal quality control, audits, preventive maintenance of field equipment, and corrective action are described in other sections of this QAPP.

3.1 DATA TYPES

The data types required for a project are based on the type of investigation, the project DQOs, the end use of the analytical data, and the level of documentation. Three data types will be collected during the ACS pilot and treatability studies. Data Type A includes data that will be used for informational purposes or for methods that have limited QC or data validation requirements. Included in Data Type A are all field data, microbial population data, and physical and chemical soil characteristic data from ASTM methods. Data Type B includes all samples analyzed using EPA SW-846, EPA/600, or standard air methods and the respective associated QC. Standard data packages will be provided with Level B data and the data will be validated based on QC sample results and other data validation criteria (i.e., holding times). Data Type C includes samples analyzed using EPA SW-846 methods and the respective associated QC, however, the data reports will be CLP-like. All Type C data will be validated in accordance with the functional guidelines for data validation (U. S. EPA, 1994). The data types and reporting criteria are described in detail in Table 3-1.

For this project, DQOs also include the practical quantitation limits (PQLs) for the analytical methods. The PQLs are based on the extent to which the laboratory instruments or analytical process can provide accurate measurements of a reliable quality for specific constituents. The PQL for a given analysis will vary depending on methodology, instrument sensitivity, and matrix effects. The project PQLs are listed in Attachment C and discussed in Section 7.0.

3.2 DATA QUALITY DEFINITION AND MEASUREMENT

The effectiveness of a QA program is measured by the quality of data generated in the field and by the laboratory. For the ACS pilot and treatability studies, data quality will be assessed in terms of its precision, accuracy, representativeness, completeness, and comparability (PARCC). These terms are described in the following sections.

3.2.1. Precision

Definition. Precision is the reproducibility of measurements under a given set of conditions. For large data sets, precision is expressed as the variability of a group of measurements compared to their average value (i.e., standard deviation). For field duplicate measurements, precision will be expressed as the relative percent difference (RPD) of a pair of duplicate analyses and will be calculated using the following equation:

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

where A and B are the reported concentrations for duplicate sample analyses.

Precision assessment criteria are listed in Attachment D.

Field Precision Objectives. Field precision will be assessed through the collection and measurement of field duplicates at a rate of 1 duplicate per 10 groundwater, condensate, soil vapor, and treatment off-gas samples; and 1 duplicate per 5 soil samples. Field duplicates will be analyzed for each analytical method for Type B and C data.

Laboratory Precision Objectives. Analytical laboratory precision will be assessed through calculated RPD between matrix spike and matrix spike duplicate (MS/MSD) sample data for organic parameters and metals, and through calculated RPDs between sample and matrix duplicate sample pairs for wet chemistry methods. Precision in the laboratory also is assessed by relative standard deviations (RSD) for three or more replicated samples (e.g., response factors for calibration standards).

3.2.2. Accuracy

Definition. Accuracy is the degree of agreement of a measurement or an average of measurements with an accepted reference or "true" value, and is a measure of bias in the system. The accuracy of a measurement system is affected by errors introduced through the sampling process, field contamination, preservation, handling, sample matrix, sample preparation, and analytical techniques.

Accuracy will be evaluated using the percent recovery calculated using the following equation:

$$\text{Percent Recovery} = \frac{|A - B|}{C} \times 100$$

where: A is the target analyte concentration determined analytically from the spiked sample

B is the background level determined by a separate analysis of the unspiked sample

C is the concentration of spike added.

Field Accuracy Objectives. Although accuracy of the field program cannot be assessed quantitatively, the following criteria will be used for a qualitative accuracy assessment for this project: sample handling, preservation, and holding time criteria, the proper calibration and use of the field instruments, and the use of trip blanks to identify problems with sample handling procedures.

Laboratory Accuracy Objectives. Laboratory accuracy will be assessed quantitatively through the analysis of MS/MSD or standard reference materials (SRM), which include

surrogate spikes, laboratory control sample and the spike duplicate (LCS/SD), and response factors for calibration standards. The QC limits for accuracy are provided in Attachment D.

3.2.3. Representativeness

Definition. Representativeness is a qualitative expression of the degree to which sample data accurately and precisely represent a characteristic of a population, a sampling point, or an environmental condition. Representativeness is maximized by ensuring that, for a given project, the number and location of sampling points and the sample collection and analysis techniques are appropriate for the specific investigation, and that the sampling and analysis program will provide information that reflects "true" site conditions.

Measures to Ensure Representativeness of Field Data. Representativeness of field data is dependent upon the proper design of the data collection procedures. The sampling and field measurement procedures to be used for the ACS pilot and treatability studies are based on existing analytical data; the hydrogeology, physical setting, and past land use history of the ACS site; and constraints inherent to the Superfund program. These procedures are described in the field sampling plan (FSP) included as Appendices A through D of this QAPP. Representativeness of the field data will be evaluated by assessing whether the FSPs (Appendices A through D) and this QAPP were followed during sample collection and field measurement data collection. In addition, the analytical results from field duplicate samples also will be used to evaluate the representativeness of field sampling procedures.

Measures to Ensure Representativeness of Laboratory Data. Laboratory data will be evaluated for representativeness by assessing whether the laboratory followed the specified analytical criteria in this QAPP and their SOPs, evaluating holding time criteria, and the results of method and trip blank samples and field duplicate samples. The laboratory SOPs for each method are included in Attachment A.

3.2.4. Comparability

Definition. Comparability is a qualitative parameter that expresses the confidence with which one data set may be compared to another. Comparability is dependent on similar QA

objectives and is achieved through the use of standardized methods for sample collection and analysis, and the use of standardized units of measure.

Measures to Ensure Comparability of Field Data. Comparability is dependent on the design of the sampling program, and will be assessed by evaluating compliance with the specific field measurement SOPs presented in the FSPs.

Measures to Ensure Comparability of Laboratory Data. Laboratory data comparability is dependent on the use of similar sampling and analytical methodology for different projects at a specific site. Comparability of laboratory data will be maximized for the ACS pilot and treatability studies by specifying in the FSP and the QAPP methodologies that are similar to those used in previous ACS projects (as appropriate). Laboratory data comparability will be assessed by comparing data collected during the ACS pilot and treatability studies to historical data (as available) and assessing whether the sampling and analytical methodology presented in the FSP and this QAPP were followed.

3.2.5. Completeness

Definition. Completeness is defined as the percentage of valid data relative to the total number of measurements. Completeness will be calculated using the following equation:

$$\text{Completeness} = \frac{\text{Number of valid data points}}{\text{Total number of measurements}} \times 100$$

Field Data Completeness Objectives. Field data completeness is a quantitative measure of the percentage of valid measurements obtained for all field measurements and will be calculated using the completeness equation. The field data completeness goal for this project is 90 percent (minimum criterion).

Laboratory Data Completeness Objectives. Laboratory data completeness is a quantitative measure of the percentage of valid data for all Type B and C data, as determined by the precision, accuracy, and holding time criteria evaluation. Completeness will be calculated using the completeness equation by dividing the total number of valid data points by the total number of data points. Laboratory completeness for this project is 95 percent (minimum criterion).

3.3 QUALITY CONTROL SAMPLES

The quality control samples that will be used to evaluate analytical data for this program are described and listed by data type and analytical method in Table 3-2. These include QC samples prepared both in the field and by the laboratory. Table 3-3 summarizes the QC sample evaluation of laboratory data in terms of the PARCC parameters. The QC limits that will be used to assess laboratory data are included in Attachment D. The scheduled analytical samples for the treatability studies are listed in the following tables: ISVE Pilot Study—Table 3-4, ISVE/Air Sparging—Table 3-5, and the LTTT Treatability Study, Appendix D—Tables 4-1 through 4-9.

TABLE 3-1

DATA TYPE AND REPORTING FORMAT SUMMARY

Data Type	Data Description	Analysis	Data Reporting Requirements	Report Format (per data type)
A - Field Data	• Soil data collected on site using portable meters	• pH	• Instrument calibration information • pH Data (pH units)	• Field form • Field form
	• Soil vapor data collected on site using portable meters	• Carbon dioxide (CO_2)	• Instrument calibration information • CO_2 Data (percent)	• Field form • Field form
		• Oxygen (O_2)	• Instrument calibration information • O_2 Data (percent)	• Field form • Field form
		• Total volatile hydrocarbons (TVH)	• Instrument calibration information • TPH Data (meter units)	• Field form • Field form
	• Groundwater parameters collected on site using portable meters	• Temperature	• Instrument calibration information • Temperature Data (degrees Centigrade [°C])	• Field form • Field form
		• pH	• Instrument calibration information • pH Data (pH units)	• Field form • Field form
		• Dissolved oxygen (DO)	• Instrument calibration information • DO Data (milligrams/liter [mg/l])	• Field form • Field form
		• Reduction-oxidation potential (Eh)	• Instrument calibration information • Eh Data (millivolts)	• Field form • Field form
		• Specific conductivity (SC)	• Instrument calibration information • SC Data (µmhos)	• Field form • Field form
		• Turbidity	• Instrument calibration information • Turbidity Data (Neptholometric turbidity units [NTU])	• Field form • Field form
	• Field data collected during the OFCA Pre-Treatment Materials Handling Treatability Study	• Mass measurements of soil and debris	• Total tonnage per test pit • Total tonnage of soil per grid • Total tonnage of debris per grid • Percent debris per grid • Percent debris type per grid (i.e., metal) • PCB Field screening data • Screening throughput in tons/hour	• Field form • Field form • Field form • Field form • Field form • Field form • Field form
A - Microbial Data	• Microbial population counts for soil and groundwater	• Heterotrophic plate count	• Colony forming units (CFUs)	• Data report
A - Meterological Data	• Field data collected using a field weather station	• Meterological data	• Ambient air temperature (°F) • Ambient relative humidity • Ambient barometric pressure • Ambient wind speed and direction • Precipitation	• Instrument report • Instrument report • Instrument report • Instrument report • Field form
A - Soil chemical and physical characteristic data	• ASTM methods used to assess the chemical and physical characteristics of soil during the OFCA LTTT treatability study	• Ultimate/Proximate (ASTM D5373/D4239/E-422/D-5142)	• Sample analysis results	• Data report
		• Leachable chlorides (GLI G-40/ME-4)	• Sample analysis results	• Data report
		• Ash Fusability (ASTM D1857)	• Sample analysis results	• Data report
		• Soil pH (SW-846 9045)	• Sample analysis results	• Data report
		• Heating value (ASTMD-1989)	• Sample analysis results	• Data report
		• Particle size distribution (ASTM D-422)	• Sample analysis results	• Data report
		• Atterberg limits (ASTM D-4318)	• Sample analysis results	• Data report
		• Proctor density (ASTM D-698)	• Sample analysis results	• Data report
		• Soil classification (ASTM D-2487)	• Sample analysis results	• Data report

TABLE 3-1

DATA TYPE AND REPORTING FORMAT SUMMARY

Data Type	Data Description	Analysis	Data Reporting Requirements	Report Format (per data type)
A - Standard Methods of Analysis	EPA/600 methods and associated QC	• Acidity (EPA/600 310.1) • Water pH (EPA 150.1, SW-846 9040/9045) • Total sulfur (EPA/600 375.2/375.4/300) • Total chloride (EPA/600325.2/325.3)	• Sample analysis results • Sample analysis results • Sample analysis results • Sample analysis results	• Data report • Data report • Data report • Data report
B- Standard Methods of Analysis	• OFCA LTTT off-gas data from EPA SW-846	• VOCs (SW-846 8260A) • SVOCs (SW-846 8270A) • Pesticides/PCBs (SW-846 8081) • Dioxins/Furans (SW-846 8290)	• Sample analysis results • Sample analysis results • Sample analysis results • Sample analysis results	• Data report • Data report • Data report • Data report
B - Standard Methods of Analysis	• EPA SW-846 methods and associated QC • Standard air methods and associated QC • EPA/600 methods and associated QC	• SW-846 8260A (VOCs) • SW-846 8270A (SVOCs) • SW-846 8081 (Pesticides/PCBs) • SW-846 8151 (Pentachlorophenol) • SW-846 8290 (Dioxin/furan) • SW-846 6010A (As, Ca, Be, Fe, Mg, Mn, P, K,Na, Tl) • TO-13 SVOCs (Air) • TO-14 VOCs (Air) • EPA/600 325.2 (Chloride) • EPA/600 340.2 (Fluoride) • EPA/600 375.4) (Sulfate) • EPA/600 376.1 (Sulfide) • EPA/600 353.2 (Nitrate and Nitrite) • EPA/600 350.3 (Ammonia) • EPA/600 351.2 (Total Kjeldahl nitrogen) • EPA/600 365.2 (ortho-Phosphate) • SW-846 9060 (Total organic carbon, soil) • EPA/600 415.1 (Total organic carbon, water) • TO-3 Methane/ethane (air) • SW-846 8290 Dioxins/Furans	• Case narrative • Completed chain of custodies (form and internal tracking documents) • Initial calibration summary form • Continuing calibration summary form • Injection logs • Target compound results for all samples, including field QC samples including dilution factors, reanalysis, batching information, and bracketing information • Method blank results • Ambient Air Blank (air samples only) • MS/MSD results, if applicable (spike concentration, actual values, and percent recovery) • LCS results, if applicable (spike concentration, actual values, and percent recovery) • Surrogate results, if applicable (spike concentration, actual values, and percent recovery) • Holding time summary	• Hard copy of data report • Hard copy of data report • Hard copy of data report • Hard copy of data report • Hard copy of data report • Hard copy of data report • Electronic copy of data • Hard copy of data report • Electronic copy of data • Hard copy of data report • Electronic copy of data • Hard copy of data report • Electronic copy of data • Hard copy of data report • Electronic copy of data • Hard copy of data report • Electronic copy of data • Hard copy of data report • Electronic copy of data • Hard copy of data report

TABLE 3-1

DATA TYPE AND REPORTING FORMAT SUMMARY

Data Type	Data Description	Analysis	Data Reporting Requirements	Report Format (per data type)
B - Standard Methods of Analysis	• SW-846 Methodologies for 40 CFR 1311 TCLP RCRA Metals	• SW-846 6010A (Ag, As, Ba, Cd, Cr, Pb, Se)	• Case narrative	• Hard copy of data report
		• SW-846 7471 (Mercury)	• Target compound results for all samples, including field QC samples	• Hard copy of data report • Electronic copy of data
			• Date sample collected, extracted, and analyzed	• Hard copy of data report • Electronic copy of data
			• Method blank results	• Hard copy of data report • Electronic copy of data
			• MS results, if applicable (spike concentration, actual values, and percent recovery)	• Hard copy of data report • Electronic copy of data
			• LCS results, if applicable (spike concentration, actual values, and percent recovery)	• Hard copy of data report • Electronic copy of data
C - Standard Methods of Analysis	• GC/MS SW-846 methods and associated QC with CLP-like data reports	• SW-846 8260A (VOCs) • SW-846 8270A (SVOCs)	• Inventory Sheet	• Hard copy of data report
			• Case Narrative	• Hard copy of data report
			• Cover Sheet/Traffic Report	• Hard copy of data report
			• QC Summary	
			—System monitoring compound summary	• Hard copy of data report
			—Surrogate results	• Hard copy of data report • Electronic copy of data
			—LCS results	• Hard copy of data report • Electronic copy of data
			—Method blank results	• Hard copy of data report
			—MS/MSD results	• Hard copy of data report • Electronic copy of data
			—GC/MS instrument Performance Check	• Hard copy of data report
			—Internal standard area and RT summary	• Hard copy of data report
			• Sample Data	
			—Target compound results	• Hard copy of data report • Electronic copy of data
			—TICs	• Hard copy of data report • Electronic copy of data
			—Reconstructed total ion chromatograms (RIC)	• Hard copy of data report
			—Raw spectra and background-subtracted mass spectra of target compounds identified	• Hard copy of data report
			• Standards Data (All instruments)	
			—Initial calibration data	• Hard copy of data report
			—RICs and quantitation reports for all standards	• Hard copy of data report
			—Continuing calibration data	• Hard copy of data report
			• Raw QC Data	
			—BFB or DFTPP/tubing standards (as appropriate)	• Hard copy of data report
			—System blank data	• Hard copy of data report
			—Laboratory control sample data	• Hard copy of data report

TABLE 3-1

DATA TYPE AND REPORTING FORMAT SUMMARY

Data Type	Data Description	Analysis	Data Reporting Requirements	Report Format (per data type)
C - Standard Methods of Analysis	• GC SW-846 methods and associated QC with CLP-like data reports	• SW-846 8081 (Pesticides/PCBs) • SW-846 8151 (Pentachlorophenol, groundwater only)	• Inventory Sheet	• Hard copy of data report
			• Case Narrative	• Hard copy of data report
			• Cover Sheet/Traffic Report	• Hard copy of data report
			• QC Summary	
			—Surrogate results	• Hard copy of data report • Electronic copy of data
			—LCS results	• Hard copy of data report • Electronic copy of data
			—Method blank results	• Hard copy of data report • Electronic copy of data
			—MS/MSD results	• Hard copy of data report • Electronic copy of data
			• Sample Data	
			—Target compound results	• Hard copy of data report
			—Chromatograms (primary column)	• Hard copy of data report
			—Chromatograms (second column confirmation)	• Hard copy of data report
			—GC integration report of data system printout	• Hard copy of data report
			—Manual work sheets	• Hard copy of data report
			• Standards Data	
			—Initial calibration of single component analysis	• Hard copy of data report
			—Initial calibration of muticomponent analysis	• Hard copy of data report
			—Analyte resolution summary	• Hard copy of data report
			—Performance evaluation mixture	• Hard copy of data report
			—Individual standard mixture A	• Hard copy of data report
			—Individual standard mixture B	• Hard copy of data report
			—Calibration verification summary (LCP 1)	• Hard copy of data report
			—Calibration verification summary (LCP-2)	• Hard copy of data report
			—Analytical sequence	• Hard copy of data report
			—Florisil cartridge check	• Hard copy of data report
			—Pesticide identification summary for multicomponent analyte	• Hard copy of data report
			—Chromatograms and data system printouts (retention times and corresponding peak areas or peak heights	• Hard copy of data report
			• Raw QC Data	
			—System blank data	• Hard copy of data report
			—Laboratory control sample data	• Hard copy of data report
			—Florisil data	• Hard copy of data report
			—LCS data	• Hard copy of data report
			—Analytical sequence	• Hard copy of data report

TABLE 3-1

DATA TYPE AND REPORTING FORMAT SUMMARY

Data Type	Data Description	Analysis	Data Reporting Requirements	Report Format (per data type)
C - Standard Methods of Analysis	• Inorganic SW-846 methods and associated QC with CLP-like data reports	• SW-846 7196 (Hexavalent chromium) • SW-846 6010A - Soil (Cd, Ba, Pb, Sb) • SW-846 6010A - Water (As, Tl)	• Inventory Sheet	• Hard copy of data report
			• Case Narrative	• Hard copy of data report
			• Cover Sheet/Traffic Report	• Hard copy of data report
			• Sample Data	
			—Target compound results for both native and QC samples	• Hard copy of data report • Electronic copy of data
			• Quality Control Data	
			—Initial and continuing calibration verification	• Hard copy of data report
			—CRDL standard for AA and ICP	• Hard copy of data report
			—Blanks	• Hard copy of data report
			—ICP interference check sample	• Hard copy of data report
			—Spike sample recovery	• Hard copy of data report
			—Post digest spike sample recovery	• Hard copy of data report
			—Duplicates	• Hard copy of data report
			—Laboratory control sample	• Hard copy of data report
			—Standard addition results	• Hard copy of data report
			—ICP serial dilutions	• Hard copy of data report
			—Preparation log	• Hard copy of data report
			—Analysis run log	• Hard copy of data report
			• Quarterly Verification of Instrument Parameters	
			—Instrument detection limits	• Hard copy of data report
			—ICP inter-element correction factors (Form XI, Part 1)	• Hard copy of data report
			—ICP inter-element correction factors (Form XI, Part 2)	• Hard copy of data report
			• Raw Data	
			— All raw date used to obtain each reported value including real time instrument readouts and digestion or distillation logs	• Hard copy of data report

CRDL Contract-required detection limit

(a) For this project 10 TICs will be reported for SW-846 VOCs and 20 for SW-846 SVOCs (10 for base neutrals and 10 for acids).

TABLE 3-2

QUALITY CONTROL SAMPLE SUMMARY

Method	Data Type	Investigative Sample Media	QC Sample Type	Rationale	Frequency	Description	QC Sample Data Assessment
SW-846 8260A (VOCs), SW-8270B (SVOCs), SW-846 8081 (Pesticides/PCBs), SW-846 8151 (Pentachlororophenol), SW-8290 (Dioxins/Furans)							
Field QC Samples	B/C	Condensate Groundwater Soil LTTT Off-Gas	Trip Blank	Identify target analytes that may have been introduced into samples during sample handling, shipping, or storage at the laboratory	One per each cooler containing samples for VOC analysis	Two 40 milliliter (ml) amber glass vials with Teflon septum caps containing reagent-grade water and preserved to a pH of <2. Prepared by the laboratory.	All target analyte detections will be evaluated in accordance with the functional guidelines for data validation (U.S. EPA 1994).
		Condensate Groundwater Soil LTTT Off-Gas	Field Duplicate	Assess sampling and analytical precision	One per 10 condensate or groundwater samples and one per 20 soil samples for each analytical method	Duplicate of a specific sample submitted "blind" to the laboratory.	RPDs will be calculated between the sample and its duplicate. The RPD for field duplicate organic sample analysis will be ± 50 percent.
			Source Water	Assess quality of water used for equipment decontamination	One per batch of water type used for equipment decontamination	Distilled water that is carried through the same sample collection, handling, and analysis procedures as the investigative samples	All target analyte detections will be evaluated in accordance with the functional guidelines for data validation (U.S. EPA, 1994).
			Equipment Blank	Assess the completeness of equipment decontamination	Ten percent of total number of samples for each analysis type when non-dedicated equipment is used for sampling	Source water that is carried through the same sample collection, handling, and analysis procedures as the investigative samples	All target analyte detections will be evaluated in accordance with the functional guidelines for data validation (U.S. EPA, 1994).
			Temperature Blank	Assess sample temperature criterion	Each sample cooler	A 40-ml amber glass bottle filled with reagent-grade water. The temperature of this sample is measured at the time samples are received by laboratory.	Assess whether temperature criterion has been met for representativeness evaluation.
Laboratory QC Samples	B/C		Method Blank	Identify target analytes that may have been introduced into the sample during analysis	Each sample or extraction batch (20 samples) for each analytical method	Reagent-grade water that is carried through the same analytical process as native samples.	All target analyte detections will be evaluated in accordance with the functional guidelines for data validation (U.S. EPA 1994).
			Surrogate Spikes	Assess analytical accuracy	Each sample for VOC analysis including both investigative and QC samples for each method, except for SW-846 8290	Each sample will be spiked in the laboratory with surrogate spikes in accordance with the laboratories SOPs for the respective methods.	Percent recovery will be calculated for each spiked analyte and compared to the QC limits for surrogate recoveries for each respective method.
			Matrix Spike/Matrix Spike Duplicate	Identify media interference during analysis	One per 20 samples for each media for each analytical method	Condensate and groundwater samples will be collected in triplicate. Two of the samples will be used for MS/MSD analysis. One container will be labeled for MS analysis, the other for MSD analysis. Additional sample volume is not required for soils. The samples will be spiked in the laboratory in accordance with their SOPs for the respective methods.	Percent recovery and the RPD for each spiked analyte will be calculated and compared to laboratory established QC limits for the respective methods.
			Laboratory Control Sample	Assess media interference in the event of MS/MSD analysis that fail QC criteria and to assess laboratory accuracy	One per analytical or extraction batch for each analytical method	LCS are prepared by the laboratory and consist of reagent-grade water spiked with the analytes specified in the laboratories SOPs for the respective methods.	Percent recovery for each spiked analyte will be calculated and compared to the laboratory-established QC limits for each respective method.

TABLE 3-2

QUALITY CONTROL SAMPLE SUMMARY

Method	Data Type	Investigative Sample Media	QC Sample Type	Rationale	Frequency	Description	QC Sample Data Assessment
TO-13 (SVOCs), TO-14 (VOCs), TO-3 (Ethane and Methane)							
Field QC Samples	B	Air Soil Vapor Treatment System Off-Gas	Field Duplicate	Assess sampling and analytical precision	One per 10 samples for each analytical method	Duplicate of a specific native sample submitted "blind" to the laboratory.	RPDs will be calculated between the sample and its duplicate. The RPD for field duplicate samples for organic analysis will be ± 50 percent.
		Air	Ambient Air	Identify target analytes present in the ambient air to establish background concentrations	5 percent of the total number of samples for each analysis	One Tedlar™ bag or Summa canister containing ambient air.	All target analyte detections will be evaluated in accordance with the functional guidelines for data validation (U.S. EPA 1994).
Laboratory QC Samples	B		Method Blank	Identify target analytes that may have been introduced into the sample during analysis	Each sample or extraction batch for each analytical method	Hydrocarbon-free air that is carried through the same analytical process as native samples.	All target analyte detections will be evaluated in accordance with the functional guidelines for data validation (U.S. EPA 1994).
			Laboratory Control Sample and Laboratory Control Sample Duplicate	To assess laboratory precision and accuracy	One per 20 samples for each media for each analytical batch.	LCS are prepared by the laboratory and consist of hydrocarbon-free air spiked with the analytes specified in the laboratories SOPs for the respective methods.	Percent recovery for each spiked analyte will be calculated and compared to the suggested QC limits for the respective methods.
SW-846 6010A (ICP Metals)							
Field QC Samples	B/C	Soil Groundwater Condensate	Field Duplicate	Assess sampling and analytical precision	One per 10 groundwater samples and one per 20 soil samples for each analytical method.	Duplicate of a specific sample submitted "blind" to the laboratory.	RPDs will be calculated between the sample and its duplicate. The RPD for field duplicate inorganic analysis will be ± 25 percent.
Laboratory QC Samples	B/C		Method Blank	Identify target analytes that may have been introduced into the sample during analysis	Each sample or extraction batch for each analysis.	Reagent-grade water that is carried through the same sample prep and analytical process as native samples.	All target analyte detections will be evaluated in accordance with the functional guidelines for data validation (U.S. EPA 1994).
			Matrix Spike/Matrix Spike Duplicate	Identify media interference during analysis	One per 20 samples for each media for each analytical method.	Condensate or groundwater samples will be collected in triplicate. Two of the samples will be used for MS/MSD analysis. One container will be labeled for MS analysis, the other for MSD analysis. *Additional sample volume is not* required for soils. The samples will be spiked in the laboratory in accordance with their SOPs for the respective methods.	Percent recovery and the RPD for each spiked analyte will be calculated and compared to laboratory established QC limits for the respective methods.
			Laboratory Control Sample	Assess media interference in the event of MS/MSD analysis that fail QC criteria and to assess laboratory accuracy	One per 20 samples for each media for each analytical or extraction batch.	LCS are prepared by the laboratory and consist of reagent-grade water spiked with the analytes specified in the laboratories SOPs for the respective methods.	Percent recovery for each spiked analyte will be calculated and compared to laboratory-established QC limits for each respective method.

TABLE 3-2

QUALITY CONTROL SAMPLE SUMMARY

Method	Data Type	Investigative Sample Media	QC Sample Type	Rationale	Frequency	Description	QC Sample Data Assessment
TCLP Metals	**SW-846 1311/6000/7000**						
Field QC Samples	B/C	Soil	Field Duplicate	Assess sampling and analytical precision	One per one per 20 soil samples for each analytical method.	Duplicate of a specific sample submitted "blind" to the laboratory.	RPDs will be calculated between the sample and its duplicate. The RPD for organic analysis will be ± 25 percent.
			Source Water	Assess quality of water used for equipment decontamination	One per batch of water type used for equipment decontamination	Distilled water that is carried through the same sample collection, handling, and analysis procedures as the investigative samples	All target analyte detections will be evaluated in accordance with the functional guidelines for data validation (U.S. EPA, 1994).
			Equipment Blank	Assess the completeness of equipment decontamination	Ten percent of total number of samples for each analysis type when non-dedicated equipment is used for sampling	Source water that is carried through the same sample collection, handling, and analysis procedures as the investigative samples	All target analyte detections will be evaluated in accordance with the functional guidelines for data validation (U.S. EPA, 1994).
			Temperature Blank	Assess sample temperature criterion	Each sample cooler	A 40-ml amber glass bottle filled with reagent-grade water. The temperature of this sample is measured at the time samples are received by laboratory.	Assess whether temperature criterion has been met for representativeness evaluation.
Laboratory QC Samples	B/C		Method Blank	Identify target analytes that may have been introduced into the sample during analysis	Each sample or extraction batch for each analysis.	Reagent-grade water that is carried through the same sample prep and analytical process as native samples.	All target analyte detections will be evaluated in accordance with the functional guidelines for data validation (U.S. EPA 1994).
			Matrix Spike	Identify media interference during analysis	One per 20 samples for each analytical method.	The leachate will be spiked in the laboratory in accordance with their SOPs for the respective methods.	Percent recovery and the RPD for each spiked analyte will be calculated and compared to laboratory established QC limits for the respective methods.
			Laboratory Control Sample	Assess laboratory accuracy	One per analytical or extraction batch for each analytical method.	LCS are prepared by the laboratory and consist of reagent-grade water spiked with the analytes specified in the laboratories SOPs for the respective methods.	Percent recovery for each spiked analyte will be calculated and compared to laboratory-established QC limits for each respective method.
Wet Chemistry SW-846 7196 (Hexavalent Chromium), SW-846 9060 (TOC Soils) EPA/600 (TOC Water, Chloride, Fluoride, Sulfate, Sulfide, Nitrate/Nitrite, Ammonia, Total Kjeldahl Nitrogen, and ortho-Phosphate)							
Field QC Samples	B/C	Groundwater Soil	Field Duplicate	Assess sampling and analytical precision	One per 10 condensate or groundwater samples and one per 20 soil samples for each analytical method.	Duplicate of a specific native sample submitted "blind" to the laboratory.	RPDs will be calculated between the sample and its duplicate. The RPDs will be compared to laboratory established RPDs for matrix duplicate analysis.
			Source Water	Assess quality of water used for equipment decontamination	One per batch of water type used for equipment decontamination	Distilled water that is carried through the same sample collection, handling, and analysis procedures as the investigative samples	All target analyte detections will be evaluated in accordance with the functional guidelines for data validation (U.S. EPA, 1994).
			Equipment Blank	Assess the completeness of equipment decontamination	Ten percent of total number of samples for each analysis type when non-dedicated equipment is used for sampling	Source water that is carried through the same sample collection, handling, and analysis procedures as the investigative samples	All target analyte detections will be evaluated in accordance with the functional guidelines for data validation (U.S. EPA, 1994).

TABLE 3-2

QUALITY CONTROL SAMPLE SUMMARY

Method	Data Type	Investigative Sample Media	QC Sample Type	Rationale	Frequency	Description	QC Sample Data Assessment
Field QC Samples (con't)			Temperature Blank	Assess sample temperature criterion	Each sample cooler	A 40-ml amber glass bottle filled with reagent-grade water. The temperature of this sample is measured at the time samples are received by laboratory.	Assess whether temperature criterion has been met for representativeness evaluation.
Laboratory QC Samples	B/C		Method Blank	Identify target analytes that may have been introduced into the sample during analysis	Each sample or extraction batch (20 samples) for each analytical method.	Reagent-grade water that is carried through the same analytical process as native samples.	All target analyte detections will be evaluated in accordance with the functional guidelines for data validation (U.S. EPA 1994).
			Matrix Spike	Identify media interference during analysis	One per 20 samples for each media for each analytical method.	Condensate and groundwater samples will be collected in triplicate. Two of the samples will be used for MS/MSD analysis. One container will be labeled for MS analysis, the other for MSD analysis. Additional sample volume is not required for soils. The samples will be spiked in the laboratory in accordance with their SOPs for the respective methods.	Percent recovery and the RPD for each spiked analyte will be calculated and compared to laboratory established QC limits for the respective methods.
		Groundwater Soil	Matrix Duplicate	Assess precision of the analytical method	One per analytical or extraction batch for each analytical method.	Groundwater samples will be collected in duplicate. One of the samples will be used for matrix duplicate analysis. Additional sample volume is not required for soils. The samples will be analyzed in accordance with laboratory SOPs.	RPD will be calculated and compared to the laboratory established QC limits for the respective methods.
			Laboratory Control Sample	Assess analytical accuracy	One per analytical or extraction batch for each analytical method, except EPA-SW 7196.	LCS are prepared by the laboratory and consist of reagent-grade water spiked with target analytes as specified in the laboratory SOPs.	Percent recovery for each spiked analyte will be calculated and compared to QC limits based on historical laboratory performance data.

EPA Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW-846, 3rd Edition (EPA, 1992).

EPA Methods Methods for Chemical Analysis of Water and Wastes, EPA Manual, 600/4-79-020 (USEPA, 1983, with additions).

TABLE 3-3

QUALITY CONTROL SAMPLE EVALUATION IN TERMS OF PARCCs

PARCC	QC Program	Evaluation Criteria
Precision	Field Duplicate Pairs	Relative Percent Difference
	Matrix Spike/Matrix Spike Duplicate	Relative Percent Difference
	Investigative Sample/Matrix Duplicate	Relative Percent Difference
Accuracy	Laboratory Spike Duplicate/Laboratory Control Samples (SD)	Percent Recovery
	Matrix Spike	Percent Recovery
	Matrix Spike Duplicate	Percent Recovery
	Surrogate Spikes	Percent Recovery
	LCS/SD	Percent Recovery
	Response Factors	Percent Recovery
	Trip Blanks	Target Analyte Detections
	Holding Time	Quantitative/Qualitative, Degree of Confidence
Representativeness	Method Blanks	Qualitative, Degree of Confidence
	Trip Blanks	Qualitative, Degree of Confidence
	Field Duplicates	Qualitative, Degree of Confidence
	Holding Time	Quantitative/Qualitative, Degree of Confidence
	Equipment Blank (if collected)	Quantitative/Qualitative, Degree of Confidence
	Source Water (if collected)	Quantitative/Qualitative, Degree of Confidence
Completeness	Valid Data	Percent Valid Data[a]
Comparability	Standard Analytical Methods	Qualitative, Degree of Confidence
	Standard Units of Measure	Qualitative, Degree of Confidence
	Standard Sampling Methods	Qualitative, Degree of Confidence

(a) Percent Valid Data = $\frac{\text{number of valid data points}}{\text{total number of measurements}} \times 100$

PARCC Precision, accuracy, representativeness, completeness, and comparability
QC Quality Control
LCS/SD Laboratory control sample and spike duplicate

TABLE 3-4

SAMPLING SCHEDULE FOR THE ISVE PILOT STUDY

Site Identification	Sample Identification	Date Sampled (MMM-DD-YY)	Time Sampled (HH:MM)	Media	Sample Type	VOCs-Air (TO -14)	SVOCs-Air (TO -13)	Ethane and Methane (TO-3)	VOCs-Soil and Condensate (SW-846 8260A)	SVOCs Soil and Condensate (SW-846 8270B)	Pest/PCBs Soil and Condensate (SW-846 8081)	Pentachlorophenol-Condensate (SW-846 8151)	Metals-Soil SW-846 6010A Sb, Cd, Ba, Pb, P, K	Hexavalent Chromium -Soil (SW-846 7196)	Metals-Condensate: As, Be, Mn, Tl (SW-846 6010A)	TOC-Soil (SW-846 9060)	TKN (EPA/600 353.2)	o-Phosphate-Soil (EPA/600 365.2)	Heterotrophic Plate Count-Soil and Groundwater
ISVE Pre-Test Soil Samples																			
ISVE-VEW1	ISVE-VEW1-SO-5			SO	INV	NS	NS		1	1	1	NS	1	1	NS	1	1	1	1
ISVE-VEW2	ISVE-VEW2-SO-15			SO	INV	NS	NS		1	1	1	NS	1	1	NS	1	1	1	1
ISVE-VP1	ISVE-VP1-SO-5			SO	INV	NS	NS		1	1	1	NS	1	1	NS	1	1	1	1
	ISVE-VP1-SO-15			SO	INV	NS	NS		1	1	1	NS	1	1	NS	1	1	1	1
ISVE-VP2	ISVE-VP2-SO-5			SO	INV	NS	NS		1	1	1	NS	1	1	NS	1	1	1	1
	ISVE-VP2-SO-5 MS/MSD			SO	MS/MSD	NS	NS		1	1	1	NS	1	1	NS	NS	NS	NS	1
	ISVE-VP2-SO-5 MS/MD			SO	MS/MD	NS	NS		NS	NS	NS	NS	NS	NS	NS	1	1	1	NS
	ISVE-VP2-SO-5 MD				MD				NS	NS	NS	NS	NS	1	NS	NS	NS	NS	NS
	ISVE-VP2-SO-15			SO	INV	NS	NS		1	1	1	NS	1	1	NS	1	1	1	1
ISVE-VP3	ISVE-VP3-SO-5			SO	INV	NS	NS		1	1	1	NS	1	1	NS	1	1	1	1
	ISVE-VP3-SO-15			SO	INV	NS	NS		1	1	1	NS	1	1	NS	1	1	1	1
	ISVE-VP550-SO-5			SO	FD	NS	NS		1	1	1	NS	1	1	NS	1	1	1	1
ISVE-VP4	ISVE-VP4-SO-5			SO	INV	NS	NS		1	1	1	NS	1	1	NS	1	1	1	1
	ISVE-VP4-SO-15			SO	INV	NS	NS		1	1	1	NS	1	1	NS	1	1	1	1
Total Pre-ISVE Soil Samples																			
	Investigative	NA	NA	SO	INV	NS	NS		10	10	10	NS	10	10	NS	10	10	10	10
	Field Duplicates	NA	NA	SO	FD	NS	NS		1	1	1	NS	1	1	NS	1	1	1	1
	Matrix Spike/Matrix Spike Duplicate	NA	NA	SO	MS/MSD	NS	NS		1	1	1	NS	1	1	NS	NS	NS	NS	1
	Matirx Spike/Matrix Duplicate	NA	NA	SO	MS/MD	NS	NS		NS	NS	NS	NS	NS	NS	NS	1	1	1	NS
	Matrix Duplicate	NA	NA	SO	MD	NS	NS		NS	NS	NS	NS	NS	1	NS	NS	NS	NS	NS

TABLE 3-4

SAMPLING SCHEDULE FOR THE ISVE PILOT STUDY

Site Identification	Sample Identification	Date Sampled (MMM-DD-YY)	Time Sampled (HH:MM)	Media	Sample Type	VOCs-Air (TO -14)	SVOCs-Air (TO -13)	Ethane and Methane (TO-3)	VOCs-Soil and Condensate (SW-846 8260A)	SVOCs Soil and Condensate (SW-846 8270B)	Pest/PCBs Soil and Condensate (SW-846 8081)	Pentachlorophenol-Condensate (SW-846 8151)	Metals-Soil SW-846 6010A Sb, Cd, Ba, Pb, P, K	Hexavalent Chromium -Soil (SW-846 7196)	Metals-Condensate: As, Be, Mn, Tl (SW-846 6010A)	TOC-Soil (SW-846 9060)	TKN (EPA/600 353.2)	o-Phosphate-Soil (EPA/600 365.2)	Heterotrophic Plate Count-Soil and Groundwater
ISVE—Initial Vapor Sampling																			
ISVE-VEW1	ISVE-VEW1-SV			SV	INV	1	1	1	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS
ISVE-VEW2	ISVE-VEW2-SV			SV	INV	1	1	1	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS
ISVE-VP1A	ISVE-VP1A-SV			SV	INV	1	1	1	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS
ISVE-VP1B	ISVE-VP1B-SV			SV	INV	1	1	1	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS
	ISVE-VP51B-SV			SV	FD	1	1	1	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS
ISVE-VP1C	ISVE-VP1C-SV			SV	INV	1	1	1	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS
ISVE-VP2A	ISVE-VP2A-SV			SV	INV	1	1	1	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS
	ISVE-VP52A-SV			SV	FD	1	1	1	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS
ISVE-VP2B	ISVE-VP2B-SV			SV	INV	1	1	1	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS
ISVE-VP2C	ISVE-VP2C-SV			SV	INV	1	1	1	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS
ISVE-VP3A	ISVE-VP3A-SV			SV	INV	1	1	1	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS
ISVE-VP3B	ISVE-VP3B-SV			SV	INV	1	1	1	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS
ISVE-VP3C	ISVE-VP3C-SV			SV	INV	1	1	1	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS
ISVE-VP4A	ISVE-VP4A-SV			SV	INV	1	1	1	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS
ISVE-VP4B	ISVE-VP4B-SV			SV	INV	1	1	1	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS
ISVE-VP4C	ISVE-VP4C-SV			SV	INV	1	1	1	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS
Total Pre-ISVE Soil Vapor Samples																			
	Investigative	NA	NA	SV	INV	14	14	14	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS
	Field Duplicates	NA	NA	SV	FD	2	2	2	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS
	Matrix Spike/Matrix Spike Duplicate	NA	NA	NA	NA	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS
	Matirx Spike/Matrix Duplicate	NA	NA	NA	NA	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS
	Matrix Duplicate	NA	NA	NA	NA	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS

TABLE 3-4

SAMPLING SCHEDULE FOR THE ISVE PILOT STUDY

Site Identification	Sample Identification	Date Sampled (MMM-DD-YY)	Time Sampled (HH:MM)	Media	Sample Type	VOCs-Air (TO -14)	SVOCs-Air (TO -13)	Ethane and Methane (TO-3)	VOCs-Soil and Condensate (SW-846 8260A)	SVOCs Soil and Condensate (SW-846 8270B)	Pest/PCBs Soil and Condensate (SW-846 8081)	Pentachlorophenol-Condensate (SW-846 8151)	Metals-Soil SW-846 6010A Sb, Cd, Ba, Pb, P, K	Hexavalent Chromium -Soil (SW-846 7196)	Metals-Condensate: As, Be, Mn, Tl (SW-846 6010A)	TOC-Soil (SW-846 9060)	TKN (EPA/600 353.2)	o-Phosphate-Soil (EPA/600 365.2)	Heterotrophic Plate Count-Soil and Groundwater
ISVE-Long Term Constant Rate Test—Soil Vapor Samples																			
ISVE-EV1[a]	ISVE-EV1-SV			SV	INV	1	1	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS
	ISVE-EV50-SV			SV	FD	1	1	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS
ISVE-EV1[a]	ISVE-EV1-SV			SV	INV	1	1	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS
ISVE-EV1[a]	ISVE-EV1-SV			SV	INV	1	1	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS
ISVE-EV1[a]	ISVE-EV1-SV			SV	INV	1	1	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS
ISVE-EV1[a]	ISVE-EV1-SV			SV	INV	1	1	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS
ISVE-EV1[a]	ISVE-EV1-SV			SV	INV	1	1	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS
ISVE-EV1[a]	ISVE-EV1-SV			SV	INV	1	1	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS
ISVE-EV1[a]	ISVE-EV1-SV			SV	INV	1	1	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS
ISVE-EV1[a]	ISVE-EV1-SV			SV	INV	1	1	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS
ISVE-EV1[a]	ISVE-EV1-SV			SV	INV	1	1	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS
ISVE-EV1[a]	ISVE-EV1-SV			SV	INV	1	1	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS
ISVE-EV1[a]	ISVE-EV1-SV			SV	INV	1	1	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS
ISVE-EV1[a]	ISVE-EV1-SV			SV	INV	1	1	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS
ISVE-EV1[a]	ISVE-EV1-SV			SV	INV	1	1	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS
Total ISVE-Long Term Constant Rate Test—Soil Vapor Samples																			
	Investigative	NA	NA	SV	INV	14	14	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS
	Field Duplicates	NA	NA	SV	FD	1	1	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS
	Matrix Spike/Matrix Spike Duplicate	NA	NA	NA	MS/MSD	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS
	Matirx Spike/Matrix Duplicate	NA	NA	NA	MS/MD	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS
	Matrix Duplicate	NA	NA	NA	MD	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS

TABLE 3-4

SAMPLING SCHEDULE FOR THE ISVE PILOT STUDY

Site Identification	Sample Identification	Date Sampled (MMM-DD-YY)	Time Sampled (HH:MM)	Media	Sample Type	VOCs-Air (TO -14)	SVOCs-Air (TO -13)	Ethane and Methane (TO-3)	VOCs-Soil and Condensate (SW-846 8260A)	SVOCs Soil and Condensate (SW-846 8270B)	Pest/PCBs Soil and Condensate (SW-846 8081)	Pentachlorophenol-Condensate (SW-846 8151)	Metals-Soil SW-846 6010A Sb, Cd, Ba, Pb, P, K	Hexavalent Chromium -Soil (SW-846 7196)	Metals-Condensate: As, Be, Mn, Tl (SW-846 6010A)	TOC-Soil (SW-846 9060)	TKN (EPA/600 353.2)	o-Phosphate-Soil (EPA/600 365.2)	Heterotrophic Plate Count-Soil and Groundwater
ISVE-Long Term Constant Rate Test—Soil Vapor Treatment Off-Gas Samples																			
ISVE-TV1[(b)]	ISVE-TV1-SV			SV	INV	1	1	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS
ISVE-TV1[(b)]	ISVE-TV1-SV			SV	INV	1	1	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS
ISVE-TV1[(b)]	ISVE-TV1-SV			SV	INV	1	1	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS
ISVE-TV1[(b)]	ISVE-TV1-SV			SV	INV	1	1	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS
	ISVE-TV50-SV			SV	FD	1	1	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS
ISVE-TV1[(b)]	ISVE-TV1-SV			SV	INV	1	1	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS
ISVE-TV1[(b)]	ISVE-TV1-SV			SV	INV	1	1	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS
ISVE-TV1[(b)]	ISVE-TV1-SV			SV	INV	1	1	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS
ISVE-TV1[(b)]	ISVE-TV1-SV			SV	INV	1	1	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS
ISVE-TV1[(b)]	ISVE-TV1-SV			SV	INV	1	1	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS
ISVE-TV1[(b)]	ISVE-TV1-SV			SV	INV	1	1	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS
ISVE-TV1[(b)]	ISVE-TV1-SV			SV	INV	1	1	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS
ISVE-TV1[(b)]	ISVE-TV1-SV			SV	INV	1	1	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS
ISVE-TV1[(b)]	ISVE-TV1-SV			SV	INV	1	1	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS
Total ISVE-Long Term Constant Rate Test—Soil Vapor Treatment Off-Gas Samples																			
	Investigative	NA	NA	SV	INV	14	14	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS
	Field Duplicates	NA	NA	SV	FD	1	1	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS
	Matrix Spike/Matrix Spike Duplicate	NA	NA	NA	MS/MSD	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS
	Matirx Spike/Matrix Duplicate	NA	NA	NA	MS/MD	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS
	Matrix Duplicate	NA	NA	NA	MD	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS

TABLE 3-4

SAMPLING SCHEDULE FOR THE ISVE PILOT STUDY

Site Identification	Sample Identification	Date Sampled (MMM-DD-YY)	Time Sampled (HH:MM)	Media	Sample Type	VOCs-Air (TO -14)	SVOCs-Air (TO -13)	Ethane and Methane (TO-3)	VOCs-Soil and Condensate (SW-846 8260A)	SVOCs Soil and Condensate (SW-846 8270B)	Pest/PCBs Soil and Condensate (SW-846 8081)	Pentachlorophenol-Condensate (SW-846 8151)	Metals-Soil SW-846 6010A Sb, Cd, Ba, Pb, P, K	Hexavalent Chromium -Soil (SW-846 7196)	Metals-Condensate: As, Be, Mn, Tl (SW-846 6010A)	TOC-Soil (SW-846 9060)	TKN (EPA/600 353.2)	o-Phosphate-Soil (EPA/600 365.2)	Heterotrophic Plate Count-Soil and Groundwater
ISVE-Long Term Constant Rate Test—Condensate Samples																			
ISVE-CD1[(b)]	ISVE-CD1-WG			WG	INV	NS	NS	NS	1	1	1	1	1	NS	NS	NS	NS	NS	NS
ISVE-CD1[(b)]	ISVE-CD1-WG MS/MSD			WG	MS/MSD	NS	NS	NS	1	1	1	1	1	NS	NS	NS	NS	NS	NS
ISVE-CD1[(b)]	ISVE-CD1-WG			WG	INV	NS	NS	NS	1	1	1	1	1	NS	NS	NS	NS	NS	NS
ISVE-CD1[(b)]	ISVE-CD1-WG			WG	INV	NS	NS	NS	1	1	1	1	1	NS	NS	NS	NS	NS	NS
ISVE-CD1[(b)]	ISVE-CD1-WG			WG	INV	NS	NS	NS	1	1	1	1	1	NS	NS	NS	NS	NS	NS
ISVE-CD1[(b)]	ISVE-CD50-WG			WG	FD	NS	NS	NS	1	1	1	1	1	NS	NS	NS	NS	NS	NS
ISVE-CD1[(b)]	ISVE-CD1-WG			WG	INV	NS	NS	NS	1	1	1	1	1	NS	NS	NS	NS	NS	NS
ISVE-CD1[(b)]	ISVE-CD1-WG			WG	INV	NS	NS	NS	1	1	1	1	1	NS	NS	NS	NS	NS	NS
ISVE-CD1[(b)]	ISVE-CD1-WG			WG	INV	NS	NS	NS	1	1	1	1	1	NS	NS	NS	NS	NS	NS
ISVE-CD1[(b)]	ISVE-CD1-WG			WG	INV	NS	NS	NS	1	1	1	1	1	NS	NS	NS	NS	NS	NS
ISVE-CD1[(b)]	ISVE-CD1-WG			WG	INV	NS	NS	NS	1	1	1	1	1	NS	NS	NS	NS	NS	NS
ISVE-CD1[(b)]	ISVE-CD1-WG			WG	INV	NS	NS	NS	1	1	1	1	1	NS	NS	NS	NS	NS	NS
ISVE-CD1[(b)]	ISVE-CD1-WG			WG	INV	NS	NS	NS	1	1	1	1	1	NS	NS	NS	NS	NS	NS
ISVE-CD1[(b)]	ISVE-CD1-WG			WG	INV	NS	NS	NS	1	1	1	1	1	NS	NS	NS	NS	NS	NS
Total ISVE-Long Term Constant Rate Test—Condensate Samples																			
	Investigative	NA	NA	WG	INV	NS	NS	NS	13	13	13	13	13	NS	NS	NS	NS	NS	NS
	Field Duplicates	NA	NA	WG	FD	NS	NS	NS	1	1	1	1	1	NS	NS	NS	NS	NS	NS
	Matrix Spike/Matrix Spike Duplicate	NA	NA	WG	MS/MSD	NS	NS	NS	1	1	1	1	1	NS	NS	NS	NS	NS	NS
	Matirx Spike/Matrix Duplicate	NA	NA	NA	MS/MD	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS
	Matrix Duplicate	NA	NA	NA	MD	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS

TABLE 3-4

SAMPLING SCHEDULE FOR THE ISVE PILOT STUDY

Site Identification	Sample Identification	Date Sampled (MMM-DD-YY)	Time Sampled (HH:MM)	Media	Sample Type	VOCs-Air (TO -14)	SVOCs-Air (TO -13)	Ethane and Methane (TO-3)	VOCs-Soil and Condensate (SW-846 8260A)	SVOCs Soil and Condensate (SW-846 8270B)	Pest/PCBs Soil and Condensate (SW-846 8081)	Pentachlorophenol-Condensate (SW-846 8151)	Metals-Soil SW-846 6010A Sb, Cd, Ba, Pb, P, K	Hexavalent Chromium -Soil (SW-846 7196)	Metals-Condensate: As, Be, Mn, Tl (SW-846 6010A)	TOC-Soil (SW-846 9060)	TKN (EPA/600 353.2)	o-Phosphate-Soil (EPA/600 365.2)	Heterotrophic Plate Count-Soil and Groundwater
ISVE Post Test Soil Vapor Samples																			
ISVE-VEW1	ISVE-VEW1-SV			SV	INV	1	1	1	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS
ISVE-VEW2	ISVE-VEW2-SV			SV	INV	1	1	1	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS
	ISVE-VEW51-SV			SV	FD	1	1	1	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS
ISVE-VP1A	ISVE-VP1A-SV			SV	INV	1	1	1	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS
ISVE-VP1B	ISVE-VP1B-SV			SV	INV	1	1	1	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS
ISVE-VP1C	ISVE-VP1C-SV			SV	INV	1	1	1	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS
ISVE-VP2A	ISVE-VP2A-SV			SV	INV	1	1	1	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS
ISVE-VP2B	ISVE-VP2B-SV			SV	INV	1	1	1	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS
ISVE-VP2C	ISVE-VP2C-SV			SV	INV	1	1	1	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS
ISVE-VP3A	ISVE-VP3A-SV			SV	INV	1	1	1	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS
ISVE-VP3B	ISVE-VP3B-SV			SV	INV	1	1	1	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS
ISVE-VP3C	ISVE-VP3C-SV			SV	INV	1	1	1	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS
	ISVE-VP54C-SV			SV	FD	1	1	1	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS
ISVE-VP4A	ISVE-VP4A-SV			SV	INV	1	1	1	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS
ISVE-VP4B	ISVE-VP4B-SV			SV	INV	1	1	1	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS
ISVE-VP4C	ISVE-VP4C-SV			SV	INV	1	1	1	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS
Total ISVE Post Test Soil Vapor Samples																			
	Investigative	NA	NA	SV	INV	14	14	14	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS
	Field Duplicates	NA	NA	SV	FD	1	1	1	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS
	Matrix Spike/Matrix Spike Duplicate	NA	NA	SV	MS/MSD	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS
	Matirx Spike/Matrix Duplicate	NA	NA	NA	MS/MD	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS
	Matrix Duplicate	NA	NA	NA	MD	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS

TABLE 3-4

SAMPLING SCHEDULE FOR THE ISVE PILOT STUDY

Site Identification	Sample Identification	Date Sampled (MMM-DD-YY)	Time Sampled (HH:MM)	Media	Sample Type	VOCs-Air (TO -14)	SVOCs-Air (TO -13)	Ethane and Methane (TO-3)	VOCs-Soil and Condensate (SW-846 8260A)	SVOCs Soil and Condensate (SW-846 8270B)	Pest/PCBs Soil and Condensate (SW-846 8081)	Pentachlorophenol-Condensate (SW-846 8151)	Metals-Soil SW-846 6010A Sb, Cd, Ba, Pb, P, K	Hexavalent Chromium -Soil (SW-846 7196)	Metals-Condensate: As, Be, Mn, Tl (SW-846 6010A)	TOC-Soil (SW-846 9060)	TKN (EPA/600 353.2)	o-Phosphate-Soil (EPA/600 365.2)	Heterotrophic Plate Count-Soil and Groundwater
Total ISVE Post Test Soil Samples																			
ISVE-SB500	ISVE-SB501-SO-5			SO	INV	NS	NS	NS	1	1	1	NS	1	1	NS	1	1	1	1
	ISVE-SB501-SO-15			SO	INV	NS	NS	NS	1	1	1	NS	1	1	NS	1	1	1	1
ISVE-SB501	ISVE-SB502-SO-5			SO	INV	NS	NS	NS	1	1	1	NS	1	1	NS	1	1	1	1
	ISVE-SB502-SO-15			SO	INV	NS	NS	NS	1	1	1	NS	1	1	NS	1	1	1	1
	ISVE-SB552-SO-15				FD	NS	NS	NS	1	1	1	NS	1	1	NS	1	1	1	1
ISVE-SB502	ISVE-SB503-SO-5			SO	INV	NS	NS	NS	1	1	1	NS	1	1	NS	1	1	1	1
	ISVE-SB503-SO-15			SO	INV	NS	NS	NS	1	1	1	NS	1	1	NS	1	1	1	1
ISVE-SB503	ISVE-SB504-SO-5			SO	INV	NS	NS	NS	1	1	1	NS	1	1	NS	1	1	1	1
	ISVE-SB504-SO-15			SO	INV	NS	NS	NS	1	1	1	NS	1	1	NS	1	1	1	1
ISVE-SB504	ISVE-SB505-SO-5			SO	INV	NS	NS	NS	1	1	1	NS	1	1	NS	1	1	1	1
ISVE-SB504	ISVE-SB505-SO-6 MS/MSD			SO	MS/MSD	NS	NS	NS	1	1	1	NS	1	1	NS	NS	NS	NS	1
	ISVE-SB505-SO-7 MS/MD			SO	MS/MD	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	1	1	1	NS
	ISVE-SB505-SO-7 MD			SO	MD	NS	NS	NS	NS	NS	NS	NS	NS	1	NS	NS	NS	NS	NS
ISVE-SB504	ISVE-SB505-SO-15			SO	INV	NS	NS	NS	1	1	1	NS	1	1	NS	1	1	1	1
Total Post-ISVE Soil Samples																			
	Investigative	NA	NA	SO	INV	NS	NS	NS	10	10	10	NS	10	10	NS	10	10	10	10
	Field Duplicates	NA	NA	SO	FD	NS	NS	NS	1	1	1	NS	1	1	NS	1	1	1	1
	Matrix Spike/Matrix Spike Duplicate	NA	NA	SO	MS/MSD	NS	NS	NS	1	1	1	NS	1	1	NS	1	1	1	1
	Matirx Spike/Matrix Duplicate	NA	NA	SO	MS/MD	NS	NS	NS	1	1	1	NS	1	1	NS	1	1	1	1
	Matrix Duplicate	NA	NA	SO	MD	NS	NS	NS	NS	NS	NS	NS	NS	1	NS	NS	NS	NS	NS
Purge Water Sampling																			
ISVE-WP	ISVE-WP			WG	WG	NS	NS	NS	1	1	1	1	NS	NS	1	NS	NS	NS	NS
Trip Blanks[c]																			
	ISVE-TB-(date)-(number)[d]			WQ	TB	NS	NS	NS	1	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS

TABLE 3-4

SAMPLING SCHEDULE FOR THE ISVE PILOT STUDY

Site Identification	Sample Identification	Date Sampled (MMM-DD-YY)	Time Sampled (HH:MM)	Media	Sample Type	VOCs-Air (TO -14)	SVOCs-Air (TO -13)	Ethane and Methane (TO-3)	VOCs-Soil and Condensate (SW-846 8260A)	SVOCs Soil and Condensate (SW-846 8270B)	Pest/PCBs Soil and Condensate (SW-846 8081)	Pentachlorophenol-Condensate (SW-846 8151)	Metals-Soil SW-846 6010A Sb, Cd, Ba, Pb, P, K	Hexavalent Chromium -Soil (SW-846 7196)	Metals-Condensate: As, Be, Mn, Tl (SW-846 6010A)	TOC-Soil (SW-846 9060)	TKN (EPA/600 353.2)	o-Phosphate-Soil (EPA/600 365.2)	Heterotrophic Plate Count-Soil and Groundwater
Ambient Air Samples[e]																			
	ISVE-AA1-(date)-1			AA	AA	1	1	1	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS
	ISVE-AA1-(date)-2			AA	AA	1	1	1	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS
	ISVE-AA1-(date)-3			AA	AA	1	1	1	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS
	ISVE-AA1-(date)-4			AA	AA	1	1	1	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS

(a) Sampled every 2 days during week 1 (3 samples), twice during week 2 (2 samples), weekly from week 3 — 6 (4 samples), and monthly for the rest of the test (5 samples assuming a 6-month test).

(b) Samples collected biweekly for the duration of the test (13 samples total assuming a 6-month test).

(c) The number of trip blanks will be dependent on the duration of the field program. One trip blank will be shipped in each cooler that contains soil or water samples for VOC analysis.

(d) Trip blanks will be designated "TB" followed by the date, and by a sequential number for trip blanks collected on a given day (i.e., the first trip blank on any day will be 1, the second 2, etc.).

(e) One ambient air sample will be collected at the beginning of the study, and then will be collected at a frequency of five percent of the total number of soil vapor samples collected.

Note: Additional sample volume is not required for MS/MSD and MS/MD analyses for soil samples.

TABLE 3-5

SAMPLING SCHEDULE FOR THE ISVE/AIR SPARGING PILOT STUDY

Site Identification	Sample Identification	Date Sampled (MMM-DD-YY)	Time Sampled (HH:MM)	Media	Sample Type	VOCs-Air (TO -14)	SVOCs-Air (TO -13)	Ethane and Methane (TO-3)	VOCs-Soil and Water (SW-846 8260A)	SVOCs Soil and Water (SW-846 8270B)	Pest/PCBs Soil and Water (SW-846 8081)	Pentachlorophenol-Water (SW-846 8151)	Metals-Soil SW-846 6010A Sb, Cd, Ba, Pb, P, K	Hexavalent Chromium -Soil (SW-846 7196)	Metals-Condensate: As, Be, Mn, Tl (SW-846 6010A)	Metals/Cations-Groundwater: Be, Ca, Fe, Mg, Mn, P, K, Na (SW-846 6010A); As (SW-846 7060); Tl (SW-846 7841)	TOC-Soil (SW-846 9060)	TOC-Water (SW-846 415.1)	Chloride (EPA/600 325.2), Fluoride (EPA/600 340.2), Sulfate (EPA/600 375.4), Sulfide (EPA/600 376.1)	TKN (EPA/600 353.2)	Nitrate/Nitrite-Groundwater (EPA 600/353.2)	Ammonia-Groundwater (EPA 600/350.1)	o-Phosphate-Soil and Groundwater (EPA/600 365.2)	Heterotrophic Plate Count-Soil and Groundwater
Pre-ISVE/Air Sparging Soil Samples																								
VEAS-MW501	VEAS-MW501-SO-1			SO	INV	NS	NS	NS	1	1	1	NS	1	1	NS	NS	1	NS	NS	1	NS	NS	1	1
	VEAS-MW501-SO-10			SO	INV	NS	NS	NS	1	1	1	NS	1	1	NS	NS	1	NS	NS	1	NS	NS	1	1
VEAS-MW502	VEAS-MW502-SO-1			SO	INV	NS	NS	NS	1	1	1	NS	1	1	NS	NS	1	NS	NS	1	NS	NS	1	1
	VEAS-MW502-SO-10			SO	INV	NS	NS	NS	1	1	1	NS	1	1	NS	NS	1	NS	NS	1	NS	NS	1	1
VEAS-MW503	VEAS-MW503-SO-1			SO	INV	NS	NS	NS	1	1	1	NS	1	1	NS	NS	1	NS	NS	1	NS	NS	1	1
	VEAS-MW503-SO-10			SO	INV	NS	NS	NS	1	1	1	NS	1	1	NS	NS	1	NS	NS	1	NS	NS	1	1
VEAS-MW504	VEAS-MW504-SO-1			SO	INV	NS	NS	NS	1	1	1	NS	1	1	NS	NS	1	NS	NS	1	NS	NS	1	1
	VEAS-MW550-SO-1			SO	FD	NS	NS	NS	1	1	1	NS	1	1	NS	NS	1	NS	NS	1	NS	NS	1	1
	VEAS-MW504-SO-10			SO	INV	NS	NS	NS	1	1	1	NS	1	1	NS	NS	1	NS	NS	1	NS	NS	1	1
VEAS-AS1	VEAS-AS1-SO-1			SO	INV	NS	NS	NS	1	1	1	NS	1	1	NS	NS	1	NS	NS	1	NS	NS	1	1
	VEAS-AS1-SO-10			SO	INV	NS	NS	NS	1	1	1	NS	1	1	NS	NS	1	NS	NS	1	NS	NS	1	1
	VEAS-AS1-SO-10 MS/MSD			SO	MS/MSD	NS	NS	NS	1	1	1	NS	1	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS
	VEAS-AS1-SO-10 MS/MD			SO	MS/MD	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	1	NS	NS	1	NS	NS	1	1
	VEAS-AS1-SO-10 MD			SO	MD	NS	NS	NS	NS	NS	NS	NS	NS	1	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS
VEAS-AS2	VEAS-AS2-SO-1			SO	INV	NS	NS	NS	1	1	1	NS	1	1	NS	NS	1	NS	NS	1	NS	NS	1	1
	VEAS-AS2-SO-10			SO	INV	NS	NS	NS	1	1	1	NS	1	1	NS	NS	1	NS	NS	1	NS	NS	1	1
Total Pre-ISVE/Air Sparging Soil Samples																								
	Investigative	NA	NA	SO	INV	NS	NS	NS	12	12	12	NS	12	12	NS	NS	12	NS	NS	12	NS	NS	12	12
	Field Duplicates	NA	NA	SO	FD	NS	NS	NS	1	1	1	NS	1	1	NS	NS	1	NS	NS	1	NS	NS	1	1
	Matrix Spike/Matrix Spike Duplicate	NA	NA	SO	MS/MSD	NS	NS	NS	1	1	1	NS	1	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS
	Matirx Spike/Matrix Duplicate	NA	NA	SO	MS/MD	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	1	NS	NS	1	NS	NS	1	1
	Matrix Duplicate	NA	NA	SO	MD	NS	NS	NS	NS	NS	NS	NS	NS	1	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS
Pre-ISVE/Air Sparging Groundwater Samples																								
VEAS-MW501	VEAS-MW501-WG			WG	INV	NS	NS	NS	1	1	1	1	NS	NS	NS	1	NS	1	1	NS	1	1	1	1
	VEAS-MW551-WG			WG	FD	NS	NS	NS	1	1	1	1	NS	NS	NS	1	NS	1	1	NS	1	1	1	1
VEAS-MW502	VEAS-MW502-WG			WG	INV	NS	NS	NS	1	1	1	1	NS	NS	NS	1	NS	1	1	NS	1	1	1	1
	VEAS-MW502-WG MS/MSD			WG	MS/MSD	NS	NS	NS	1	1	1	1	NS	NS	NS	1	NS	NS	NS	NS	NS	NS	NS	NS
	VEAS-MW502-WGMS/MD			WG	MS/MD	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	1	1	NS	1	1	1	1
VEAS-MW503	VEAS-MW503-WG			WG	INV	NS	NS	NS	1	1	1	1	NS	NS	NS	1	NS	1	1	NS	1	1	1	1
VEAS-MW504	VEAS-MW504-WG			WG	INV	NS	NS	NS	1	1	1	1	NS	NS	NS	1	NS	1	1	NS	1	1	1	1

TABLE 3-5

SAMPLING SCHEDULE FOR THE ISVE/AIR SPARGING PILOT STUDY

Site Identification	Sample Identification	Date Sampled (MMM-DD-YY)	Time Sampled (HH:MM)	Media	Sample Type	VOCs-Air (TO -14)	SVOCs-Air (TO -13)	Ethane and Methane (TO-3)	VOCs-Soil and Water (SW-846 8260A)	SVOCs Soil and Water (SW-846 8270B)	Pest/PCBs Soil and Water (SW-846 8081)	Pentachlorophenol-Water (SW-846 8151)	Metals-Soil SW-846 6010A Sb, Cd, Ba, Pb, P, K	Hexavalent Chromium -Soil (SW-846 7196)	Metals-Condensate: As, Be, Mn, Tl (SW-846 6010A)	Metals/Cations-Groundwater: Be, Ca, Fe, Mg, Mn, P, K, Na (SW-846 6010A); As (SW-846 7060); Tl (SW-846 7841)	TOC-Soil (SW-846 9060)	TOC-Water (SW-846 415.1)	Chloride (EPA/600 325.2), Fluoride (EPA/600 340.2), Sulfate (EPA/600 375.4), Sulfide (EPA/600 376.1)	TKN (EPA/600 353.2)	Nitrate/Nitrite-Groundwater (EPA 600/353.2)	Ammonia-Groundwater (EPA 600/350.1)	o-Phosphate-Soil and Groundwater (EPA/600 365.2)	Heterotrophic Plate Count-Soil and Groundwater
Total Pre-ISVE/Air Sparging Groundwater Samples																								
	Investigative	NA	NA	WG	INV	NS	NS	NS	4	4	4	4	NS	NS	NS	4	NS	4	4	NS	4	4	4	4
	Field Duplicates	NA	NA	WG	FD	NS	NS	NS	1	1	1	1	NS	NS	NS	1	NS	1	1	NS	1	1	1	1
	Matrix Spike/Matrix Spike Duplicate	NA	NA	WG	MS/MSD	NS	NS	NS	1	1	1	1	NS	NS	NS	1	NS	NS	NS	NS	NS	NS	NS	NS
	Matirx Spike/Matrix Duplicate	NA	NA	WG	MS/MD	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	1	1	NS	1	1	1	1
	Matrix Duplicate	NA	NA	WG	MD	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS
ISVE System Startup—Soil Vapor Samples (Air sparging system not in operation)																								
VEAS-EV2	VEAS-EV2-SV			SV	INV	1	1	1	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS
VEAS-VP5	VEAS-VP5-SV			SV	INV	1	1	1	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS
VEAS-VP6	VEAS-VP6-SV			SV	INV	1	1	1	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS
VEAS-VP7	VEAS-VP7-SV			SV	INV	1	1	1	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS
	VEAS-VP54-SV			SV	FD	1	1	1	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS
VEAS-VP8	VEAS-VP8-SV			SV	INV	1	1	1	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS
Total ISVE System Startup Soil Vapor Sample																								
	Investigative	NA	NA	SV	INV	5	5	5	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS
	Field Duplicates	NA	NA	SV	FD	1	1	1	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS
	Matrix Spike/Matrix Spike Duplicate	NA	NA	NA	MS/MSD	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS
	Matirx Spike/Matrix Duplicate	NA	NA	NA	MS/MD	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS
	Matrix Duplicate	NA	NA	NA	MD	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS
ISVE Two Week Constant-Rate Test—Soil Vapor Samples																								
VEAS-EV2	VEAS-EV2-SV			SV	INV	1	1	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS
VEAS-EV2	VEAS-EV2-SV			SV	INV	1	1	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS
	VEAS-EV51-SV			SV	FD	1	1	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS
VEAS-EV2	VEAS-EV2-SV			SV	INV	1	1	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS
VEAS-EV2	VEAS-EV2-SV			SV	INV	1	1	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS
VEAS-EV2	VEAS-EV2-SV			SV	INV	1	1	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS

TABLE 3-5

SAMPLING SCHEDULE FOR THE ISVE/AIR SPARGING PILOT STUDY

Site Identification	Sample Identification	Date Sampled (MMM-DD-YY)	Time Sampled (HH:MM)	Media	Sample Type	VOCs-Air (TO -14)	SVOCs-Air (TO -13)	Ethane and Methane (TO-3)	VOCs-Soil and Water (SW-846 8260A)	SVOCs Soil and Water (SW-846 8270B)	Pest/PCBs Soil and Water (SW-846 8081)	Pentachlorophenol-Water (SW-846 8151)	Metals-Soil SW-846 6010A Sb, Cd, Ba, Pb, P, K	Hexavalent Chromium -Soil (SW-846 7196)	Metals-Condensate: As, Be, Mn, Tl (SW-846 6010A)	Metals/Cations-Groundwater: Be, Ca, Fe, Mg, Mn, P, K, Na (SW-846 6010A); As (SW-846 7060); Tl (SW-846 7841)	TOC-Soil (SW-846 9060)	TOC-Water (SW-846 415.1)	Chloride (EPA/600 325.2), Fluoride (EPA/600 340.2), Sulfate (EPA/600 375.4), Sulfide (EPA/600 376.1)	TKN (EPA/600 353.2)	Nitrate/Nitrite-Groundwater (EPA 600/353.2)	Ammonia-Groundwater (EPA 600/350.1)	o-Phosphate-Soil and Groundwater (EPA/600 365.2)	Heterotrophic Plate Count-Soil and Groundwater
Total Soil Vapor Samples																								
	Investigative	NA	NA	SV	INV	5	5	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS
	Field Duplicates	NA	NA	SV	FD	1	1	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS
	Matrix Spike/Matrix Spike Duplicate	NA	NA	NA	MS/MSD	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS
	Matirx Spike/Matrix Duplicate	NA	NA	NA	MS/MD	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS
	Matrix Duplicate	NA	NA	NA	MD	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS
ISVE Two Week Constant-Rate Test—Vapor Treatment System Off-Gas Samples																								
VEAS-TV2	VEAS-TV2-SV			SV	INV	1	1	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS
	VEAS-TV250-SV			SV	FD	1	1	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS
Total Vapor Treatment System Off-Gas Samples																								
	Investigative	NA	NA	SV	INV	1	1	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS
	Field Duplicates	NA	NA	SV	FD	1	1	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS
	Matrix Spike/Matrix Spike Duplicate	NA	NA	NA	MS/MSD	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS
	Matirx Spike/Matrix Duplicate	NA	NA	NA	MS/MD	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS
	Matrix Duplicate	NA	NA	NA	MD	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS
ISVE Two Week Constant-Rate Test—Condensate Samples																								
VEAS-CD2	VEAS-CD2-WG			WG	INV	NS	NS	NS	1	1	1	1	NS	NS	1	NS	NS	NS	NS	NS	NS	NS	NS	NS
	VEAS-CD250-WG			WG	FD	NS	NS	NS	1	1	1	1	NS	NS	1	NS	NS	NS	NS	NS	NS	NS	NS	NS
VEAS-CD2	VEAS-CD2-WG			WG	INV	NS	NS	NS	1	1	1	1	NS	NS	1	NS	NS	NS	NS	NS	NS	NS	NS	NS
	VEAS-CD2-WG MS/MSD			WG	MS/MSD	NS	NS	NS	1	1	1	1	NS	NS	1	NS	NS	NS	NS	NS	NS	NS	NS	NS
Total Condensate Samples																								
	Investigative	NA	NA	WG	INV	NS	NS	NS	2	2	2	2	NS	NS	2	NS	NS	NS	NS	NS	NS	NS	NS	NS
	Field Duplicates	NA	NA	WG	FD	NS	NS	NS	1	1	1	1	NS	NS	1	NS	NS	NS	NS	NS	NS	NS	NS	NS
	Matrix Spike/Matrix Spike Duplicate	NA	NA	WG	MS/MSD	NS	NS	NS	1	1	1	1	NS	NS	1	NS	NS	NS	NS	NS	NS	NS	NS	NS
	Matirx Spike/Matrix Duplicate	NA	NA	NA	MS/MD	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS
	Matrix Duplicate	NA	NA	NA	MD	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS
Air Sparging System Start-up (ISVE not in operation)—No Samples Required																								

TABLE 3-5

SAMPLING SCHEDULE FOR THE ISVE/AIR SPARGING PILOT STUDY

Site Identification	Sample Identification	Date Sampled (MMM-DD-YY)	Time Sampled (HH:MM)	Media	Sample Type	VOCs-Air (TO -14)	SVOCs-Air (TO -13)	Ethane and Methane (TO-3)	VOCs-Soil and Water (SW-846 8260A)	SVOCs Soil and Water (SW-846 8270B)	Pest/PCBs Soil and Water (SW-846 8081)	Pentachlorophenol-Water (SW-846 8151)	Metals-Soil SW-846 6010A Sb, Cd, Ba, Pb, P, K	Hexavalent Chromium -Soil (SW-846 7196)	Metals-Condensate: As, Be, Mn, Tl (SW-846 6010A)	Metals/Cations-Groundwater: Be, Ca, Fe, Mg, Mn, P, K, Na (SW-846 6010A); As (SW-846 7060); Tl (SW-846 7841)	TOC-Soil (SW-846 9060)	TOC-Water (SW-846 415.1)	Chloride (EPA/600 325.2), Fluoride (EPA/600 340.2), Sulfate (EPA/600 375.4), Sulfide (EPA/600 376.1)	TKN (EPA/600 353.2)	Nitrate/Nitrite-Groundwater (EPA 600/353.2)	Ammonia-Groundwater (EPA 600/350.1)	o-Phosphate-Soil and Groundwater (EPA/600 365.2)	Heterotrophic Plate Count-Soil and Groundwater
Combined ISVE/Air Sparging Two-Week Test—Soil Vapor Samples																								
VEAS-EV2	VEAS-EV2-SV			SV	INV	1	1	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS
VEAS-EV2	VEAS-EV2-SV			SV	INV	1	1	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS
	VEAS-EV2-SV			SV	FD	1	1	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS
VEAS-EV2	VEAS-EV2-SV			SV	INV	1	1	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS
VEAS-EV2	VEAS-EV2-SV			SV	INV	1	1	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS
VEAS-EV2	VEAS-EV2-SV			SV	INV	1	1	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS
Total Soil Vapor Samples																								
	Investigative	NA	NA	SV	INV	5	5	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS
	Field Duplicates	NA	NA	SV	FD	1	1	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS
	Matrix Spike/Matrix Spike Duplicate	NA	NA	NA	MS/MSD	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS
	Matirx Spike/Matrix Duplicate	NA	NA	NA	MS/MD	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS
	Matrix Duplicate	NA	NA	NA	MD	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS
Combined ISVE/Air Sparging Two-Week Test—Vapor Treatment System Off-Gas Samples																								
VEAS-TV2	VEAS-TV2-SV			SV	INV	1	1	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS
VEAS-TV2	VEAS-TV2-SV			SV	INV	1	1	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS
	VEAS-TV51-SV			SV	FD	1	1	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS
Total Vapor Treatment Off-Gas Samples																								
	Investigative	NA	NA	SV	INV	2	2	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS
	Field Duplicates	NA	NA	SV	FD	1	1	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS
	Matrix Spike/Matrix Spike Duplicate	NA	NA	SV	MS/MSD	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS
	Matirx Spike/Matrix Duplicate	NA	NA	NA	MS/MD	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS
	Matrix Duplicate	NA	NA	NA	MD	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS
Combined ISVE/Air Sparging Two-Week Test—Condensate Samples																								
VEAS-CD2	VEAS-CD2-WG			WG	1	NS	NS	NS	1	1	1	NS	NS	NS	1	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS
	VEAS-CD2500-WG			WG	FD	NS	NS	NS	1	1	1	NS	NS	NS	1	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS
VEAS-CD2	VEAS-CD2-WG			WG	1	NS	NS	NS	1	1	1	NS	NS	NS	1	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS
	VEAS-CD2-WG MS/MSD			WG	MS/MSD	NS	NS	NS	1	1	1	NS	NS	NS	1	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS

TABLE 3-5

SAMPLING SCHEDULE FOR THE ISVE/AIR SPARGING PILOT STUDY

Site Identification	Sample Identification	Date Sampled (MMM-DD-YY)	Time Sampled (HH:MM)	Media	Sample Type	VOCs-Air (TO -14)	SVOCs-Air (TO -13)	Ethane and Methane (TO-3)	VOCs-Soil and Water (SW-846 8260A)	SVOCs Soil and Water (SW-846 8270B)	Pest/PCBs Soil and Water (SW-846 8081)	Pentachlorophenol-Water (SW-846 8151)	Metals-Soil SW-846 6010A Sb, Cd, Ba, Pb, P, K	Hexavalent Chromium -Soil (SW-846 7196)	Metals-Condensate: As, Be, Mn, Tl (SW-846 6010A)	Metals/Cations-Groundwater: Be, Ca, Fe, Mg, Mn, P, K, Na (SW-846 6010A.); As (SW-846 7060); Tl (SW-846 7841)	TOC-Soil (SW-846 9060)	TOC-Water (SW-846 415.1)	Chloride (EPA/600 325.2), Fluoride (EPA/600 340.2), Sulfate (EPA/600 375.4), Sulfide (EPA/600 376.1)	TKN (EPA/600 353.2)	Nitrate/Nitrite-Groundwater (EPA 600/353.2)	Ammonia-Groundwater (EPA 600/350.1)	o-Phosphate-Soil and Groundwater (EPA/600 365.2)	Heterotrophic Plate Count-Soil and Groundwater
Total Condensate Samples																								
	Investigative	NA	NA	WG	INV	NS	NS	NS	2	2	2	NS	NS	NS	2	NS	NS	NS	NS	NS	NS	NS	NS	NS
	Field Duplicates	NA	NA	WG	FD	NS	NS	NS	1	1	1	NS	NS	NS	1	NS	NS	NS	NS	NS	NS	NS	NS	NS
	Matrix Spike/Matrix Spike Duplicate	NA	NA	WG	MS/MSD	NS	NS	NS	1	1	1	NS	NS	NS	1	NS	NS	NS	NS	NS	NS	NS	NS	NS
	Matirx Spike/Matrix Duplicate	NA	NA	WG	MS/MD	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS
	Matrix Duplicate	NA	NA	WG	MD	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS
ISVE/Air Sparging Post-Test—Soil Vapor Samples																								
VEAS-EV2	VEAS-EV2-SV			SV	INV	1	1	1	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS
VEAS-VP5	VEAS-VP5-SV			SV	INV	1	1	1	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS
	VEAS-VP63-SV			SV	FD	1	1	1	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS
VEAS-VP6	VEAS-VP6-SV			SV	INV	1	1	1	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS
VEAS-VP7	VEAS-VP7-SV			SV	INV	1	1	1	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS
VEAS-VP8	VEAS-VP8-SV			SV	INV	1	1	1	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS
Total Post-Test Soil Vapor Sampling																								
	Investigative	NA	NA	SV	INV	5	5	5	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS
	Field Duplicates	NA	NA	SV	FD	1	1	1	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS
	Matrix Spike/Matrix Spike Duplicate	NA	NA	SV	MS/MSD	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS
	Matirx Spike/Matrix Duplicate	NA	NA	NA	MS/MD	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS
	Matrix Duplicate	NA	NA	NA	MD	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS
ISVE/Air Sparging Post-Test—Groundwater Sampling																								
VEAS-MW501	VEAS-MW501-WG			WG	INV	NS	NS	NS	1	1	1	1	NS	NS	NS	1	NS	1	1	NS	1	1	1	1
VEAS-MW502	VEAS-MW502-WG			WG	INV	NS	NS	NS	1	1	1	1	NS	NS	NS	1	NS	1	1	NS	1	1	1	1
	VEAS-MW552-WG			WG	FD	NS	NS	NS	1	1	1	1	NS	NS	NS	1	NS	1	1	NS	1	1	1	1
VEAS-MW503	VEAS-MW503-WG			WG	INV	NS	NS	NS	1	1	1	1	NS	NS	NS	1	NS	1	1	NS	1	1	1	1
VEAS-MW504	VEAS-MW504-WG			WG	INV	NS	NS	NS	1	1	1	1	NS	NS	NS	1	NS	1	1	NS	1	1	1	1
	VEAS-MW504-WG MS/MSD			WG	MS/MSD	NS	NS	NS	1	1	1	1	NS	NS	NS	1	NS	NS	NS	NS	NS	NS	NS	NS
	VEAS-MW504-WG MS/MD			WG	MS/MD	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	1	1	NS	1	1	1	1

TABLE 3-5

SAMPLING SCHEDULE FOR THE ISVE/AIR SPARGING PILOT STUDY

Site Identification	Sample Identification	Date Sampled (MMM-DD-YY)	Time Sampled (HH:MM)	Media	Sample Type	VOCs-Air (TO -14)	SVOCs-Air (TO -13)	Ethane and Methane (TO-3)	VOCs-Soil and Water (SW-846 8260A)	SVOCs Soil and Water (SW-846 8270B)	Pest/PCBs Soil and Water (SW-846 8081)	Pentachlorophenol-Water (SW-846 8151)	Metals-Soil SW-846 6010A Sb, Cd, Ba, Pb, P, K	Hexavalent Chromium -Soil (SW-846 7196)	Metals-Condensate: As, Be, Mn, Tl (SW-846 6010A)	Metals/Cations-Groundwater: Be, Ca, Fe, Mg, Mn, P, K, Na (SW-846 6010A); As (SW-846 7060); Tl (SW-846 7841)	TOC-Soil (SW-846 9060)	TOC-Water (SW-846 415.1)	Chloride (EPA/600 325.2), Fluoride (EPA/600 340.2), Sulfate (EPA/600 375.4), Sulfide (EPA/600 376.1)	TKN (EPA/600 353.2)	Nitrate/Nitrite-Groundwater (EPA 600/353.2)	Ammonia-Groundwater (EPA 600/350.1)	o-Phosphate-Soil and Groundwater (EPA/600 365.2)	Heterotrophic Plate Count-Soil and Groundwater
Total Post-Test Groundwater Samples																								
	Investigative	NA	NA	WG	INV	NS	NS	NS	4	4	4	4	NS	NS	NS	4	NS	4	4	NS	4	4	4	4
	Field Duplicates	NA	NA	WG	FD	NS	NS	NS	1	1	1	1	NS	NS	NS	1	NS	1	1	NS	1	1	1	1
	Matrix Spike/Matrix Spike Duplicate	NA	NA	WG	MS/MSD	NS	NS	NS	1	1	1	1	NS	NS	NS	1	NS	NS	NS	NS	NS	NS	NS	NS
	Matirx Spike/Matrix Duplicate	NA	NA	WG	MS/MD	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	1	1	NS	1	1	1	1
	Matrix Duplicate	NA	NA	WG	MD	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS
ISVE/Air Sparging Post-Test—Soil Sampling																								
VEAS-SB506	VEAS-SB506-SO-1			SO	INV	NS	NS	NS	1	1	1	NS	1	1	NS	NS	1	NS	NS	1	NS	NS	1	1
	VEAS-SB506-SO-10			SO	INV	NS	NS	NS	1	1	1	NS	1	1	NS	NS	1	NS	NS	1	NS	NS	1	1
VEAS-SB507	VEAS-SB507-SO-1			SO	INV	NS	NS	NS	1	1	1	NS	1	1	NS	NS	1	NS	NS	1	NS	NS	1	1
	VEAS-SB507-SO-10			SO	INV	NS	NS	NS	1	1	1	NS	1	1	NS	NS	1	NS	NS	1	NS	NS	1	1
VEAS-SB508	VEAS-SB508-SO-1			SO	INV	NS	NS	NS	1	1	1	NS	1	1	NS	NS	1	NS	NS	1	NS	NS	1	1
	VEAS-SB508-SO-10			SO	INV	NS	NS	NS	1	1	1	NS	1	1	NS	NS	1	NS	NS	1	NS	NS	1	1
VEAS-SB509	VEAS-SB509-SO-1			SO	INV	NS	NS	NS	1	1	1	NS	1	1	NS	NS	1	NS	NS	1	NS	NS	1	1
	VEAS-SB509-SO-10			SO	INV	NS	NS	NS	1	1	1	NS	1	1	NS	NS	1	NS	NS	1	NS	NS	1	1
	VEAS-SB551-SO-10				FD	NS	NS	NS	1	1	1	NS	1	1	NS	NS	1	NS	NS	1	NS	NS	1	1
VEAS-SB510	VEAS-SB510-SO-1			SO	INV	NS	NS	NS	1	1	1	NS	1	1	NS	NS	1	NS	NS	1	NS	NS	1	1
	VEAS-SB510-SO-10			SO	INV	NS	NS	NS	1	1	1	NS	1	1	NS	NS	1	NS	NS	1	NS	NS	1	1
VEAS-SB511	VEAS-SB511-SO-1			SO	INV	NS	NS	NS	1	1	1	NS	1	1	NS	NS	1	NS	NS	1	NS	NS	1	1
	VEAS-SB511-SO-1 MS/MSD			SO	MS/MSD	NS	NS	NS	1	1	1	NS	1	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS
	VEAS-SB511-SO-1 MS/MD			SO	MS/MD	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	1	NS	NS	1	NS	NS	1	1
	VEAS-SB511-SO-1 MD			SO	MD	NS	NS	NS	NS	NS	NS	NS	NS	1	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS
	VEAS-SB511-SO-10			SO	INV	NS	NS	NS	1	1	1	NS	1	1	NS	NS	1	NS	NS	1	NS	NS	1	1
Total Post-Test Soil Samples																								
	Investigative	NA	NA	SO	INV	NS	NS	NS	12	12	12	NS	12	12	NS	NS	12	NS	NS	12	NS	NS	12	12
	Field Duplicates	NA	NA	SO	FD	NS	NS	NS	1	1	1	NS	1	1	NS	NS	1	NS	NS	1	NS	NS	1	1
	Matrix Spike/Matrix Spike Duplicate	NA	NA	SO	MS/MSD	NS	NS	NS	1	1	1	NS	1	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS
	Matirx Spike/Matrix Duplicate	NA	NA	SO	MS/MD	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	1	NS	NS	1	NS	NS	1	1
	Matrix Duplicate	NA	NA	SO	MD	NS	NS	NS	NS	NS	NS	NS	NS	1	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS
Trip Blanks[a]																								
	VEAS-TB-(date)-(number)[b]			WQ	TB	NS	NS	NS	1	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS

TABLE 3-5

SAMPLING SCHEDULE FOR THE ISVE/AIR SPARGING PILOT STUDY

Site Identification	Sample Identification	Date Sampled (MMM-DD-YY)	Time Sampled (HH:MM)	Media	Sample Type	VOCs-Air (TO -14)	SVOCs-Air (TO -13)	Ethane and Methane (TO-3)	VOCs-Soil and Water (SW-846 8260A)	SVOCs Soil and Water (SW-846 8270B)	Pest/PCBs Soil and Water (SW-846 8081)	Pentachlorophenol-Water (SW-846 8151)	Metals-Soil SW-846 6010A Sb, Cd, Ba, Pb, P, K	Hexavalent Chromium -Soil (SW-846 7196)	Metals-Condensate: As, Be, Mn, Tl (SW-846 6010A)	Metals/Cations-Groundwater: Be, Ca, Fe, Mg, Mn, P, K, Na (SW-846 6010A); As (SW-846 7060); Tl (SW-846 7841)	TOC-Soil (SW-846 9060)	TOC-Water (SW-846 415.1)	Chloride (EPA/600 325.2), Fluoride (EPA/600 340.2), Sulfate (EPA/600 375.4), Sulfide (EPA/600 376.1)	TKN (EPA/600 353.2)	Nitrate/Nitrite-Groundwater (EPA 600/353.2)	Ammonia-Groundwater (EPA 600/350.1)	o-Phosphate-Soil and Groundwater (EPA/600 365.2)	Heterotrophic Plate Count-Soil and Groundwater
Ambient Air Samples[c]																								
	VEAS-AA2-(date)-1			AA	AA	1	1	1	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS
	VEAS-AA2-(date)-2			AA	AA	1	1	1	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS
	VEAS-AA2-(date)-3			AA	AA	1	1	1	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS
	VEAS-AA2-(date)-4			AA	AA	1	1	1	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS
	VEAS-AA2-(date)-5			AA	AA	1	1	1	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS
	VEAS-AA2-(date)-6			AA	AA	1	1	1	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS
	VEAS-AA2-(date)-7			AA	AA	1	1	1	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS
Purge Water from Test Area																								
VEAS-WP	VEAS-WP			WG	INV	NS	NS	NS	1	1	1	1	NS	NS	1	NS	NS	NS	NS	NS	NS	NS	NS	NS

NA Not applicable
NS Not scheduled

Refer to Tables A-2 and B-2 for a description of the sample identification number.

(a) The number of trip blanks will be dependent on the duration of the field program. One trip blank will be shipped in each cooler that contains soil or groundwater samples for VOC analysis.
(b) Trip blanks will be designated "TB" followed by the date the blank was collected, and by a sequential number for trip blanks collected on a given day (i.e., the first trip blank on any day will be 1, the s
(c) One ambient air sample will be collected at the beginning of the study, and then will be collected at a frequency of five percent of the total number of soil vapor samples collected.

Note: Additional sample volume is not required for MS/MSD and MS/MD analyses for soil samples.

4.0 SAMPLING PROCEDURES

4.1 SAMPLE COLLECTION PROCEDURES

During the pilot and treatability studies soil, groundwater, condensate, soil vapor, and treatment off-gas samples will be collected. As described in the FSPs (Appendices A through D) standard procedures will be used for sample collection to meet the project DQOs. The SOPs for soil, groundwater, condensate, and soil vapor sample collection for the ISVE and ISVE/Air Sparging pilot studies are included in Appendices F and G. Sample designation criteria for these studies are described in Sections A.4.0. and B.4.0. of the Appendices A and B. The procedures for sample collection during the Pre-Treatment Materials Handling pilot study and the LTTT treatability study are described in Section 4.0 of Appendix C and Section 4.0 of Appendix D, respectively. The following sections describe sample collection order, the types of containers required for sample collection, and the procedures for QC sample collection.

4.1.1. Sample Collection Order and Containers

For all soil, groundwater, or condensate samples the sample containers will be filled in order of compound volatility or stability as follows:

1. VOCs (SW-846 8260)
2. Hexavalent chromium (SW-846 7196)
3. SVOCs (SW-846 8270)
4. Pentachlorophenol (SW-846 8151)
5. Pesticides/PCBs (SW-846 8080)
6. Total Organic Carbon (SW-846 9060 and EPA/600 415.1)
5. Dioxins/Furans (SW-846 8290)
6. Metals and Cations (SW-846 6010/7000)
7. Anions: chloride, fluoride, sulfate, sulfide, TKN, nitrate/nitrite, ammonia, ortho-phosphate (EPA/600)
8. Soil samples for geophysical, chemical and physical characteristics, microbial population count analysis, or groundwater samples for microbial population count analysis.

Soil vapor or treatment off-gas samples for the ISVE and ISVE/Air Sparging pilot studies will be collected in Summa canisters or Tedlar™ bags and the air samples scheduled for the Pre-Treatment Materials Handling pilot study will be collected in Summa canisters. The sampling order for air, soil vapor, or off-gas samples is:

1. VOCs (TO-14)
2. SVOCs (TO-13)
3. Methane and ethane (TO-3)

All soil, groundwater, and condensate samples will be placed in contaminant-free containers as specified in the *U.S. EPA Specifications and Guidance for Obtaining Contaminant-Free Sample Containers* (U.S. EPA, 1992). The containers for soil, groundwater, and condensate samples, and the preservatives for each analytical method for the ISVE and ISVE/Air Sparging Pilot Studies are listed in Table 4-1. The sample containers and preservatives for the Pre-Treatment Materials Handling Pilot Study are described in Section 5.2 of Appendix C and the sample containers and preservatives for the LTTT treatability study are in Table 4-10 of Appendix D.

As discussed previously, air samples collected during the Pre-Treatment Materials Handling pilot study will be collected in Summa canisters. For the ISVE and ISVE/Air Sparging pilot studies, soil vapor and off-gas samples will be collected in either Tedlar™ bags or Summa canisters. Summa canisters are reusable and are cleaned and provided by the analytical laboratory. After cleaning, hydrocarbon-free air or nitrogen gas is injected into the canister. The hydrocarbon-free air or nitrogen is then analyzed for target analytes. All target analytes must be below the practical quantitation limit for the Summa canister to be acceptable for reuse. The laboratory will provide the analytical data for each canister. If Tedlar™ bags are used for sampling, a new, certified clean Tedlar™ bag will be used for each sample. There are no preservative or temperature requirements for air, soil vapor, or off-gas samples.

4.2 QUALITY CONTROL SAMPLE COLLECTION

The following field QC samples will be used to assess data quality for the pilot and treatability studies:

- Trip blanks
- Temperature blanks
- Equipment blanks
- Ambient air blanks
- Field duplicates
- Matrix spike and matrix spike duplicate samples
- Matrix duplicate samples

4.2.1. Trip Blanks

Trip blanks will be prepared by the laboratory and will consist of two 40-milliliter (ml) amber glass bottles filled with preserved reagent-grade water. The bottles will be filled so that there is no head space and will be capped with a Tedlar™ septum. Trip blanks will be placed in each cooler containing soil, groundwater, or condensate samples for VOC analysis, except for soil samples from the LTTT treatability study. Each trip blank will be designated with a "TB" and the date it was submitted for analysis (e.g., TB-9/30/96).

4.2.2. Temperature Blanks

A temperature blank will accompany each cooler of soil, groundwater, or condensate samples shipped to the laboratory, except for soil samples from the LTTT treatability study. The temperature blank will be prepared by the laboratory and will consist of a 40-ml amber glass bottle filled with reagent-grade water. The temperature of the blank will be measured upon arrival at the laboratory. If the temperature of the blank is outside the 2° to 6° C temperature criterion, both the Laboratory Manager and Montgomery Watson Project Manager will be notified immediately and the appropriate corrective actions will be taken. Each temperature blank will be designated with a "T°C" and the date it was submitted for analysis (e.g., T°C -9/30/96).

4.2.3. Source Water Blanks

A sample of each batch of source water (distilled water used for equipment decontamination) will be collected for all scheduled analysis, except for heterotrophic plate counts. These data will be used to assess source water quality.

4.2.4. Equipment Blanks

Equipment blanks will be collected at a frequency of 10 percent when non-dedicated or non-disposable equipment is used for sampling. Equipment blanks will be collected for each analytical parameter for which the associated environmental sample was collected. Equipment blanks will be collected immediately after the sampling equipment is decontaminated by pouring the source water used for equipment decontamination over the sampling equipment, then collecting it in the appropriate sample containers. Equipment blanks will have the same designation as the investigative sample collected immediately before the equipment blank, except that an "EB" will follow the sample designation (e.g., ISVE-SB501-SO-5EB).

4.2.5. Ambient Air Blanks

Upwind and downwind ambient air blanks for VOC and SVOC analyses will be collected prior to excavation for the Pre-Treatment Materials Handling Pilot Study. The ambient air blank will be collected in a Summa canister by opening the vacuum valve on the Summa canister so that ambient air enters the container. The sample will be labeled "AU" (air upwind) or "AD" (air downwind), followed by a 0 (day 0 of the study), and the date (e.g., AU-0-9/63/96).

For the ISVE and ISVE/Air Sparging pilot studies, ambient air blanks will be collected in either Tedlar™ bags or Summa canisters by either pumping ambient air into the Tedlar™ bag or opening the vacuum valve on the Summa canister so that ambient air enters the container. During the ISVE and ISVE/Air Sparging pilot studies, ambient air blanks will be collected for five percent of the total number of samples. Ambient air samples will be collected over the duration of the project (i.e., ambient blanks will not be collected 5 days in a row and then none collected for the remainder of the project). Ambient air blanks

will be identified with the pilot study identification ("ISVE" or "VEAS"), an "AA," and the date (e.g., VEAS-AA2-9/30/96).

4.2.6. Field Duplicates

A field duplicate sample pair is a single grab sample that is split into two samples during collection. Two sets of sample containers will be prepared prior to sample collection. One set will contain the actual sample, the other the field duplicate. For groundwater field duplicate sample collection, all sample fractions, except those for VOC analysis, will be collected by alternately pouring a bailer-volume or discharging a pump-volume of water into the sample and duplicate sample containers. For VOCs, the investigative sample will be collected in one bailer pour or pump discharge. The field duplicate will be collected immediately after the investigative sample using the same procedures as the investigative sample. Because groundwater samples for metals and cation analyses (other than hexavalent chromium) will be filtered with a disposable 0.45-micron filter prior to being placed in the containers, the field duplicates for these analyses also will be filtered. Both samples will be sent to the same laboratory for analysis.

Soil samples will be collected with split spoon samplers during the ISVE and ISVE/Air Sparging pilot studies. Each field duplicate soil sample for VOC analysis will be collected by removing soil adjoining the soil collected for the investigative sample and placing it directly into the sample container. The soil sample container will be filled so that there is no head space. For the other sample fractions, the soil remaining in the split spoon sampler will be removed, placed into a stainless steel bowl, and thoroughly homogenized. The soil in the bowl will be split evenly between the two sets of sample containers. Both samples will be sent to the same laboratory for analysis.

Soil duplicate samples for the LTTT treatability study are described in Section 4.0 of Appendix D.

As discussed previously, soil vapor or air samples will be collected with either Tedlar™ bags or Summa canisters. The field duplicate will be collected immediately after the actual sample using the same sampling procedures.

All field duplicates will be submitted "blind" to the laboratory. The field duplicates will be designated with fictitious sample identifications as shown in Tables 3-4 and 3.5, and Table 4-1 of Appendix D. The actual sample location will be noted on the sample tracking log (Tables 3-4 and 3-5).

4.2.7. Matrix Spike and Matrix Spike Duplicate Samples

Matrix duplicate and matrix spike duplicate samples will be collected for VOC, SVOC, pesticide/PCB, pentachlorophenol, dioxins/furans, and metals analyses for all media types (Table 3-2). Matrix spike samples will be collected for TOC, chloride, fluoride, sulfate, sulfide, ortho-phosphate, nitrate/nitrite, ammonia, and TKN analyses for all media types. A matrix spike only will be analyzed for TCLP metals. Sample media for matrix spike and matrix spike analysis will be specific to the ACS site so that media interference can be assessed. For groundwater samples, the same procedures used for field duplicate sample collection will be used for MS/MSD sample collection, except that a fictitious sample identification will not be used for the duplicate sample. Groundwater samples collected for MS/MSD analyses will have the same designation as their associated investigative sample, except that an "MS" or "MSD" will follow the sample designation (e.g., VEAS-MW-502-WG-MS).

Because additional soil volume is not required for MS/MSD analysis, there are no requirements for sample labeling. However, the soil samples scheduled for MS/MSD analysis will be clearly identified on the chain-of-custody and on the sample tracking form (Tables 3-4 and 3-5, and Table 4-1 of Appendix D).

4.2.8. Matrix Duplicate Samples

Matrix duplicate samples will be analyzed for TOC, chloride, fluoride, sulfate, sulfide, ortho-phosphate, nitrate/nitrite, ammonia, and TKN. The same sampling procedures used to collect field duplicates for groundwater samples will be used for matrix duplicate samples, except that a fictitious sample identification will not be used for the matrix duplicate sample. Additional sample volume is not required for soil samples.

Groundwater samples collected for matrix duplicate analyses will have the same designation as their associated actual sample, except that an "MD" will follow the sample

designation (e.g., VEAS-MW-502-WG-MD). Because additional soil volume is not required for matrix duplicate analysis, there are no requirements for sample labeling. However, the soil samples scheduled for matrix duplicate analysis form (Tables 3-4 and 3-5) will be clearly identified on the chain-of-custody and on the sample tracking form (Tables 3-4 and 3-5).

TABLE 4-1

STANDARD UNITS OF MEASUREMENT AND HOLDING TIME

Analysis (Method)[a]	Sample Container	Preservative	Unit of Measure	Holding Time
GROUNDWATER AND CONDENSATE				
VOCs (SW-846 8260A)	2 40-ml amber glass bottles with a Teflon™ septum cap; No head space	HCl; pH < 2 Chill to 4°C	µg/l	14 days from sample collection to analysis
SVOCs (SW-846 8270B)	1 1-liter amber glass bottle with a Teflon™ lined cap	No preservation Chill to 4°C	µg/l	7 days from sample collection to extraction 40 days from sample extraction to analysis
Pentachlorophenol (SW-846 8151)	1 1-liter amber glass bottle with a Teflon™ lined cap	No preservation Chill to 4°C	µg/l	7 days from sample collection to extraction 40 days from sample extraction to analysis
Pesticides/PCBs (SW-846 8081)	1 1-liter amber glass bottle with a Teflon™ lined cap	No preservation Chill to 4°C	µg/l	7 days from sample collection to extraction 40 days from sample extraction to analysis
Metals[a] (SW-846 6010A)	1 1-liter polyethylene bottle with a Teflon™ lined cap	HNO_3; pH < 2 Chill to 4°C	µg/l	180 days from sample collection to analysis
Ortho-Phosphate (EPA/600 365.2)	50-ml polyethylene with a Teflon™ lined cap	Filter immediately Chill to 4°C	mg/l	48 hours from sample collection to analysis
Nitrate/Nitrite (EPA/600 353.2)	100-ml polyethylene bottle with a Teflon™ lined cap	H_2SO_4; pH < 2 Chill to 4°C	mg/l	28 days from sample collection to analysis
Ammonia (EPA/600 350.3)	500-ml polyethylene bottle with a Teflon™ lined cap	H_2SO_4; pH < 2 Chill to 4°C	mg/l	28 days from sample collection to analysis
Chloride (EPA/600 352.2)	50-ml polyethylene bottle with a Teflon™ lined cap	Chill to 4°C	mg/l	28 days from sample collection to analysis
Fluoride (EPA/600 340.2)	500-ml polyethylene bottle with a Teflon™ lined cap	Chill to 4°C	mg/l	28 days from sample collection to analysis
Sulfate (EPA/600 375.4)	50-ml polyethylene bottle with a Teflon™ lined cap	Chill to 4°C	mg/l	28 days from sample collection to analysis
Sulfide (EPA/600 376.1)	500-ml polyethylene bottle with a Teflon™ lined cap	HCl; pH < 2 Chill to 4°C	mg/l	28 days from sample collection to analysis
TOC (EPA/600 415.1)	50-ml polyethylene bottle with a Teflon™ lined cap	H_2SO_4; < 2 Chill to 4°C	mg/l	28 days from sample collection to analysis
Heterotrophic plate count	Sterile 125-ml polyethylene container	0.5 ml $Na_2S_2O_3$	CFU	30 hours from sample collection to analysis
SOIL				
VOCs (SW-8260A)	4-oz glass jar; No head space	No preservation Chill to 4°C	µg/kg	14 days from sample collection to analysis
SVOCs (SW-846 8270B)	8-oz wide-mouth glass jar with a Teflon™ lined cap	No preservation Chill to 4°C	µg/kg	14 days from sample collection to extraction 40 days from sample extraction to analysis
Pesticides/PCBs (SW-846 8081)	Included in SVOCs container	No preservation Chill to 4°C	µg/kg	14 days from sample collection to extraction 40 days from sample extraction to analysis
Dioxins/Furans (SW-846 8290)	4-oz glass jar; No head space	No preservation Chill to 4°C	µg/kg	30 days from sample collection to extraction 40 days from sample extraction to analysis
Metals[b] (SW-846 6010A)	4-oz glass jar; No head space	No preservation Chill to 4°C	µg/g	180 days from sample collection to analysis

TABLE 4-1

STANDARD UNITS OF MEASUREMENT AND HOLDING TIME

Analysis (Method)[a]	Sample Container	Preservative	Unit of Measure	Holding Time
SOIL (con't)				
Metals[c] (SW-846 6010A)	4-oz glass jar; No head space	No preservation Chill to 4°C	µg/g	180 days from sample collection to analysis
Mercury (SW-7470)	Included in metals container	No preservation Chill to 4°C	µg/g	28 days from sample collection to analysis
RCRA metals	4-oz glass jar	No preservation Chill to 4°C	µg/l	180 days from sample collection to analysis
Hexavalent Chromium (SW-846 7196)	4-oz glass jar; No head space	No preservation Chill to 4°C	µg/g	48 hours from sample collection to extraction, 24 hours from extraction to analysis
Ortho-Phosphate (EPA/600 365.2)	Included in hexavalent chromium container	No preservation Chill to 4°C	mg/kg	48 hours from sample collection to analysis
Total Kjeldahl Nitrogen	Included in hexavalent chromium container	No preservation Chill to 4°C	mg/kg	28 days from sample collection to analysis
TOC (SW-846 9060)	Included in hexavalent chromium container	No preservation Chill to 4°C	mg/kg	28 days from sample collection to analysis
pH	Included in hexavalent chromium container	No preservation	pH units	Analyze immediately
Heterotrophic plate count (9215B)	4-oz sterilized glass container	Chill to 4°C	CFU	30 hours from sample collection to analysis
Soil Particle Size Distribution (ASTM D-422)	4-inch brass sleeve	None	Percent	None
AIR				
TO-13 SVOCs	Tedlar™ Bag or Summa Canister	None	µg/sample	14 days from sample collection to analysis
TO-14 VOCs	Tedlar™ Bag or Summa Canister	None	ppbv	36 hours from sample collection to analysis
TO-3 Ethane and Methane	Tedlar™ Bag or Summa Canister	None	ppbv	36 hours from sample collection to analysis

EPA Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW-846, 3rd Edition (EPA, 1992).
EPA Methods Methods for Chemical Analysis of Water and Wastes, EPA Manual, 600/4-79-020 (USEPA, 1983, with additions).
Compendium of Methods for Determination of Toxic Organic Compounds in Ambient Air, April 1994.

RCRA metals: Arsenic, barium, cadmium, chromium, lead, mercury, selenium, silver

CFU Colony-forming unit
mg/kg Milligram per kilogram
µg/l Microgram per liter
ppbv Part per billion by volume
mg/l Milligram/liter

(a) Arsenic, beryllium, thallium, manganese, calcium, iron, magnesium, phosphorous, potassium, sodium
(b) Antimony, cadmium, barium, lead, phosphorous, potassium
(c) Beryllium, antimony, thallium, sodium, potassium, phosphorous

5.0 SAMPLE CUSTODY

To ensure that samples are identified correctly and remain representative of the environment, the sample documentation and custody procedures, as outlined in this section, will be used during the sampling program to maintain and document sample integrity during collection, transportation, storage, and analysis. Field sampling personnel will be responsible for ensuring that proper documentation and custody procedures are initiated at the time of sample collection, and that individual samples can be tracked from the time of sample collection until custody of the samples is transferred to the laboratory. The laboratory will be responsible for maintaining sample custody and documentation from the time the laboratory receives samples until final sample disposition.

5.1 CHAIN OF CUSTODY

Chain of custody (COC) procedures provide an accurate written record of the possession of each sample from the time it is collected in the field through laboratory analysis. A sample is considered in custody if one of the following applies:

- It is in an authorized person's immediate possession
- It is in view of an authorized person after being in physical possession
- It is in a secure area after having been in an authorized person's physical possession
- It is in a designated secure area, restricted to authorized personnel only.

The following sections describe the COC procedures that will be used in the field and in the laboratories.

5.2 COC FIELD PROCEDURES

5.2.1. Field Documentation

Sample custody and documentation procedures will be initiated at the time each sample is collected. The field sampler has ultimate responsibility for the samples until they are submitted to the laboratory for analysis. If a commercial carrier is used to transport the samples to the laboratory, the field sampler's responsibility ends when the carrier assumes responsibility for the samples. Sample identification numbers will be assigned prior to sample collection and are described in Section A.4.0. of Appendix A, Section B.4.0. of Appendix B, Section 4.2 of Appendix C, and Section 4.1 of Appendix D. Sample collection details will be documented on the appropriate field forms including the chain-of-custody (COC; Figure 5-1), the Soil Boring Log Form (Appendix G, Attachment 1), the Groundwater Sampling Log Form (Appendix F, Attachment 2), and in the field log book. All entries will be made using indelible ink. Any errors will be corrected by drawing a single line through the incorrect entry, entering the correct information, and then initialing and dating the change.

Field logbooks will be bound, field survey books or notebooks. Logbooks will be assigned to field personnel. After completion of the project all field log books will stored in the final evidence file. Each logbook will be identified by a project-specific document number.

The title page of each field logbook will contain the following:

- Name and address of the field contact person (individual who is assigned the field book)
- Logbook number
- Project name and location
- Project start date
- Project end date.

Information to be entered in the logbook will include personnel on Site (including visitors), weather conditions, the date(s) soil, vapor probe, or monitoring well borings were started and completed, the type of field equipment used, field equipment calibration methods, sampling methodology, sample depths, samples submitted to the laboratory for analysis, sample type (e.g., field duplicates), sample destinations and how they were transported (e.g., name of laboratory and shipping agent), photograph numbers and descriptions (if applicable), decontamination procedures, any deviations from the FSP, and any other observations that may be relevant to the field program.

5.2.2. Sample Labels

Sample labels will be completed and attached to the sample containers at the time each sample is collected. An example of the type of label that will be used is shown in Figure 5-2. The following information will be included on the sample label:

- Label number
- Project name/location
- Sample location
- Field sample identification
- Sample type
- Date and time of sample collection
- Type of analyses to be performed
- Preservative (if applicable)
- Sampler's initials.

5.2.3. Chain of Custody Record

Each COC form will be completed properly to ensure that sample custody is documented, appropriate sample fractions have been collected, and scheduled analyses are properly assigned. As described previously, all entries will be made using indelible ink. Any errors will be corrected by drawing a single line through the incorrect entry, entering the correct information, and then initialing and dating the change. An example of the type of COC record that will be used is shown in Figure 5-1.

Unused portions of the COC form will be crossed out and initialed. A completed COC record will be included with each sample cooler. Because it is expected that all samples will be transported via Federal Express Priority Service, the air bill number will be recorded on the COC record. The signed air bills will serve as evidence of custody transfer between the field sampler and carrier, and carrier and laboratory. The sampler will retain and file copies of the COC record and the air bill after the samples are shipped. The carrier will relinquish samples to the laboratory upon arrival, and the laboratory personnel will then complete the COC.

5.2.4. Sample Packaging and Shipping Procedures

All groundwater, soil, and condensate samples will be packaged and shipped to the laboratories on the day they are collected using the following procedures:

- Sample labels will be completed and attached to sample containers as described previously.

- A completed sample tag will be attached with a wire to each sample container for each investigative or quality control sample. All entries will be made using indelible ink. Any errors will be corrected by drawing a single line through the incorrect entry, entering the correct information, and then initialing and dating the change. The tag will include the field sample number, location (if not encoded in the sample ID), date and time of sample collection, and type of analysis. There will also be a space available for entry of the lab sample ID number.

- The samples will be placed upright in a waterproof metal (or equivalent strength plastic) ice chest or cooler.

- Wet ice in double Ziploc™ bags (to prevent leakage) will be placed around, among, and on top of the sample bottles. Enough ice will be used so that the samples will be chilled and maintained at 4 ± 2°C during transport to the laboratory.

- To prevent the sample containers from sliding around the cooler, the remaining space in cooler will be filled with inert cushioning material, such as shipping peanuts, additional bubble pack, or cardboard dividers.

- The original copy of the completed COC Form will be placed in a waterproof plastic bag and taped to the inside of the cooler lid.

- The lid will be secured by wrapping strapping tape completely around the cooler in two locations.

- "This Side Up" labels will be placed on two sides of the cooler.

- Custody seals such as those shown in Figure 5-3 will be placed in two locations (the front right and back left of the cooler) across the cooler closure to ensure that any tampering is detected. The date and initials of the sampler will be written on the custody seal.

- A copy of the COC record and the signed air bill will be retained for the project files.

Air, soil vapor, or treatment system off-gas samples will be packaged and shipped to the laboratory on the day they are collected using the following procedures:

- A completed sample tag will be attached with a wire to each Summa canister or Tedlar™ bag for each investigative or quality control sample. All entries will be made using indelible ink. Any errors will be corrected by drawing a single line through the incorrect entry, entering the correct information, and then initialing and dating the change. The tag will include the field sample number, location (if not encoded in the sample ID), date and time of sample collection, and type of analysis. There will also be a space available for entry of the lab sample ID number.

- The samples in Tedlar™ bags will be placed upright in a waterproof metal (or equivalent strength plastic) ice chest or cooler. The Summa canisters will be placed in their original shipping container.

- To prevent the Tedlar™ bags from sliding around the cooler, the cooler will be filled with inert cushioning material, such as shipping peanuts, additional bubble pack, or cardboard dividers.

- The original copy of the completed COC Form will be placed in a waterproof plastic bag and taped to the inside of the cooler lid or the Summa canister containers.

- The coolers or Summa canister containers will be secured by wrapping strapping tape completely around the containers in two locations.

- "This Side Up" labels will be placed on two sides of the containers.

- Custody seals such as those shown in Figure 5-3 will be placed in two locations (the front right and back left of the cooler) across the container closures to ensure that any tampering is detected. The date and initials of the sampler will be written on the custody seal.

- A copy of the COC record and the signed air bill will be retained for the project files.

5.3 LABORATORY COC PROCEDURES

Two laboratories will be used for Data Type B and C data analysis: IEA and Quanterra. The laboratory COC procedures described below apply to both facilities.

Upon receipt in the laboratory, the integrity of the shipping container will be checked by verifying that the custody seal is not broken. The cooler will be opened and examined for evidence of proper cooling, and the presence of trip blanks and temperature blanks, and the individual sample containers will be checked for breakage, damage, or leakage. The contents of the shipping container will then be verified against the COC. If any problems are found, they will be documented on the sample custody form(s) and the Montgomery Watson Project Manager will be notified immediately. The shipping receipts will be placed the COC records and stored in the project file.

A permanent logbook will be maintained in the sample control area to document the following:

- Date of sample receipt
- Sample identification number
- Number of samples
- Source of samples

All deficiencies and/or discrepancies will be reported immediately to the Laboratory Project Manager, and an Anomaly Form will be completed. The Laboratory Project Manager either will resolve the problem internally or contact Montgomery Watson's Project Manager for resolution.

If the samples and documentation are acceptable, each sample container will be assigned a unique laboratory identification number from the Laboratory Information Management Systems (LIMS) database. Sample tracking will be documented on the LIMS (one of the functions of the LIMS is to assist in tracking samples while they are in the custody of the laboratory). Other information that will be recorded includes date and time of sampling, sample description, due dates, and required analytical tests.

When the LIMS log-in has been completed, the samples will be transferred to the appropriate refrigerators in the Sample Control area. Separate refrigerators will be used for samples suspected to contain high concentrations of organic compounds and for samples for VOC analyses. The sample refrigerators will be kept at 4°C ± 2°C and their temperatures will be recorded daily with thermometers calibrated against National Institute of Standards and Technology (NIST) thermometers. Storage blanks will be used to assess the cleanliness of sample storage areas.

The Laboratory Department Managers or their designee will be notified that the samples have been logged into the LIMS. The LIMS will maintain records of the sample custody. A sample location list is entered into the LIMS and printed on each sample label. Access to samples will be limited to authorized personnel, and a Sample Check In/Out is tracked with the LIMS. Samples will be distributed for analysis from Sample Control by either a sample custodian or a laboratory chemist.

Sample custody will be maintained within the laboratory's secure facility until sample disposal has been recorded in the sample logbook. Any marks or notes made on the chain-of-custody document by the Sample Custodian will be clearly distinguishable from original field notation.

Thirty days after a laboratory report has been generated and submitted to Montgomery Watson, the samples are transferred to the sample disposal area. This transfer will be documented on the Sample Control Form. Samples will be disposed according to the laboratory's SOP, which is based on both State and Federal guidelines.

5.4 FINAL EVIDENCE FILES CUSTODY PROCEDURES

The final evidence files for investigation data are maintained by the Montgomery Watson project office in Addison, Illinois and will be under the custody of the Montgomery Watson Project Manager in a locked, secured area. At a minimum, the evidence file will contain all relevant records including:

- Field logbooks
- Field data and data deliverables
- Photographs
- Design drawings
- All original soil, monitoring well, piezometer, extraction well, air sparging, and soil vapor probe boring logs
- All soil, monitoring well, piezometer, extraction well, air sparging, and soil vapor probe construction details
- Laboratory data deliverables
- Data validation reports

- Data assessment reports
- Progress reports, QA reports, interim project reports, etc.
- All custody documentation (tags, forms, airbills, etc.).

This document has been developed for a specific application and may not be used without the written approval of Montgomery Watson.

QUALITY CONTROL				
	Graphic Standards *Montgomery Watson*	Technical Review *J. He*	Management Review *D.C. Drain*
	Lead Professional *D. Bolaris*	Project Manager *R. Schlicher*	Other

CHAIN OF CUSTODY RECORD/LAB WORK REQUEST

PROJECT NO.: _____

LABORATORY _____

Chain of Custody Number _____
Cooler Number _____
Lot Control No. _____
Page _____ **of** _____
Air Bill No. _____

MONTGOMERY WATSON
Phone (801) 272-1900 FAX (801) 272-0430
MW Contact _____
Project _____
Project Number _____
Date Due _____
Samplers Signature _____

Lab ID No. (Lab Only)	Location ID	Sample ID	Depth Interval (ft)	Date Collected	Time Collected	Matrix [a]	Sampling Technique [b]	ANALYSES REQUESTED											

[a] **Matrix:**
SO - Soil
SE - Sediment
WS - Surface Water
WG - Ground Water
LF - Product
AA - Air
SW - Wipe
EP/TCLP Leachate
WQ - Trip Blank, Equipment Blanks
WW - Wastewater

[b] **Sampling Technique:**
Composite=C
Grab=G
Hand Auger=HA
Cone Penetrometer=CN
Bladder Pump=BP
Submersible Pump=SP
Bailer=B

SPECIAL INSTRUCTIONS:

Relinquished by/Affiliation	Received by/Affiliation	Date	Time	Relinquished by/Affiliation	Received by/Affiliation	Date	Time

LABORATORY USE ONLY

SAMPLES WERE:

1 Shipped or hand delivered
Notes:

2 Ambient or Chilled
Notes:

3 Temperature _____

4 Received Broken/Leaking (Improperly Sealed)
Y N
Notes:

5 Properly Preserved
Y N
Notes:

6 Received Within Holding Times
Y N
Notes:

COC Tape Was:

1 Present on Outer Package
Y N NA

2 Unbroken on Outer Package
Y N NA

3 Present on Sample
Y N NA

4 Unbroken on Sample
Y N NA
Notes:

Discrepancies Between Sample Labels and COC Record?
Y N
Notes:

Developed By *J. He* Drawn By *D. Bolaris*
Approved By *R. Schlicher* Date *8/12/96*
Reference
Revisions *None*

CUSTODY SEAL

TREATABILITY STUDY
AMERICAN CHEMICAL SERVICE, INC.
NPL SITE
GRIFFITH, INDIANA

MONTGOMERY WATSON

Drawing Number 4077.0191

FIGURE 5-1

This document has been developed for a specific application and may not be used without the written approval of Montgomery Watson.

QUALITY CONTROL				
	Graphic Standards *Montgomery Watson*	Technical Review *J. He*	Management Review *D.C. Drain*
	Lead Professional *D. Bolaris*	Project Manager *R. Schlicher*	Other

CHAIN OF CUSTODY RECORD/LAB WORK REQUEST (EXTENSION SHEET)

Chain of Custody Number _____
Cooler Number _____
Lot Control ID _____
Page _____ of _____
Air Bill No. _____

LABORATORY _____

MONTGOMERY WATSON
Phone (801) 272-1900 FAX (801) 272-0430

Lab ID No. (Lab Only)	Location ID	Sample ID	Depth Interval (ft)	Date Collected	Time Collected	Matrix [a]	Sampling Tech. [b]	ANALYSES REQUESTED

LABORATORY USE ONLY

SAMPLES WERE:

1 Shipped or hand delivered
Notes:

2 Ambient or Chilled
Notes:

3 Temperature _____

4 Received Broken/Leaking (Improperly Sealed)
Y N
Notes:

5 Properly Preserved
Y N
Notes:

6 Received Within Holding Times
Y N
Notes:

COC Tape Was:

1 Present on Outer Package
Y N NA

2 Unbroken on Outer Package
Y N NA

3 Present on Sample
Y N NA

4 Unbroken on Sample
Y N NA
Notes:

Discrepancies Between Sample Labels and COC Record?
Y N
Notes:

CUSTODY SEAL

Developed By *J. He* Drawn By *D. Bolaris*
Approved By *R. Schlicher* Date *8/12/96*
Reference
Revisions *None*

TREATABILITY STUDY
AMERICAN CHEMICAL SERVICE, INC.
NPL SITE
GRIFFITH, INDIANA

MONTGOMERY WATSON

Drawing Number
4077.0191

FIGURE 5-1a

QUALITY CONTROL	Graphic Standards Lead Professional	Montgomery Watson D. Bolaris	Technical Review Project Manager	J. He R. Schlicher	Management Review Other	D.C. Drain

MONTGOMERY WATSON	LABEL NUMBER	
PROJECT NAME/LOCATION	SAMPLE DATE	SAMPLE TIME
	PRESERVATIVE	
SAMPLE LOCATION	SAMPLER'S INITIALS	
FIELD SAMPLE ID	GRAB	COMPOSITE
ANALYSIS		

This document has been developed for a specific application and may not be used without the written approval of Montgomery Watson.

FIGURE 5-2

Developed By *J. He* Drawn By *D. Bolaris*	**SAMPLE LABEL**	Drawing Number 4077.0191
Approved By *R. Schlicher* Date *8/12/96*	TREATABILITY STUDY AMERICAN CHEMICAL SERVICE, INC. NPL SITE GRIFFITH, INDIANA	MONTGOMERY WATSON
Reference		
Revisions *None*		

This document has been developed for a specific application and may not be used without the written approval of Montgomery Watson.

QUALITY CONTROL					
	Graphic Standards	*Montgomery Watson*	Technical Review	*J. He*	Management Review *D.C. Drain*
	Lead Professional	*D. Bolaris*	Project Manager	*R. Schlicher*	Other

LAB SAMPLE
DO NOT TAMPER

DATE _____

INITIALS _____

Lynn Peavey Co.
1-800-255-6499

FIGURE 5-3

Developed By *J. He*	Drawn By *D. Bolaris*	**CUSTODY SEAL**	Drawing Number 4077.0191
Approved By *R. Schlicher*	Date *8/12/96*		
Reference		TREATABILITY STUDY AMERICAN CHEMICAL SERVICE, INC. NPL SITE GRIFFITH, INDIANA	**MONTGOMERY WATSON**
Revisions *None*			

6.0 CALIBRATION PROCEDURES AND FREQUENCY

This section describes the general requirements for calibrating field instruments or equipment and for laboratory instrument calibration and preparing standards. Instrument calibration is necessary for accurate sample quantitation, and establishes the dynamic range of an instrument. Criteria for calibration are specific to each method and instrument manufacturer. The following paragraphs outline the calibration procedures for the field equipment and laboratory instrumentation.

6.1 FIELD INSTRUMENT CALIBRATION

Field instruments will be used to measure oxygen (O_2), carbon dioxide (CO_2), and total volatile hydrocarbons in soil gas; pH, specific conductivity, and temperature of groundwater; and the elevation of groundwater. The instruments and equipment used to collect these data will be calibrated with sufficient frequency and in such a manner that accuracy and reproducibility of results are consistent with the manufacturer's specifications and the procedures outlines in the SOPs included in Appendix E.

Each field instrument of piece of equipment used to collect field data will be examined prior to use to ensure that it is in good working order. This includes reviewing and understanding the manufacturer's operation manual for each instrument and ensuring that all maintenance requirements are followed. The following paragraphs briefly describe calibration procedures for the instruments that will be used during the treatability and pilot studies.

Organic Vapor Meter, Oxygen, and Carbon dioxide Meters. An organic vapor meter (flame ionization detector) will be used to measure total volatile hydrocarbons and an O_2/CO_2 meter will be used to measure oxygen and carbon dioxide in soil gas. Each meter will be calibrated prior to use on a daily basis in accordance with the manufacturers specifications with standards provided by the manufacturer or other approved source. Any time that instrument drift is suspected, the calibration of the meter will be checked and if necessary, the instrument will be recalibrated. In addition, a calibration check will be performed at the conclusion of each day of use in order to evaluate instrument performance. Instruments will not be adjusted before the

final calibration check has been performed and recorded. Calibration gases that have a shelf life will not be used past the expiration date.

pH, Temperature, and Specific Conductivity Meters. The instrument(s) used to measure pH, temperature, and specific conductivity will be calibrated daily prior to use according to the manufacturer's instructions. The meter(s) will be calibrated using calibration solutions supplied by the manufacturer that bracket the expected environmental conditions. Periodically, the calibration of the instrument(s) will be checked using independent reference solutions including two pH buffers that bracket the measured pH (generally pH 7 and pH 10 or pH 4 and 7) and a 1,000 micromhos per centimeter (µmhos/cm) standard conductivity solution. All reference solutions that have a shelf life will not be used past the expiration date. The temperature probe will be checked periodically against an National Institute of Standards and Testing (NIST)-traceable thermometer to confirm measurements.

Water-Level Sounder. Electric water-level sounders will be used to measure groundwater elevation. The sounders will be checked before the beginning of field activities by comparing the scale on the water-level tape against an engineering measurement tape.

6.2 LABORATORY INSTRUMENT CALIBRATION

6.2.1. Calibration Standards

Data accuracy is dependent upon the accuracy of the standards used for instrument calibration. To ensure the highest quality standard, primary reference standards used by IEA are obtained from the National Institute of Standards and Technology (NIST), EPA CRADA vendors, or other reliable commercial sources. When standards are received at the laboratory, the date received, supplier, lot number, purity, concentration, and expiration date are recorded in a standards log book. Vendor certification for the standards are retained in the files.

Standards are obtained either in their pure form, or in stock or working standard solutions. Dilutions are made from vendor standards. All standards are given a standard identification number and the following information is recorded in the standards log book; source of the standard, the initial concentration of the standard, the

final concentration of the standard, the volume of the standard that was diluted, the volume of the final solution, the solvent and the source and lot number of the solvent used for standard preparation, and the preparer's initials. All standards are validated prior to use.

Validation procedures for standards include a check for chromatographic purity, and verification of the standard's concentration by comparing its response to a standard of the same analyte prepared or obtained from a different source. Reagents also are analyzed for purity; for example, every lot of dichloromethane (used for organic extraction) is analyzed for contaminants prior to use in the laboratory. Standards are checked routinely for signs of deterioration (e.g. discoloration, formation of precipitates, and changes in concentration) and are discarded if deterioration is suspected or the expiration date has passed. Expiration dates are based on vendor recommendation, the analytical method, or internal research. Stock solutions for VOCs are not to be held for more than 30 days. Fresh working calibration standards will be prepared every week. Stock solutions for semi-volatile organic compounds will not be held for more than 90 days. Dilutions below 1 part per million (ppm) will not be held more than 30 days.

6.2.2. Instrument Calibration

Laboratory instrument calibration is required to ensure that the analytical system is operating correctly and functioning at the sensitivity necessary to meet established reporting limits. Each instrument will be calibrated according to the manufacturer's guidelines using standard solutions appropriate to the type of instrument and the linear range established for the analytical method. All instrument calibration information is documented, and at a minimum includes the equipment to be calibrated, the reference standards used for calibration, the calibration techniques, actions, acceptable performance tolerances, frequency of calibration, and calibration documentation format. The laboratory will maintain records of standard preparation and instrument calibration.

Calibration records will include daily checks using standards prepared independently of the calibration standards and instrument response will be evaluated against established criteria. The analysis logbook, maintained for each analytical instrument,

will include at a minimum: the date and time of calibration, the initials of the person performing the calibration, the calibrator reference number and concentration. IEA and Quanterra instrument calibration procedures are included with the SOPs for each analytical method in Attachment A.

7.0 ANALYTICAL PROCEDURES

The analytical methods to be used by each laboratory for Type B and C data are defined in this section. Standard methodologies, PQLs, and units of measure will be used for all Types B and C Data for the pilot and treatability studies.

7.1 IEA, INC.

All soil, groundwater, and condensate samples collected for Type B and C data will be analyzed by IEA Inc., of Cary, North Carolina, except those samples for dioxin/furan analysis. All samples will be prepared and analyzed in accordance with the *Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW-846, 3rd Edition* (U.S. EPA, 1992) *and EPA Methods for Chemical Analysis of Water and Wastes, EPA Manual, 600/4-79-020* (U.S. EPA, 1983, with additions) and in accordance with the laboratory's SOPs (Attachment A). The analytical methods, holding times, and units of measure are listed in Table 4-1. All soil data will be reported on a dry weight basis. IEA's PQLs for each analyte are listed in Attachment C. The PQLs are laboratory-specific target reporting limits that can be met in the absence of matrix interferences or high contaminant concentrations, and are at least as stringent as the reporting limits specified for the individual analytical methods. PQLs are developed based on statistical method detection limit studies. Method detection limits (MDLs) are determined on an annual basis following guidelines in the 40 CFR Part 136, Appendix B.

7.2 QUANTERRA ENVIRONMENTAL SERVICES

All samples collected for dioxins/furans will be analyzed by Quanterra (formerly Enseco) of West Sacramento, California, in accordance with the Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW-846, 3rd Edition (U.S. EPA 1992) and in accordance with the laboratory's SOPs (Attachment A). All soil vapor, treatment off-gas, and air samples collected for Data Type B will be analyzed by Quanterra of City of Industry, California (TO-14 for VOCs and TO-3 for ethane and methane) and West Sacramento, California (Modified TO-13 for SVOCs). All samples will be prepared and analyzed in accordance with the *Compendium of Methods for the Determination of Toxic Organic Compounds in Ambient Air, April, 1984* and in accordance with the laboratory's SOPs (Attachment A). The methods and holding times are listed in Table 4-1. The units

of measure and Quanterra PQLs for each analyte are listed in Attachment C. The PQLs are laboratory-specific target reporting limits that can be met in the absence of matrix interferences or high contaminant concentrations, and are developed based on statistical MDL studies. MDLs are determined on an annual basis following guidelines in the 40 CFR Part 136, Appendix B.

8.0 INTERNAL QUALITY CONTROL CHECKS

Internal quality control checks are used to evaluate whether a method is performing within acceptable limits of precision and accuracy. The following sections describe the internal QC for both field and laboratory activities.

8.1 FIELD SAMPLE COLLECTION

Quality control samples used to assess field sampling techniques and environmental conditions during sample collection and transportation will include field duplicates, trip blanks, ambient air blanks, source water blanks (if required), and equipment blanks (if required). The type of QC samples for each analytical method, the sampling frequency, and how the resulting data will be used are described in Table 3-2. The total number of investigative and QC samples for each analysis and media type are listed in Tables 3-4 and 3-5, and in Table 4-1 in Appendix D.

8.2 FIELD MEASUREMENTS

QC procedures for field measurements will include checking the reproducibility of the measurements by obtaining multiple readings on a single grab sample, and calibrating the field instruments or equipment in accordance with the SOPs included in Appendix E.

8.3 LABORATORY ANALYSIS

The general objectives of the laboratory QC program are to:

- Ensure that all procedures are documented, including any changes in administrative and/or technical procedures
- Ensure that all analytical procedures are validated and conducted according to method guidelines and laboratory SOPs
- Monitor the performance of the laboratory using a systematic inspection program

- Ensure that all data are properly reported and archived.

Each analytical laboratory, IEA and Quanterra, will conduct internal quality control for analytical in accordance with their SOPs, the individual method requirements, and this QAPP. The laboratory will notify the Montgomery Watson Project Manager in writing before making significant changes to the QAPP or analytical methodology.

Laboratory quality control consists of two distinct components, a laboratory component and a matrix component. The laboratory component measures the performance of the laboratory analytical process during the sample analyses, while the matrix component measures the effects of a specific media on the method performance. The QC samples that will be used to assess the laboratory component and the media component of analysis are listed in Table 3-2. The criteria against which the QC data will be evaluated are listed in Attachment D. Corrective actions for instrument calibrations or QC sample data out of compliance are listed in Attachment B. The data reporting requirements for each Data Type are listed in Table 3-1.

9.0 DATA REDUCTION, VALIDATION, AND REPORTING

9.1 DATA VALIDATION AND REPORTING BY DATA TYPE

The specific objectives for the ISVE, ISVE/Air Sparging, and Pre-Treatment Material Handling pilot studies and the LTTT treatability study are listed in Table 1-2. In addition, Table 1-2 defines the DQOs, data to be collected, data type, and the data usage for each treatability or pilot study sub-task. The following paragraphs summarize the data reporting and validation requirements for the pilot and treatability studies:

- Data Type A includes all data that will be used for informational purposes or data gathered using methods that have limited QC or data validation requirements. Included in Data Type A are all field data, microbial population data, and physical and chemical soil characteristic data from ASTM methods (Table 3-1). These data will be reviewed by the Montgomery Watson QAO to ensure that the procedures specified in this QAPP were followed. These data will be reported in a format that will facilitate their review. These data will not be validated.

- Data Type B includes all samples analyzed using EPA SW-846, EPA/600, or standard air methods and the respective associated QC. Standard data packages will be provided with Level B data (Table 3-1). These data will be used primarily for technology evaluation and design. The data will be reduced as appropriate to their intended use, and will be reported in a format that will facilitate their review. All Type B data will be validated.

- Data Type C includes samples analyzed using EPA SW-846 methods and the respective associated QC; however, the data reports will be CLP-like (Table 3-1). These data will be used primarily for technology evaluation and design. The data will be reduced as appropriate to their intended use, and will be in a format that will facilitate their review. All Type C data will be validated.

9.2 FIELD DATA

9.2.1. Reporting

Field data generated for the Site will be presented in a format that will facilitate data review and evaluation. A narrative will be included in the final treatability study report to describe any deviations from the procedures, explain any qualifications regarding the data quality, and discuss any significant problems encountered during field measurement. Tables, graphs, or figures will be used to present the data.

9.2.2. Validation

All field data will be reviewed by the Montgomery Watson QAO to ensure that the procedures specified in this QAPP were followed; however, no formal data validation will be performed.

9.3 LABORATORY DATA

Laboratory data will be reduced and validated as specified by the analytical methods and the laboratory's SOPs. Data reduction calculations are specific to the analytical instruments that are used for analysis, the level of automation, and the type of software used to reduce the data. The procedures used for data reduction and validation for each analytical method are described in the laboratory's SOPs, which are included as Attachment A to this document. Refer to Sections 3.2.1., 3.2.2., and 3.2.5. for calculations that will be used by the laboratory to assess precision, accuracy, and completeness, respectively. The following sections briefly describe IEA's and Quanterra's data reduction and validation.

9.3.1. IEA Laboratories

Data Reduction and Validation. IEA will perform in-house analytical data reduction and validation under the direction of the Laboratory Project Manager and the Laboratory QA Manager before the data are released to Montgomery Watson. The Laboratory Project Manager and QAO are responsible for assessing the data quality and qualifying any data that may be unreliable. IEA's review of the data includes assessing QC data compliance with the control limits in IEA's SOPs and this QAPP (Attachment D) and

making sure that all corrective actions were followed appropriately (Attachment B). The laboratory will prepare and retain full analytical and QC documentation. The data reduction and review will be conducted as follows:

- The bench analyst will convert the raw data into reportable data, and conduct the initial data review. The analyst will review preliminary data entries, calculations, holding times, calibration check standards, precision, and accuracy. The analyst also will provide explanation and/or corrective action summaries for any method control parameters that are outside control criteria. The analyst will sign the analytical batch control form when the review is complete.

- The Department Manager or designee (not the bench analyst) will review the analytical control documentation associated with each batch, as well as any corrective action explanations provided by the analyst. If the Department Manager or designee is not satisfied with all corrective action explanations and analytical control results, additional explanation will be required for the batch. The Department Manager is responsible for determining whether the analytical data meet quality control criteria established by the analytical methods and by this QAPP. The Department Manager will sign the analytical batch control form when satisfied with the data quality. If there are QC problems, the Laboratory QC Officer will initiate and follow up on corrective actions taken to rectify the problem. A permanent record of these actions will be kept in IEA's files.

- The Laboratory Project Manager will review analytical data batches that have been approved by the analyst and the Department Manager, and will sign the batch control form when satisfied with the data. The Project Manager also is responsible for reviewing all final data reports for proper format and reporting consistency prior to releasing the reports to Montgomery Watson.

Data Reporting. The analytical data will be reported in a format that will facilitate independent data validation. All of the data, including QC data, will be reported in chronological order. All data will be reported as specified in Table 3-1 of this QAPP and will be qualified in accordance with EPA CLP Guidance.

9.3.2. Quanterra

Data Reduction and Validation. Quanterra will perform in-house analytical data reduction and validation at three levels before the data are released to Montgomery Watson. Technical review will be performed by the analyst and a supervisor or designee. The Laboratory Project Manager is responsible for ensuring that project requirements are met in each analytical report. The laboratory will prepare and retain full analytical and QC documentation. The Quanterra data reduction and validation will be conducted as follows:

- The bench analyst will convert the raw data into reportable data, and conduct the initial data review. The analyst will review preliminary data entries, calculations, holding times, calibration check standards, precision, and accuracy. The analyst will also provide explanation and/or corrective action summaries for any method control parameters that are outside control criteria. The analyst will document this review on the data review checklist.

- The Section Supervisor or designee (not the bench analyst) will review the analytical control documentation associated with each batch, as well as any corrective action explanations provided by the analyst. If the Supervisor or designee is not satisfied with all corrective action explanations and analytical control results, additional explanation will be required for the batch. The Supervisor is responsible for determining whether the analytical data meet quality control criteria established by the analytical methods and by this QAPP. The Section Supervisor or designee will document this review on the data review checklist.

- In the case of QC problems, the Laboratory QC Officer will initiate and follow up on corrective actions taken to rectify the problem. A permanent record of these in-house corrective actions is maintained by the laboratory.

- The Laboratory Project Manager will review analytical data batches that have been approved by the analyst and the Supervisor or designee for proper format

and reporting consistency prior to releasing the reports to Montgomery Watson.

Data Reporting. The analytical data will be reported in a format that will facilitate independent data validation. All of the data, including QC data, will be reported in chronological order. All data will be reported as specified in Table 3-1 of this QAPP and will be qualified in accordance with EPA CLP guidance.

10.0 PERFORMANCE AND SYSTEM AUDITS

Performance and system audits of field and laboratory activities will be conducted to verify that sampling and analysis for the ISVE, ISVE/Air Sparging, and Pre-Treatment Materials Handling pilot studies and the LTTT treatability conform to the procedures specified in this QAPP. Performance and systems audits include both internal and external audits. Internal audits will be conducted by Montgomery Watson's or the laboratories QAO. External audits will be performed by the U.S. EPA Region V or IDEM.

10.1 FIELD PERFORMANCE AND SYSTEM AUDITS

Oversight of field procedures will be the direct responsibility of the Montgomery Watson Project Manager, who will review all elements of the QAPP to ensure that the objectives of the pilot and treatability studies are met. In addition to an initial review, the sampling procedures will be reviewed as the field work progresses so that any necessary modifications can be made.

Internal audits of field activities (sampling and measurements) will be conducted by the Montgomery Watson QAO or designee. The audits will include examining field measurement records, field equipment calibration records, field sampling records, field instrument operation records, sample collection procedures, sample handling and shipping procedures, and chain-of-custody records and procedures. Field activities will be audited early in the project to verify that all of the procedures outlined the QAPP are being followed. Follow-up audits will be conducted to correct deficiencies, and to verify that QA procedures are maintained throughout the project.

External field audits are the responsibility of the U.S. EPA Region V or IDEM. Field audits will be conducted at any time during the field operations and will be based upon the information present in the QAPP. The audits may or may not be announced at the discretion of U.S. EPA Region V or IDEM.

10.2 LABORATORY PERFORMANCE AND SYSTEMS AUDITS

In-house and regulatory agency audits of laboratory systems and performance are a regular part of both IEA's and Quanterra's QA program and are outlined in IEA's QA/QC Plan and the Quanterra Quality Assurance Management Plan. Internal audits are conducted by the laboratory's QAO and consist of a review of the entire laboratory system and at a minimum include: examination of sample receiving, log-in, storage, and chain-of-custody documentation procedures; sample preparation and analysis; and instrumentation procedures. Performance audits consist of the analysis of performance evaluation (PE) samples, which are supplied and evaluated by the U.S. EPA to ensure laboratory performance is acceptable.

External audits may be performed by Montgomery Watson, U.S. EPA Region V, or IDEM personnel prior to or during the field work, to verify proper implementation of laboratory procedures and adherence to this QAPP. These audits may or may not be announced and are conducted at the discretion of the auditing agency. External audits will include (but not be limited to) review of laboratory analytical procedures, laboratory on-site audits, and/or submission of PE samples to the laboratory for analysis.

11.0 PREVENTATIVE MAINTENANCE PROCEDURES

11.1 FIELD EQUIPMENT/INSTRUMENTS

The field equipment that will be used during this project will be maintained and used according to the manufacturers' directions. Each piece of equipment will be inspected on a regular basis to ensure that the equipment is operational. Any preventative maintenance or repair conducted in the field will be recorded in the field log book. Backup instruments and equipment will be available on-site or within one-day shipment to avoid delays in the field schedule.

Field instruments will be checked and calibrated before they are shipped or carried to the field, and will be checked and calibrated daily before use. Calibration checks will be performed after every 10 samples and will be documented in the field log books. Refer to Appendix E for field instrument use and calibration procedures.

11.2 LABORATORY EQUIPMENT

Both IEA and Quanterra have routine preventative maintenance programs to minimize the occurrence of instrument failure and other system malfunctions. Each laboratory has designated individuals who perform routine scheduled maintenance for each instrument system and required support activity. All maintenance is documented in instrument-specific maintenance log books, which are kept with the instrument. The date, initials of the individual performing the maintenance, and the type of maintenance are recorded in this logbook. Receipts from routine maintenance performed by the manufacturer's representative are filed in the appropriate laboratory department (i.e., ICP maintenance receipts are stored in the inorganic section). Refer to Attachment A for laboratory instrument use and calibration procedures.

Contingency Plan. In the event of instrument failure, every effort will be made to analyze samples within holding times by an equivalent alternate means. If redundancy in equivalent instrumentation is insufficient to handle the affected samples, the Montgomery Watson Project Manager will be notified immediately and the appropriate corrective action will be determined by the Laboratory and Montgomery Watson Project Managers.

12.0 DATA VALIDATION PROCEDURES

As described in Section 3.0, the quality of the field and analytical data will be evaluated using the PARCC parameters, which are quantitative and qualitative statements that describe data quality. The PARCC parameters will be used to determine whether the data quality objectives of this investigation have been met by comparing QC sample results and standard procedures with acceptance criteria established for this project. The PARCC parameters that will be used for data evaluation are defined in Section 3.0.

12.1 FIELD DATA

Field measurement data will be assessed by the Montgomery Watson QAO. The data quality evaluation, in terms of the PARCC parameters, will focus on Data Type B and C. There will be no formal data validation for Data Type A, which includes field data; however, the field data will be evaluated qualitatively in terms of the PARCC parameters as described in Section 3.0.

12.2 LABORATORY DATA

An independent validation of all Type B and C data will be conducted by Montgomery Watson based on the assumption that the samples were collected, handled, and analyzed according to this QAPP. The data reviewer will conduct a systematic review of the data for compliance with the QC criteria established in the QAPP, and will identify any data omissions or data that do not meet the quality control criteria. The reviewer also will interact with the laboratory to correct any data deficiencies. Decisions to repeat sample collection or analyses will be made by the Montgomery Watson Project Manager based on the extent of the data deficiencies and their importance in the overall context of the project. Results of the data validation will be presented in an appendix of the report scheduled to summarize the results of this investigation.

12.2.1. Laboratory Data Validation Procedures

All Type C data will be validated and qualified in accordance with the *USEPA Contract Laboratory Program National Functional Guidelines for Organic and Inorganic Data Review* (Functional Guidelines) (EPA, 1994). For all data from those methodologies not included in the Functional Guidelines (Data Type B), data validation will be based on the results of QC sample analysis, sample holding time evaluation, and the basic principles for data validation outlined in the EPA guidance (i.e., method blank contamination). As part of the data validation process, 10 percent of the total number of Data Type B and C data packages will be submitted to U.S. EPA Region V for an independent third party data validation. The QC data required for Type B and C data validation are listed in Table 3-1, and QC samples that will be used for data validation for each data type are listed in Table 3-2. The data qualifiers that will be used for this project are listed in Table 12-1.

As discussed previously, PARCC parameters will be used to validate the quality of analytical data and determine whether the DQOs of the project have been met. Table 3-3 depicts how the QC samples will be used to assess PARCC parameters.

TABLE 12-1

DATA QUALIFIERS

T	Analyte is detected above method detection limit, but below the practical quantitation limit.
R	Result is unusable.
UB	Analyte is not detected at or above the indicated concentration due to blank contamination.
UK	Analyte is not detected at or above the indicated concentration based on data evaluation.
UJ	Possible false negative result due to related QC problems.
J	Result is estimated based on MS/MSD or surrogate recoveries.
D	Indicates that the concentration was calculated using a secondary dilution factor (i.e., the result is calculated from the analysis performed by diluting the sample).

13.0 CORRECTIVE ACTIONS

Corrective action is the process of identifying, recommending, approving, and implementing measures to counter unacceptable procedures or out of quality control performance that may affect data quality. All proposed and implemented corrective action will be documented in the regular quality assurance reports to the appropriate Project Management. Corrective action will be implemented only after approval by the Project Manager or designee, and the field team leader. If immediate corrective action is required, approvals secured by telephone from the Project Manager will be documented in an additional memorandum.

For each noncompliance, a formal corrective action program will be established and implemented at the time the problem is identified. The person who identifies the problem will be responsible for notifying the Project Manager, who in turn will notify the U.S. EPA Remedial Project Manager (RPM). Implementation of corrective action will be confirmed in writing as described previously.

Any non-conformance with the established QC procedures specified in this QAPP or the FSP will be identified and corrected in accordance with the QAPP. The U.S. EPA RPM, or designee, will issue a non-conformance report for each such condition.

Corrective actions will be implemented and documented in the field log book. No staff member will initiate corrective action without prior communication of findings through the proper channels. If corrective actions are insufficient, the U.S. EPA RPM may issue a stop work order.

13.1 FIELD CORRECTIVE ACTION

During any field activity, the field staff will be responsible for documenting and reporting all suspected technical and QA non-conformances, and suspected deficiencies. The non-conformances and/or deficiencies will be documented in the field log book and reported to the Montgomery Watson Project Manager. If the problem is associated with field measurements or sampling equipment, the field staff will take the appropriate steps to correct the problem. Typical field procedures to correct problems include the following:

- Repeating the measurement to check for error
- Making sure the meters or instruments are adjusted properly for the ambient conditions, such as temperature
- Checking or replacing batteries
- Recharging batteries
- Recalibrating the instruments
- Replacing the meters or instruments used to measure field parameters
- Stopping work until the problem is corrected (if necessary).

If a non-conformance or problem requires a major adjustment to the field procedures as outlined in the QAPP or FSP (e.g., changing sampling methodology), the Project Manager, in conjunction with the U.S. EPA RPM and QAO, will be responsible for initiating corrective actions. The Montgomery Watson's Project Manager will be responsible for:

- Evaluating the reported non-conformance
- Controlling additional work on non-conforming items
- Determining the appropriate corrective actions in conjunction with the U.S. EPA RPM
- Maintaining a log of all non-conformances and corrective actions
- Approving all changes in writing or verbally prior to field implementation, if feasible. If deemed unacceptable, the action taken during the period of deviation will be evaluated to determine the significance of any departure from established program practices and action taken

- Ensuring that explanation of non-conformances and corrective actions is included in an appendix of the report scheduled for this investigation

- Ensuring that no additional work that is dependent on the non-conforming activity is performed until the appropriate corrective actions are completed

- Reporting all changes to all affected parties, including the U.S. EPA RPM

13.2 LABORATORY CORRECTIVE ACTION

Corrective actions are required whenever unreliable analytical results prevent the quality control criteria as specified by the method or the laboratory QAPP from being met. The corrective action that is taken depends on the analysis and the non-conformance. Appendix B provides a summary of the corrective actions that will be undertaken for problems associated with specific laboratory analyses.

Corrective actions will be undertaken if one of the following occurs:

- QC data are outside the acceptance criteria for precision and accuracy
- Blanks contain contaminants above acceptance levels
- Undesirable trends are detected in spike recoveries (or spike recoveries are outside the QC limits), or RPDs between duplicate analyses are consistently outside QC limits
- There are unusual changes in detection limits
- Deficiencies are detected during QA audits
- Inquiries concerning data quality are received from Montgomery Watson.

Corrective actions are handled primarily at the bench level by the analyst who reviews the sample preparation or extraction procedures, and performs the instrument calibration and analysis. If the problem persists or its cause cannot be identified, the matter will be referred to the department supervisor or QA department for further investigation. Once resolved, full documentation of the corrective action procedure will be filed with the QA

department. A summary of the corrective actions will be included in the data package submitted to Montgomery Watson.

13.3 DATA VALIDATION CORRECTIVE ACTION

Corrective action may be initiated during data validation or data assessment. Potential types of corrective action include requesting resampling by the field team or reinjection/reanalysis of samples by the laboratory.

These actions are dependent upon the ability to mobilize the field team, how critical the data are to the project DQOs, or whether the samples are still within holding time criteria. When the data validator identifies a corrective action situation, the Montgomery Watson Project Manager will be notified and has final responsibility for authorizing the implementation of the corrective action, including resampling. All corrective actions of this type will be documented by the QAO.

14.0 QUALITY ASSURANCE REPORTS TO MANAGEMENT

Deliverables associated with the tasks identified in this QAPP will contain separate QA sections in which data quality information collected during the task is summarized. Those reports will be the responsibility of the Project Manager and will include the QAO's report on the accuracy, precision, and completeness of the data as well as the results of the performance and system audits, and any corrective action needed or taken during the project.

14.1 CONTENTS OF PROJECT QA REPORTS

The QA reports will contain all results of field audits, all information generated during the past month, specific data quality objectives, and a summary of corrective actions implemented and their effect on the project. The status of the project with respect to the Project Schedule (Section 1.0) also will be evaluated in the report. Whenever necessary, updates on training, changes in key personnel, and anticipated problems in the field or lab that will impact data quality, and proposed solutions will be reported. Detailed references to QAPP modifications will also be highlighted. All QA reports will be prepared in written, final format by the Project Manager or designee.

In the event of an emergency, or if it is essential to implement corrective action immediately, QA reports will be made by telephone to the appropriate individuals, as identified in the Project Organization (Section 2.0) or Corrective Action (Section 13.0) sections of this QAPP. However, these events, and their resolution also will be addressed in detail in the next monthly QA report.

14.2 FREQUENCY OF QA REPORTS

The QA Reports will be prepared bi-monthly and will be delivered to all recipients by the end of the first full week of the month. The reports will continue without interruption until the project has been completed. The frequency of emergency reports that must be delivered verbally cannot be estimated at the present time.

14.3 INDIVIDUALS RECEIVING/REVIEWING QA REPORTS

All individuals identified in the Project Organization chart will receive copies of the monthly QA report.

REFERENCES

U.S. EPA, 1983. Methods for Chemical Analysis of Water and Wastes, EPA Manual, 600/4-79-020 (USEPA, 1983, with additions).

U.S. Environmental Protection Agency, 1992. Record of Decision American Chemical Service Site, Griffith, Indiana.

U.S. Environmental Protection Agency, 1992. EPA Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW-846, 3rd Edition.

U.S. Environmental Protection Agency, 1992. U.S. EPA Specifications and Guidance for Obtaining Contaminant-Free Sample Containers.

U.S. Environmental Protection Agency, 1994. USEPA Contract Laboratory Program National Functional Guidelines For Organic and Inorganic Data Review.

U.S. Environmental Protection Agency, 1994. Guidance for Conducting Treatability Studies.

U.S. Environmental Protection Agency, 1995. Region V Superfund Model Quality Assurance Project Plan (May 1996, Revision 1).

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ATTACHMENT A

LABORATORY STANDARD OPERATING PROCEDURES (SOPs)

ATTACHMENT A

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1. FIELD ASSAY FOR PCBs

OHMICRON RAPID ASSAY TEST METHOD

PCB FIELD TEST PROCEDURE ADDENDUM

Ohmicron RaPID Assay Test Method

The Ohmicron RaPID Assay Method will be used for the field analysis of PCBs in soil samples for the Barrier Wall Alignment Investigation at the ACS NPL Site. The analysis is based on the immunoassay method and as such, the test kits yield only screening-level data. The purpose of the field analysis is to screen soil samples in the field to identify samples with PCB concentrations in excess of 10 ppm, and then submit these for laboratory analysis.

The Ohmicron Field Assay Test Method will be calibrated for a detection limit of 10 parts per million (ppm) PCB for Aroclor 1254, using Method 2, listed on page 4.4 of the User's Guide. Aroclor 1254 was selected because it was the congener that made up nearly 40 percent of the sample detections at the site during remedial investigation sampling and because the Ohmicron system is based on Aroclor 1254.

A supply of sodium sulfate will be maintained in the field laboratory to be used to dry samples as appropriate.

The analytical technician will be trained in the use of the test kit, and training will also be available for the Agency representatives on site during the Barrier Wall Alignment Investigation.

QA/QC for Field Analysis Screening Analysis of PCBs at the ACS NPL Site

Quality Assurance/Quality Control will be conducted at Level QA2, described on Page 3.2 of the User's Guide and detailed on page 3.4 and 3.5. The "*Other QA/QC Considerations*" listed on Page 3.5 will also be given consideration. As stated on Page 5 of the Work Plan, a performance evaluation sample with a known concentration will be analyzed, prior to starting the field work. Field duplicates will be collected to provide assessment of field sampling precision, accuracy, and integrity of the methodology. At least one duplicate analyses will be performed for each set of ten soil samples. Furthermore, soil samples that exceed 10 ppm PCBs based on field screening will be submitted for laboratory analysis to determine if the PCBs are actually present. Laboratory analyses will be performed in accordance with the Contact Laboratory Program, at Data Quality Objective Level 3.

Field Validation

Field Validation will be conducted throughout the Barrier Wall Investigation sampling, following the Guidance in the "*Immunoassay Methods for SW-846: Recommended Format*

and Documentation for New Submittals" (July 1995). As part of the process, standards and proficiency samples will be analyzed with each sample analysis run. For the ACS project in which the detection limit will be set at 10 ppm PCBs, six standards/proficiency samples will be analyzed with each set of analyses. These will include known standards with concentrations of: 2.5 ppm, 5 ppm, 10 ppm, 15 ppm, 40 ppm, and 50 ppm. In addition, all samples that indicate a detection of PCBs at 10 ppm or greater, will be submitted for laboratory analysis. This will result in 100 percent field validation of positive detections for PCBs at greater than 10 ppm.

A copy of the "*RaPID Assay Environmental User's Guide*" follows. The sections that are not applicable to the ACS Site RD/RA have been removed, and those sections have been lined out in the attached Table of Contents.

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RPA-I™ RaPID Photometric Analyzer

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(800) 544-8881
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Rev. 1/92

Part No. A00046

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SECTION 1 - RPA-I™: DESCRIPTION

SPECIFICATIONS

Wavelength Range: 340-650 nm
Supplied filter block 450/600nm
For a list of all the available filter blocks see Section 7 Accessories.

Photometric Stability: Drift ≤0.005 A per hour at 0 A.

Linearity: ≤0.005 A or 2% difference from calculated regression line. Correlation coefficient, r=0.9995 or better.

Photometric Range: 0 A to 2 A.

Reproducibility: A standard deviation of ≤0.005 A when measured with 20 samples of distilled water.

Microprocessor: 8085, 128K memory.

Keypad: Key pad with 20 numeric and special function keys.

Liquid Crystal Display: Two line, 16 characters each line.

Printer: 28 Character, thermal printer.

Read Time: < 1 second per tube.

Sample Volume: Minimum 1.0 mL.

Tube Size: 12mm glass or polystyrene test tubes.

Power Requirements:
Voltages: 100/120 or 220/240 VAC (+10%, -15% of normal line voltage setting).

Frequency: 50 or 60 cycles per second.

Volts Amps: 40 VA max.

Power Connection: Three conductor grounded outlet.

Size: 45cm W x 34cm H x 15cm D.
(17.5" W x 13.5" H x 6" D).

Weight: 6.8 kg (5 lbs.).

Light Source: Tungsten Quartz-Halogen.

Optics: Bichromatic.

The RPA-I Photometric Analyzer™ (Part No. A00003), Figure 1, is a discrete wavelength bichromatic photometer with an overall wavelength range of 340 nm to 650 nm.

- Differential absorbance of the final assay solution is measured and converted into final concentration values for all Ohmicron RaPID Assays® by use of the on-board microprocessor. The microprocessor also has extensive data reduction capabilities for handling a variety of curve fits.
- Programmed information is provided which facilitates the performance of the RaPID Assays® protocols. These programmed parameters can be customized or new protocols can be added. Space is provided for 22 protocols.
- Uniquely designed optics are used which allow readings to be made through standard, disposable test tubes.
- A heater is built into the sample compartment which, when activated, can provide a temperature of 37°C.
- Stores quality control data at the end of each assay under each protocol title.

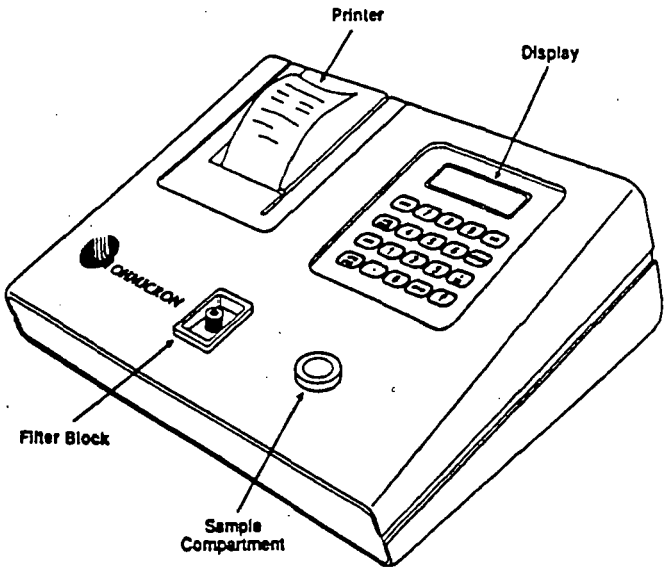


Figure 1. RPA-I™ Photometric Analyzer

FEATURES

Figure 1 shows the key features of your photometer. The main controls for routine operation are the Power Switch (see Figure 2) and the keypad (see Figure 3).

FILTER BLOCK:

- The filter block consists of a beam splitter and two narrow band interference filters set for the primary and reference wavelengths. It is located in the center of the RPA-I™ (Figure 1). Since the filter block is changeable, it is up to the user to make sure the proper filter block is installed at run time. However, the primary filter wavelength must be programmed into the protocol (Section 3 Options - Enter/Edit Protocol) to allow the photometer to select the correct gain setting.
- When it is necessary to change filter blocks, simply grasp it at the top and pull straight up. To install a filter block, insert it into the slot and position it firmly on the bottom. The filter block only fits one way into the slot. When not in use, store extra filter blocks in a dry, clean, lint-free location to avoid damage.

LIQUID CRYSTAL DISPLAY:

The liquid crystal display presents a series of prompts to guide the operator in creating protocols, or analyzing test results. The top line displays the prompt, and the bottom line displays the operator's input.

PRINTER:

The printer is a 28 column, parallel thermal printer which provides a permanent record of each assay run or assay protocol. Photocopy the printer tape to prevent its fading over time. This bi-directional device prints up to 28 characters per line.

DATA PROCESSOR:

The RPA-I™ has a microprocessor consisting of 128K of pre-programmed and operator programmable information. The program for the microprocessor is stored in a removable cartridge located in the rear panel of the photometer (see Figure 2). This feature allows software to be updated by the operator upon receipt of new cartridges. The microprocessor manipulates and stores the appropriate data during analytical processing.

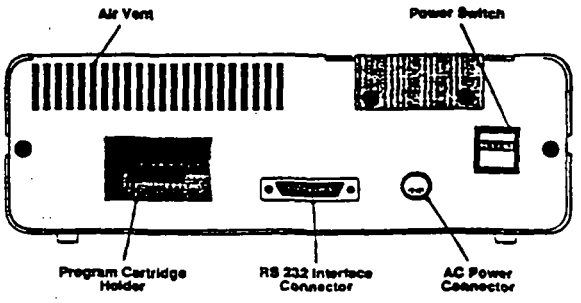


Figure 2. Rear Panel

MEMORY BACKUP:

The data reduction algorithms are contained in EPROM within the program cartridge. The instrument calibration, control data, user defined protocols, and Ohmicron protocols are maintained in battery backed up RAM within the instrument and never lost when power is disconnected. This backup system can maintain data storage for the life of the instrument.

KEYPAD:

The instrument is controlled by the operator ... key pad. Numerical keys are ... with special function keys ... operations, enter or delete ... access special functions of the photometer.

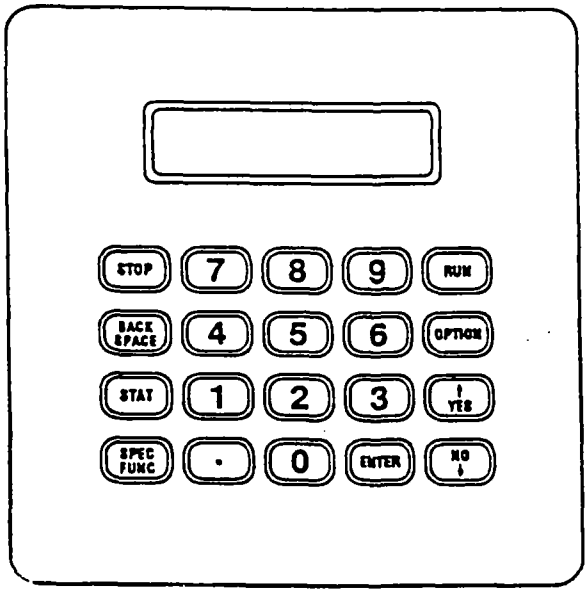


Figure 3. Keypad

[RUN]
The [RUN] key permits the operator to make the instrument perform the test protocols that are in memory. These protocols have the capability of automatically obtaining reportable results from samples which are processed. Data reduction is automatic, and an actual plot of the curves can be printed upon request by the operator (See Section 3 Special Functions - Instrument Functions: Print Curve). Quality Control data can be stored for each protocol and may be retrieved or reviewed as needed by the operator (See Section 3 Options - List Control Data).

[↑] YES
This key is a combined "up scroll" key and "YES" key. The up scroll key [↑] may be used to scroll quickly upward through any menu or protocol. This key can be used whenever the symbol [< >] appears in the top right hand corner of the display. Pressing the key allows the operator to scroll through various options which appear on the bottom line of the display. Once the required option is shown on the display, it can be entered in the instrument by pressing the [ENTER] key. This key also acts as a "YES" key whenever a question is asked which requires a YES/NO answer.

[↓] NO
The down scroll key [↓] is used to scroll quickly downward through any protocol. This key is basically the opposite of the [↑] key and enables the operator to move in the opposite direction through a menu. This key also acts as a "NO" key whenever a question is asked which requires a YES/NO answer.

[OPTION]
Pressing the [OPTION] key gives access to the part of the program in which the operator can create or make changes to assay protocols (See Section 3 Options). Pressing this key allows selection of other instrument functions that include "Enter/Edit Protocol", "Delete Protocol", "List Protocol", "List Protocol Name", and "List Control Data".

[STOP]
Pressing the [STOP] key at any time during an operation aborts the operation in progress including the printer and returns the instrument to the "Select Command" mode.

[BACKSPACE]
The [BACKSPACE] key allows the operator to delete erroneously entered letters or numbers. Each digit or letter will be deleted as the cursor "backspaces" under them. Re-type the appropriate characters as needed. It also allows the operator to step back through certain portions of a program.

[ENTER]
The [ENTER] key is used to enter any command which is shown on the display. It will also answer "YES" to any question which requires a YES/NO answer. By repeatedly pressing this key the operator can "scroll" through a menu.

[SPEC FUNC]
The [SPEC FUNC] key allows selection of the various "special functions". See Section 3 Special Functions, for details of these functions.

[STAT]
The [STAT] key allows samples to be instantly processed without generation of a new standard curve. See Section 3 Stat, for details of this function.

HEATER:
The heater, located within the sample compartment, can provide a temperature of 37°C when activated.

TRANSFORMER:
The RPA-I™ is powered by an appropriate transformer supplying 18 volts at 40 VA. The instrument's AC power transformer will be shipped from the factory with proper voltage appropriate for the operator's geographic location. This transformer should be verified by the operator by inspecting the labeled voltage rating before use.

RS 232 INTERFACE CONNECTOR:
Refer to Figure 2 for location of the RS 232 port. To send data out the external RS 232 port (with or without the printer in operation), the operator must first select this port through the Special Function menu (see Section 3 Special Functions - Instrument Functions: Assign Output). When this mode is enabled, all data normally sent to the printer will be routed out the serial port. This includes all header and format characters, as well as data. The Baud rate is internally set for 9600, 8 bit work length, 1 stop bits, and NO parity.

POWER SWITCH:
The instrument is activated by depressing the top of the power switch located on the rear panel (see Figure 2). The instrument will perform a "Self Test" which automatically analyzes the electronic components of the RPA-I™. This includes checks of EPROM and RAM memory. If there are any abnormalities in these areas, the operator will be alerted with an "Error" message. If all the parameters are satisfactory, the "Select Command" prompt will appear and the operator may continue. The RPA-I™ should warm up for 30 minutes prior to use.

PHOTOMETER:
The photometer portion of the instrument consists of a tungsten halogen lamp, an optical reading station, a bichromatic filter block, solid state detectors, and circuitry which allows light readings to be converted into digital readout.

SECTION 2 - INSTALLATION

ENVIRONMENT

- 5° C to 33° C
- 10% to 85% humidity
- Flat, level surface away from strong sources of electromagnetic interference.
- No direct sunlight or drafts.
- Removed from sources of direct heat and moisture.
- Ventilation space at least 6 inches on sides and back.

INSTALLATION

1. Inspect the carton for visible signs of damage and note the condition of the SHOCKWATCH indicator on the side of the carton. If damage has occurred, or a part is missing, immediately contact Ohmicron.

2. Open the carton and remove the brown rectangular box from the grey packing material. (Save all boxes.) This box contains the power transformer, roll of paper, and Program Cartridge. Refer to Figure 4 for identification of shipping carton contents.

3. Lift off the gray packing material to reveal the photometer. Remove it from the carton.

4. With the power OFF to the instrument, (bottom of the white toggle power switch should be depressed) insert the round end of the Power Transformer (notched end facing up) into the AC Power Connector found on the rear panel (Figure 2) of the instrument. Plug the square end of the power cable into a grounded AC outlet.

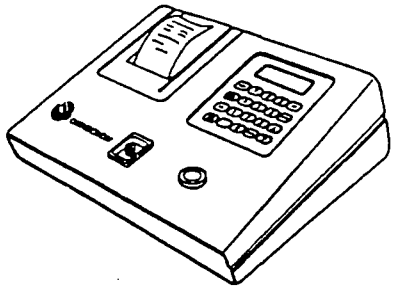
5. Insert the Program Cartridge (with the white label facing up) into the Program Cartridge Holder found on the rear panel (Figure 2) of the instrument. Push in until the white label is no longer visible. The instrument is then activated by depressing the top of the white toggle power switch. The instrument will perform a "Self Test." During this short test, the various electronic components of the RPA-I™ are automatically analyzed. This includes checks of EPROM and RAM memory. If there are any abnormalities in these areas, the RPA-I™ will alert the operator with an "Error" message. If all the parameters are satisfactory, the "Select Command" prompt will appear and the operator may continue.

6. Proceed to the Short Operating Instructions for the RPA-I™ found in Section 4.

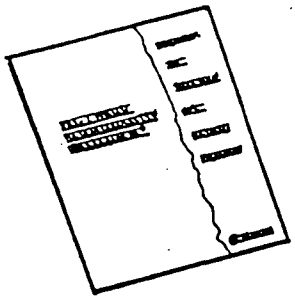
SHIPPING CARTON CONTENTS

The shipping carton should contain the following items:

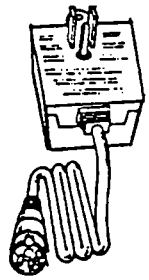
RPA-I Photometric Analyzer™ with a 450/600nm filter block.



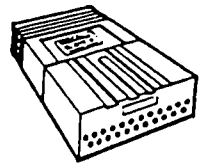
RPA-I Photometric Analyzer™ Operator's Manual



Domestic Power Cord/Mains Transformer



Program Cartridge



Printer Paper

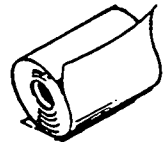


Figure 4. Shipping Carton Contents

SECTION 3 - OPERATING PROCEDURES / MENUS

RPA-I™ functions are menu driven in a top-down hierarchical manner. The top level is controlled by the keys, [RUN] and [OPTION]. After pressing one of these two keys, the [↑] [↓] scroll keys allow the operator to scroll through the options at that level. When the desired option comes into view, pressing the [ENTER] key selects that option.

NOTE - Convention for this manual: all instrument messages will be shown in quotes " " and special function keys will be shown in brackets [ ].

RUN

The RPA-I™ is provided pre-programmed with the recommended protocol for running all current RaPID Assays® procedures. These protocols can be accessed and run as described in the RPA-I™ SHORT OPERATING PROCEDURE which can be found on the front inside Section 4. The RaPID Assays® protocols may be edited as described in Section 3 Options - Enter/Edit Protocol, but such changes may effect the performance of the assay. Refer to the particular RaPID Assays® package insert for additional details on performing the individual procedures and their associated data reduction. To run RaPID Assays®, press [RUN] and follow the RPA-I™ SHORT OPERATING PROCEDURE.

EDIT CALIBRATORS:

When running a protocol, the RPA-I™ will proceed to prompt for the calibrators (standards), controls, and samples according to the program. These tubes must be read in the appropriate order. The instrument will pause after the controls have been run, and ask the operator "Edit Calibrators?" At this point, the operator may manually manipulate a standard curve that is unacceptable due to poor coefficients of variation (> 10%) or correlation coefficients (< 0.990). The operator may respond YES or NO. If YES, the instrument will offer the operator five choices [< >]:

1. Delete a Point
2. Restore a Point
3. Rerun a Point
4. Manual Entry
5. Continue Run

DELETING/RESTORING A POINT:

In deleting calibrator points, scroll with the [↑] [↓] keys until "Delete a Point" appears. Press [ENTER]. The instrument will then ask which calibrator number, and which replicate (if applicable). After the editing is completed, the instrument will display "Edit Calibrator" on the top line and "Delete a Point" on the bottom line of the display. Scroll to Continue Run, press [ENTER] and the RPA-I™ will print out the new calibrator information and the new standard curve. Notice the word "deleted" next to the deleted point. The RPA-I™ will then prompt for the control samples then display Edit Calibrators. If you want to continue editing the curve, Press [↑] YES. If you want to continue with your assay Press [↓] NO and the RPA-I™ will ask you to insert sample 1 replicate 1, etc. When the last sample has been assayed, press the [STOP] key to end the assay. To restore a point, follow the above procedure selecting "Restore a Point" after the prompt "Edit Calibrators".

RERUN A POINT:

To rerun a point, scroll with the [↑] [↓] keys until "Rerun a Point" appears. Press [ENTER]. The instrument will then ask which calibrator number, and which replicate (if applicable). After the tube is reread, the instrument will display "Edit Calibrator" on the top line and "Rerun a Point" on the bottom line of the display. Scroll to "Continue Run", press [ENTER] and the RPA-I™ will print out the new calibrator information and the new standard curve. The RPA-I™ will then prompt for the control samples then display Edit Calibrators. If you want to continue editing the curve, Press [↑] YES. If you want to continue with your assay Press [↓] NO and the RPA-I™ will ask you to insert sample 1 replicate 1, etc. When the last sample has been assayed, press the [STOP] key to end the assay.

MANUAL ENTRY:

To manually enter absorbance values obtained from another instrument, scroll with the [↑] [↓] keys until "Manual Entry" appears. Press [ENTER]. The instrument will then ask which calibrator number. Then the RPA-I™ will display the most recent absorbance reading for the selected calibrator. Your new absorbance

reading may be entered here with the keypad numbers. Press [ENTER] when finished. Scroll to "Continue Run" to have the new calibrator information and standard curve printed out. An "M" will appear beside the absorbance values that were manually entered. The RPA-I™ will then ask to re-edit the calibrators. Press [STOP] to end the assay.

CONTINUE RUN:

This function causes a new standard curve to printout after any of the above manipulations have been performed and allows the operator to continue with the analysis of sample.

OPTIONS

To access the contents of this menu, press the [OPTION] key on the top right side of the keypad. This menu includes:

a. Enter/Edit Protocol
b. Delete Protocol
c. List Protocol
d. List Protocol Name
e. List Control Data

ENTER/EDIT PROTOCOL:

The Enter/Edit Protocol menu allows users to either selectively edit existing or create new protocols. To access this function, press the [OPTION] key and scroll through until "Enter/Edit Protocol" appears. Press the [ENTER] key. The selection of which protocol to Enter/Edit is made by selecting the name of the protocol using the scrolling arrow keys.

When entering a new protocol, begin by pressing the [ENTER] key when "Unused" has been scrolled to the name position of the display. The number next to the word Unused will indicate how many blank protocols remain to be programmed. When NO more remain, a protocol must be deleted before a new one can be entered. The instrument will then lead the user through each of the protocol variables to edit or enter the appropriate information. Be sure to press [ENTER] after each selection. Once the protocol has been fully Entered/Edited, the user will have the option to print a listing of the new or revised protocol.

The RPA-I™ has alpha-numeric capability. The "alpha" capability is used for protocol names or units when entering or editing a protocol. In either of these modes, pressing the [↑] or [↓] key allows the operator to scroll through a complete alphabet of letters and symbols. Numbers are entered via the numeric keys, 0-9. Once the correct letter, number, or symbol is shown in the display, press [ENTER]. This moves the cursor to the next position and allows the next letter to be selected. When the work is complete, press [ENTER] one more time. This will enter the word into the appropriate memory location.

The values and their ranges to be defined in the protocol menu are as follows:

Instrument Display	Operator Response
"PROTOCOL NAME" NO	Enter name by using the [↑] YES and [↓] keys to choose the letters of the alphabet. Press [ENTER] after each entered letter (max. 8 letters).
"DATA REDUCT." See Section 5 of the manual for further explanation.	Select: Lin. Regression, Point to Point, Polygonal, Cal. Factor, Qualitative, or Single Point.
"XFORM OPTION" See Section 5 of the manual for further explanation.	Select: Ln/LgtB, Linear/Linear, Linear/Ln, Linear/Logit, Linear/(1/Resp.), Ln/Linear, Ln/Ln, Ln/Logit, Ln/(1/Response).
"# OF CALIBRATORS:" (2-8):	Enter a # between 2-8 (including the 0 ppb standard) which represents the # of standard concentration (ppb) levels to be used for construction of a standard curve.
"CAL. REPLICATES:" (1-3):	Enter a # between 1-3 which represents the # of tubes to be run for each standard level.

"CAL. #1 CONC:" 0.00	For the concentration of the lowest standard, enter a real number (usually 0 ppb for RaPID Assays®). Enter standard levels in ascending order of concentration.
"CAL. #2 CONC:" 0.00	For the concentration of the next higher standard, enter a real number.
Etc.	
"CORRELATION FLAG" (0.0-1.0):	Coefficients of correlation calculated for the standard curve linear regression will be flagged (*) if less this value. Enter 0.0-1.0 (usually >0.990 for RaPID Assays®).
"UNIT(eg:UG/M)"	Enter up to 6 digits/letters. All concentrations of standards, controls, and samples will be printed with these units (usually ppb for RaPID Assays®).
"NORMAL RANGE HI"	Enter a real number. Sample Concentrations greater than this value will be flagged as "Hi".
"NORMAL RANGE LOW" (0.0-100.0):	Enter a real number. Sample Concentrations less than this value will be flagged "nd" (none detected).
"# OF CONTROLS:" (0-3):	Enter a # between 0-3 which represents the # of controls that will be included. Values obtained will be stored. See Section 3 Options - List Control Data.
"CTRL REPLICATES:" (1-3):	Enter a # between 1-3 which represents the # of tubes to be run of each control.
"# OF RGT BLANKS:" (0-3):	Enter 0-3. The value obtained will be subtracted from all readings.
"WAVELENGTH" (340-650):	Enter 340-650 nm. The correct wavelength is required for automatic application of the correct electronic gain setting.
"READ MODE"	Select Absorbance (See Section 3 Special Functions - Read Absorbance) or Kinetic (See Section 3 Special Functions - Kinetic Test).
"REP. %CV FLAG:" (1-100):	Enter 1-100% Absorbance. % coefficent of variation of each standard level is calculated (usually less than or equal to 10% for RaPID Assays®).
"LIST PROTOCOL?" (Yes/No):	Press [↑] YES or [↓] NO keys.

DELETE PROTOCOL:

Stored protocols may also be deleted. To access this function, press the [OPTION] key and scroll through with the [↑] [↓] keys until "Delete Protocol" appears. Press the [ENTER] key. Next, scroll through the names of the stored protocols. Once the desired protocol appears, press [ENTER] to delete. As a safety feature, the RPA-I™ will ask a second time if you wish to delete the particular protocol. Simply press the [ENTER] or the [↑] "YES" key.

LIST PROTOCOL:

To access this function, press the [OPTION] key and scroll through with the [↑] [↓] key. until "List Protocol" appears. Press the [ENTER] key. Next, scroll through the names

of the stored protocols. Once the desired protocol appears, press [ENTER] to list. The following is an example of the printout obtained.

```
08-05-91  13:54:07

******** OHMICRON ********

PROTOCOL :  ALACHLOR

TECH ID :_____________
LOT #   :_____________
EXP DATE:_____________

Data Reduct:Lin.Regression
Xformation:          Ln/LgtB
Read Mode  :      Absorbance
Wavelength :          450 nm
Units      :             PPB

Calibrators:
------------
# of Cals  :               4
# of Reps  :               2

Concentrations :
---------------
  #1:        0.00         PPB
  #2:        0.10         PPB
  #3:        1.00         PPB
  #4:        5.00         PPB

Range      :  0.05 -  5.00
Correlation:          0.990
Rep. %CV   :           10 %

Controls   :
------------
# of Ctrls :               1
# of Reps  :               1

END OF LISTING
```

LIST PROTOCOL NAME:

To access this function, press the [OPTION] key and scroll through with the [↑] [↓] keys until "List Prot. Name" appears. Press the [ENTER] key. The RPA-I™ will print out the names of the protocols that have been programmed into its memory along with the number of available protocols that can be entered.

LIST CONTROL DATA:

The RPA-I™ has the ability to store control information about each protocol. Up to three controls can be used/stored with each assay. At the end of each assay run, the RPA-I™ will add the current control values to the running mean stored in memory. This mean consists of the previous values obtained for each particular control. The current value of this running mean can be obtained by pressing [OPTION] and scrolling through with the [↑] [↓] keys until "List Ctrl. Data" appears. Press the [ENTER] key. Here the RPA-I™ will ask for the assay desired and particular control value requested. Scroll through the names of the protocols with the [↑] [↓] keys. Once the desired protocol appears, press [ENTER]. After the control values are printed, the instrument will ask "Clear Control Data?" This allows the operator to keep the running mean current or delete the information, i.e., if a new control is being used.

STAT

In order to run a protocol in the "STAT" mode, press the [STAT] key. The instrument will then offer the protocol menu. Use the [↑] [↓] keys to select the appropriate protocol. There is no need to use calibrators when running an assay in this mode; however, a new control must be prepared. The RPA-I™ will use the latest calibration data for that protocol from its memory. It will use that information for data reduction. The RPA-I™ will also indicate the date and time at which the calibrators were last run.

SPECIAL FUNCTIONS

To access the contents of this menu, press the [SPEC FUNC] key on the bottom left side of the keypad. This menu includes:

a. Linearity Test
b. Tube Repeatability
c. Read Absorbance
d. Kinetic Test
e. Instrument Functions - This has its own menu consisting of:
    1. Set Clock Time
    2. Assign Output
    3. Set Absorbance
    4. Print Curve
    5. Print Headings
    6. Heater

LINEARITY TEST:

This function provides a quality control check on the linearity of response of the photometer portion of the RPA-I™. Recommended methods for performance of a Linearity Test included the following two techniques:

1. Prepare five equally spaced dilutions of potassium dichromate or methyl orange that cover the absorbance range of the immunoassay, i.e., 0.250 - 1.500. The absolute absorbance values are not critical. The intent of the

experiment is to make sure the optical system responds in a straight line fashion.

2. Prepare five dilutions of potassium dichromate or methyl orange at 0.25A, 0.5A, 1.0A, 1.5A, and 2.0A. Verify these absorbances on a reference spectrophotometer. Read the same tubes on the RPA-I™. The absorbances should match.

Once the standards have been prepared as described above press [ENTER] when Linearity Test is displayed.

Instrument Display	Operator Response
"NUMBER OF TUBES: (2-8)"	Enter the number of standards you have.
"TUBE #1 CONC: 0.000"	Enter the concentration values for each of the standards in ascending order.
"WAVELENGTH (340-650)"	Choose the wavelength needed.
"BLANK TUBE" "INSERT TUBE" "EVALUATING TUBE" "REMOVE TUBE" (Beep)	Insert tube with 1 ml of distilled water. Remove tube.
"STD #1" "INSERT TUBE" "EVALUATING TUBE" "REMOVE TUBE" (Beep)	Insert the first Standard tube. Remove tube.

After all the standards have been evaluated, the instrument will display:

"Printing Data"

Press [STOP] when the instrument is finished printing. Below is an example of the printout obtained.

```
Linearity Test

Wavelength :          450 nm

Tube       Abs
------------------
  1       0.002
  2       0.184
  3       0.361
  4       0.548
  5       1.153
  6       1.656

EQUATION OF LINE :
------------------
Slope     =     1.033
Intercept =    -0.010
Corr (r)  =    0.9997

  Conc Predic   Diff %Diff
-------------------------
 0.000  0.011  0.011 100.0
 0.183  x.187  0.004   2.2
 0.363  0.358 -0.005  -1.3
 0.548  0.540 -0.008  -1.5
 1.146  1.125 -0.021  -1.8
 1.594  1.612  0.018   1.1
-------------------------


END OF RUN
```

TUBE REPEATABILITY:

In this function, the RPA-I™ calculates the Mean, the Standard Deviation (SD) and the Coefficient of Variation (%CV) on a series of tubes. To run this mode, press [ENTER] when Tube Repeatability is displayed.

Instrument Display	Operator Response
"NUMBER OF TUBES:"	Enter the number of tubes to be analyzed.
"WAVELENGTH (340-650):"	Choose the wavelength needed.
"BLANK TUBE" "INSERT TUBE" "EVALUATING TUBE" "REMOVE TUBE" (Beep)	Insert tube with 1 ml of distilled water. Remove tube.
"TUBE #1" "INSERT TUBE" "EVALUATING TUBE" "REMOVE TUBE" (Beep)	Insert the first sample tube. Remove tube.

After all the tubes have been evaluated, the instrument will display:

"Printing Data"

Press the [STOP] when the instrument is finished printing. Below is an example of the printout obtained when evaluating 10 tubes.

```
Tube Repeat

Wavelength :          450 nm

Tube        Abs
------------------
  1       1.124
  2       1.116
  3       1.111
  4       1.088
  5       1.096
  6       1.113
  7       1.111
  8       1.109
  9       1.108
 10       1.106

MEAN  :       1.1081
SD    :       0.0100
%CV   :       0.9031
```

READ ABSORBANCE:

This mode allows the absorbance values to be read on individual samples. To run this mode, press [ENTER] when the "Read Absorbance" prompt appears.

Instrument Display	Operator Response
"WAVELENGTH (340-650):"	Choose the wavelength needed.
"BLANK TUBE" "INSERT TUBE" "EVALUATING TUBE" "REMOVE TUBE" (Beep)	Insert tube with 1 ml of distilled water. Remove tube.
"TUBE #1" "INSERT TUBE" "EVALUATING TUBE" "REMOVE TUBE" (Beep)	Insert the first sample tube. Remove tube.

After all the tubes have been evaluated and the instrument has finished printing, press [STOP]. Below is an example of the printout obtained when reading 10 tubes.

```
Read Absorbance

Wavelength :          450 nm

Tube        Abs
------------------
  1       1.374
  2       1.205
  3       0.661
  4       0.276
  5       0.385
  6       0.311
  7       2.078
  8       0.802
  9       1.354
 10       0.312
```

KINETIC TEST:

This mode allows the absorbance of a tube to be read continuously at specified time intervals. To run this mode, press [ENTER] when the "Kinetic Test" prompt appears.

Instrument Display	Operator Response
"WAIT TIME (MIN.): (0-60):"	Enter a time from 0 to 60 minutes to indicate the time between readings.
"WAVELENGTH (340-650):"	Enter the wavelength needed.
"BLANK TUBE" "INSERT TUBE" "EVALUATING TUBE" "REMOVE TUBE" (Beep)	Insert tube with 1 ml of distilled water. Remove tube.
"TUBE #1" "INSERT TUBE" "EVALUATING TUBE" "PRINTING DATA" "WAITING: 0.00 MIN" "TIME LEFT: 0.00"	Insert first sample tube.
"READING TUBES.." "EVALUATING TUBE" "PRINTING DATA" "WAITING: 0.00 MIN" "TIME LEFT: 0.00"	The readings will continue until the wait time has expired.

Press [STOP] to halt the readings. Below is an example of the printout when reading a kinetic reaction for 7 minutes.

```
Kinetic Test

Wait Time  :          1 min
Wavelength :        450 nm

Tube        Abs
------------------
  1       0.022
  2       0.032
  3       0.068
  4       0.128
  5       0.250
  6       0.489
  7       0.987
```

INSTRUMENT FUNCTIONS:

To access this menu, press [ENTER] when the prompt "Instrument Functions" appears on the display. After accessing this function, as described above, the operator may scroll through the menu by using the [↑] or [↓] keys. When the desired option is displayed, press [ENTER] to access a particular section.

Set Clock Time

This mode allows the operator to view or set the clock time. The current date and time appear on the bottom line of the display while MM-DD-YY HH:MM appears on the top line. To input a new date and time, enter the appropriate numbers and use the backspace key to correct digits in error. Press [STOP] to complete the editing. The clock time is maintained, even when the power switch is in the "OFF" position.

Assign Output

The assign output device option will allow three output options [< >]:

[1] Printer
[2] RS-232C Serial Port
[3] Printer and RS-232C Serial Port

On the rear panel of the RPA-I™ is a port where an RS-232C cable can be connected. See Figure 2. Use option [2] to use ONLY this output device. If combination output from the RPA-I™ printer and the RS-232C is desired, use option [3]. If only the printer is needed, use option [1]. The [ENTER] key records the choice in memory and returns the program to SELECT COMMAND.

Print Curve

The operator can choose whether or not to have a curve printed with each assay. When the prompt "Print Curve" appears, press the [ENTER] key. The RPA-I™ will ask whether a curve is desired or not. The operator needs only to answer "YES" by the [↑] or [ENTER] key, or "NO" by pressing the [↓] key.

Print Headings

This function allows the header "Ohmicron" to be printed with each assay. To select or unselect, follow the procedure under Print Curve.

Heater

The RPA-I™ is equipped with a heater to control the temperature of the sample compartment to 37°C. This function allows the operator to turn the heater ON or OFF. To access this function, press [ENTER] when "Heater ON/OFF" is displayed. The RPA-I™ will ask whether the heater should be turned ON or OFF. The operator needs only to answer "YES" by the [↑] or [ENTER] key, or "NO" by pressing the [↓] key. All RaPID Assay® protocols should be run with the heater turned OFF.

SECTION 4 - RUNNING YOUR FIRST ASSAY

PREPARATION AND RUNNING

Prior to performing your first RaPID Assay® please do the following:

1. Read through the entire package insert (found inside the box of reagents).
2. View the videotape (if available).
3. Gather together all the materials necessary to perform a RaPID Assay® in a work space.
4. Turn on the RPA-I™ or other spectrophotometer.
5. Remove the RaPID Assay® kits from the refrigerator.
6. Remove the bottles from the kit box and place them on a countertop.
7. Allow the reagent bottles to come to room temperature. The RPA-I™ should be warmed-up for 30 minutes.
8. Practice using the fixed volume and repeater pipettes with distilled water.
9. Label the top portion of the test tubes, with a permanent marker, in the following manner starting with the standard curve (in duplicate) followed by a control tube and sample tubes (in singlet).

Tube#	Contents of Tube
1,2	Zero Standard
3,4	Standard 1
5,6	Standard 2
7,8	Standard 3
9	Control
10	Sample #1
11	Sample #2
12	Etc.

10. Label 5 reservoirs, with a permanent marker, with the following:
    the first reservoir = conjugate 250µl
    the second reservoir = particles 500µl
    the third reservoir = wash 1ml
    the fourth reservoir = color 500µl
    the fifth reservoir = stop 500µl

    In addition, add the name of the pesticide you are testing for to each syringe.
11. Continue with the **Assay Procedure** section of the package insert or flowchart.

Expected Results for RaPID Assays®

- %CVs between standard duplicates of 10% or less.
- Absorbance readings for the 0 ppb standard greater than or equal to 0.800 for all assays.
- Corr (r) of 0.990 or greater for all assays.

SHORT OPERATING PROCEDURE FOR THE RPA-I™

WARM UP THE INSTRUMENT FOR 30 MINUTES PRIOR TO USE. Avoid analyzing samples with air bubbles, foam, scratches, or foreign matter. The RPA-I™ performs a self test first. If all parameters are satisfactory, the "Select Command" prompt will appear. If there are abnormalities, an "Error" message will appear.

INSTRUMENT DISPLAY	OPERATOR RESPONSE
SELECT COMMAND	Press RUN
RUN PROTOCOL: Aldicarb, Atrazine, Alachlor, etc.	Scroll through the YES [↑] or NO [↓] until the desired protocol appears. Press ENTER.
SPL. REPLICATES: (1-5)	Press 1 (Press 2 if analyzing samples in duplicate, etc.). Press ENTER.
BLANK TUBE INSERT TUBE EVALUATING TUBE REMOVE TUBE (Beep)	Insert tube with 1 mL of washing solution Remove tube
CAL. #1 REP. #1 INSERT TUBE EVALUATING TUBE REMOVE TUBE (Beep)	Insert first standard (0 ppb calibrator/tube #1) replicate. Remove Tube
Follow the prompts on the instrument display.	Note: Tube order is important here. The RPA-I expects to see the standards/calibrators in ascending order in duplicate, starting with 0 ppb.

After all the standards (calibrators) have been evaluated, the instrument will display:

PRINTING DATA LISTING XFORM DATA	Data will print.
PRINTING CURVE	Curve will print only if programmed to print (See Section 3 Special Functions - Instrument Functions: Print Curve).
CTRL. #1 REP. #1 INSERT TUBE EVALUATING TUBE REMOVE TUBE (Beep)	Insert Control Tube. Remove Tube.
EDIT CALIBRATORS YES/NO	Press NO if it is not necessary to edit the calibrators, press YES to edit (See Section 3 Run).
SPL. #1 REP. #1 INSERT TUBE EVALUATING TUBE REMOVE TUBE (Beep)	Insert first Sample Tube. Remove Tube.

Follow the prompts on the instrument display. After all the samples have been evaluated, press STOP

EXPLANATION OF DATA

Bolded areas are explained in the right hand column.

```
04-12-91  12:36:38

******** OHMICRON ********

PROTOCOL :  ATRAZINE

TECH ID :_______________
LOT #   :_______________
EXP DATE:_______________

Data Reduct:Lin.Regression
Xformation:          Ln/LgtB←
Read Mode  :     Absorbance
Wavelength :          450 nm
Units      :            PPB

EQUATION OF LINE :
------------------
Slope      =     -0.842     ←
Intercept  =     -0.106     ←
Corr (r)   =     0.9929     ←

Transformed Data :
----------------
   Conc        Abs
-------------------
  -2.30      1.926          ←
   0.00     -0.334          ←
   1.61     -1.327          ←


Calibrator Data:
----------------

  Conc     Abs   %CV   Predic
         Diff          %Diff
-------------------------
  0.00   1.032
         1.024
  Mean   1.028   0.5

  0.10   0.889          0.10←
        -0.002          -2.4←
         0.906          0.08
        -0.018         -22.7
  Mean   0.897   1.4    0.09←
        -0.011         -11.8

  1.00   0.442          1.23
         0.234          18.9
         0.416          1.39
         0.393          28.2
  Mean   0.429   4.2    1.31
         0.311          23.7

  5.00   0.228          3.91
        -1.094         -28.0
         0.203          4.67
        -0.328          -7.0
  Mean   0.216   8.4    4.26
        -0.736         -17.3

-------------------------
```

Method of transformation for data. Example, Ln refers to the natural log of the concentration and LgtB refers to the logit function of the absorbance divided by the absorbance at zero concentration.

These values are the coefficients which describe a "best fit" or linear regression straight line where Logit (B/B_0) = slope x Log_e (conc. in ppb) + intercept. The Corr(r) is the correlation coefficient which indicates "goodness of fit" of the data to the best fit line. The square of this value represents the proportion of variance (on the y axis) that is explained by the linear regression.

This section shows the average "transformed data" for each standard point. For example,

Log_e (0.1 ppb) = -2.30

Logit ($\frac{0.897}{1.028}$ or $\frac{B}{B_0}$) = 1.926

0.889 = observed absorbance
0.10 = observed concentration
-0.002 = known conc.(0.10) - observed conc.(0.10)*
-2.4 = concentration diff (-0.002) ÷ observed conc. (0.10) x 100*
1.4 = standard deviation of observed absorbances ÷ mean (0.897) x 100

*For accuracy, the data reduction software of the RPA-I™ utilizes seven significant digit numbers although only three are displayed or printed.

```
Control Data :
--------------

Ctrl#    Abs      Conc
-------------------------
  1    Ø.482     2.94

ID:_____________________

Samples Data :
--------------

Spl#     Abs      Conc      %CV
-------------------------------
  1    Ø.482     1.Ø2
       Ø.46Ø     1.13
Mean   Ø.471     1.Ø8      7.3←

ID:_____________________

  2    Ø.36Ø     1.84
       Ø.368     1.76
Mean   Ø.364     1.8Ø      3.Ø

ID:_____________________

  3    Ø.991     Ø.Ø2nd      ←
       Ø.998     Ø.Ø1nd
Mean   Ø.995     Ø.Ø2nd17.7*←

ID:_____________________

  4    1.Ø38       nd        ←
       1.Ø36       nd
Mean   1.Ø37       nd

ID:_____________________
END OF RUN
Ø4-12-91 12:39:24
```

7.3 = %CV for concentrations

"nd" indicates concentrations below the "Normal Range Low" value entered during the protocol setup (see Section 3 Options - Enter/Edit Protocol). This value is the least detectable dose (LDD) for RaPID Assay® protocls.
"*" indicates the %CV exceeds the parameter setting limit.
"nd" indicates the absorbance calculated is greater than the absorbance of the 0 ppb standard therefore a concentration cannot be calculated.

SECTION 5 - DATA REDUCTION

The prompt "Data Reduction" describes how the RPA-I™ will calculate the standard curve line. This prompt is displayed as part of the Enter/Edit Protocol which can be accessed as described in Section 3 Options - Enter/Edit Protocol. The Data Reduction types available for the RPA-I™ are as follows:

a. Single Point
b. Linear Regression
c. Point to Point
d. Polygonal
e. Cal Factor
f. Qualitative

SINGLE POINT

A Single Point data reduction generates a straight line equation between the absorbance response of the single standard and its entered concentration (known value). The line is always assumed to go through zero. This equation is then used for concentration interpolation of unknown samples by using the following calculation:

$$Cu = Cs \times (Au/As)$$

Where: Cu = unknown concentration
Cs = standard concentration
Au = absorbance of the unknown sample
As = absorbance of the standard sample

Single Point Calibration is used for all linear standard curves utilizing a single calibrator. Note that NO curve can be printed for single point calibration.

LINEAR REGRESSION

The Linear Regression data reduction generates a straight line equation by the least squares regression method from calibration curve data. This equation is then used for concentration interpolation of unknown samples. Linear Regression should always be used for linear calibration curves having two or more calibrators. When entering calibrator values, always enter the lowest value first.

POINT TO POINT (LINEAR INTERPOLATION)

In this data reduction method, a straight line equation is generated between each point on a calibration curve. Unknown concentrations are calculated by interpolation from the appropriate line segment. This method may be used for non-linear concentration response curves having at least three calibrators. The concentration of the highest unknown sample that may be reported may not exceed the concentration of the highest calibrator. Samples with concentrations greater than the highest calibrator will be reported as over range. When entering calibrators, the lowest value must be entered first.

POLYGONAL (POLYGONAL INTERPOLATION)

The Polygonal Interpolation is an advanced form of linear interpolation, in which the line is curved between each point. The method subdivides the intervals along the concentration axis and using slope averaging constructs an open polygon to describe the curve. By repeating this process, the polygon constructed more nearly follows a hyperbola. This method is used to describe non-linear calibration curves and has been demonstrated to be similar to the type of curve which would be drawn by hand on graph paper. This method is used for non-linear concentration response curves having at least three calibrators. The highest concentration that may be reported in samples will be equal to the concentration of the highest calibrator. Values higher than this will be reported as over range. When entering calibrators, the lowest value must be entered first.

CAL FACTOR (CALIBRATION FACTOR CONVERSION)

This option allows the user to convert absorbance values to concentration units and is primarily used when there is a constant relationship between absorbance and concentration. The RPA-I™ multiplies the (bichromatic, delta) absorbance reading or the absorbance delta by the calibration factor, to give the concentration. Note that entering a calibration factor of "1.0" will simply make the instrument print out absorbance values.

QUALITATIVE

The Qualitative Data Reduction option allows unknown samples to be compared to one or two references, to two constants, or to combinations of references and constants. After the number

of reference replicates has been indicated, the instrument will display "C1 = Aref1 + Bref2 + C", and "C2 = Dref1 + Eref2 + F", where C1 and C2 represent the two Cutoffs, on the upper line of the LCD. This equation will remain on the upper line until all Cutoff Constant values, (i.e., "A" through "F") have been assigned. Constant values are assigned in response to prompts displayed on the lower line of the LCD.

On the lower line of the LCD, the instrument will prompt "A:=0.00". The operator responds with the constant for multiplication of the first reference. For example, the response or a percentage of response of the first reference may be chosen as the first cutoff. The constant "A" would than be set equal to 1 or to 0.XX (where XX is equal to the required percentage.) The first reference may be deleted by setting "A" equal to zero. The use of the second reference is defined similarly by assignment of the constant "B". The arbitrary constant "C" may also be assigned a value or may be deleted by setting it equal to zero. The second cutoff is determined in the same manner, using the factors "D", "E", and "F". The instrument must be instructed to assign unknowns as positive, negative or indeterminate by comparison to the defined cutoffs.

The instrument will prompt the operator to supply the appropriate response (Positive or Negative) for each possible condition. The statement "Unkn. < CO1" will be displayed. Scroll through the options: POSITIVE (REACTIVE), NEGATIVE (NONREACTIVE). This may be read as "If the unknown response is less than the first cutoff, is the result positive or negative?" The operator responds with the appropriate result by pressing the [ENTER] key. Similarly, the prompts "Unkn. = CO1" and "Unkn. > CO1" will be displayed, and the operator will respond with the appropriate results. The instrument will print all conditional statements with operator-defined results after these conditions have been defined. This allows the operator to examine the programmed conditional statements for accuracy. The instrument will then ask for the number of controls and control replicates. "Percent CV Flag" allows the operator to designate a percent coefficient of variation. The %CV flag is optional. Pressing the [ENTER] key without data input at this option will result in the CV Flag being set automatically at 100%. The rest of the prompts necessary to define the protocol are common with Quantitative Data Reduction protocols.

TRANSFORMATIONS

The RPA-I™ is programmed with the following transformation options:

Linear/Linear	Linear/Ln
Linear/Logit	Linear/(1/Response)
Ln/Linear	Ln/Ln
Ln/Logit	Ln/(1/Response)
Ln/LgtB	

The first transform always refers to the horizontal X-axis or concentration. The second transform refers to the vertical Y-axis or absorbance response. The Ln function used here is the Log_e.

The "logit" function is defined as follows:

$$Logit = \log (R-R_{min})/(R_{max}-R)$$

Where: R = absorbance response
R_{min} = minimum absorbance response
R_{max} = maximum absorbance response x K

K is the normalizing factor for the slope of the line. For example, if the operator wants to change the slope of the line by half, then a normalizing factor of 0.5 should be entered. This will be prompted by the RPA-I™ during the programming of the protocol (see Section 3 Options - Enter/Edit Protocol). The normalizing factor will automatically be 1.0 unless changed by the operator.

The "logitB" function is defined as follows:

$$LgtB = \log (R/Ro)/(1-R/Ro)$$

Where: R = absorbance response
Ro = absorbance response at zero dose concentration

When using this transformation, a zero dose concentration standard must be run as the first standard concentration. As a result of the method of calculation, concentration for any absorbance response greater than the average response for the zero standard cannot be calculated (i.e. requires taking a logarithm of a negative number). Such values will be reported as "nd", none detected.

SECTION 6 - MAINTENANCE

NO operator service is recommended other than changing the paper.

CHANGING THE PAPER

Near the end of the paper roll, a red band will appear on the printout. A new roll of paper should be inserted as soon as possible. The paper is replaced as follows:

a. Tear off excess paper from the roll.
b. Remove printer cover by pulling the two black knobs out and lift the cover.
c. Remove the old roll from the rear of the printing mechanism.
d. Insert spindle into new roll and place roll into paper holder.
e. Push the paper into the slot near the bottom of the back of the printer, and feed paper through the printing mechanism using the manual feed wheel. This process may be aided by cleanly cutting the paper on an angle.
f. Replace the printer cover.

TROUBLESHOOTING

Refer to the Precautions and Limitations section below for common problems. If, after the power switch is turned on and the "Select Command" prompt does not appear, record the error message (if one appears) and contact Ohmicron Technical Service Department at 1-800-544-8881.

ERROR CODES

No keyboard operation is capable of damaging the RPA-I™. The system will return an error message if an improper operation is attempted. Error and general messages will appear with asterisks on the second line of the display.

Error #1 Division by zero

"Error #1" is displayed when the sample values measured result in division by zero when calculations are attempted utilizing the data reduction routines specified.

Error #5 Floating Point Underflow
Error #6 Floating Point Overflow

"Error #5" or "Error #6" indicate that the positive or negative number generated by a calculation is too large to be utilized. Generation of this type error is more frequently possible during log or exponential function calculations. "Error #5" is displayed when the result of a calculation is too large a negative number to be represented in memory. "Error #6" is displayed when the result of a calculation is too large a positive number to be represented in memory.

For accuracy, the data reduction software of the RPA-I™ utilizes seven significant digit numbers although only three are displayed or printed. The data reduction software will not process positive or negative numbers greater that 1×10^{15}.

PRECAUTIONS AND LIMITATIONS

- It is recommended that the instrument is turned off after the final assay each day to conserve lamp lifetime. Always allow 30 minutes for the instrument to warm-up, once the RPA-I™ is turned on.

- Be careful to read samples in proper sequence. All data analysis assumes that the samples are read in the required order, which will be prompted by the display.

- Check assay tubes prior to performing readings for optical uniformity. Scratches, foreign materials or defects may cause erroneous readings.

- Make sure that air bubbles are not sticking to the sides of assay tubes prior to placing them in the sample module.

- If the printer runs out of paper, STOP processing immediately to avoid damage to the printer head and loss of data.

- Do not insert or remove cartridge or move the instrument without first switching the power OFF.

- The instrument should be placed on a stable surface with adequate air circulation to the air vents on the back and the bottom of the instrument to maintain proper ventilation.

- Avoid exposing the instrument to chemical sprays or cleaners or spilling liquids into the RPA-I™.

- Keep the sample compartment and the filter block well free of debris and do not drop pencils, pens or other objects into them.

SECTION 7 - ACCESSORIES

FILTER BLOCKS

Wavelength nm	Part No.
340/600	A00024
405/600	A00025
415/600	A00026
420/600	A00027
450/600	A00028
492/600	A00029
520/690	A00030
550/630	A00031
580/690	A00032
600/450	A00033
620/450	A00034

MISCELLANEOUS

a. Dust Cover, Part No. A00041
b. Lamp Assembly, Part No. A00035
c. Paper Roll Holder, Part No. A00036
d. Power Transformer, 220/240 volts, Part No. A00037
e. Power Transformer, 100/120 volts, Part No. A00038
f. Printer Paper (12 rolls), Part No. A00039
g. Printer Paper (120 rolls), Part No. A00040
h. RPA-I™ Manual, Part No. A00046

RaPID Assay® Environmental User's Guide

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SECTION 1 - INTRODUCTION

This manual is intended as a guide for first-time users of the RaPID Assay® kits and the associated equipment and as a reference for experienced users. In addition to information on setting up the required equipment and running Ohmicron's RaPID Assays, this guide includes background information for applying the RaPID Assay system to environmental analyses.

When beginning a project, first read the background section that pertains to the analyte(s) of interest (Section 2) and then using Sections 3-5 develop the sampling plan. Section 3 provides general QA/QC guidance, while Sections 4 and 5 describe an approach to determining an appropriate project-specific cutoff concentration and optimizing the sample dilution scheme for that concentration.

Before running the first assay, read thoroughly those sections that refer to each piece of equipment to be used (Sections 6 - 14). Next, proceed to Sections 15 and 16 to run the assay. Section 17 is provided to assist the operator in resolving problems which might be encountered. For a more detailed explanation, refer to the operating manual for individual pieces of equipment and to the package insert for each assay kit.

For experienced users, the last section (18) in this manual can be directly referred to for brief instructions for the RPA-I and worksheets for common calculations. This section has been located at the end of the manual so the user can quickly find this frequently used material.

If any of the material contained in this manual is unclear or if problems are encountered, please call Ohmicron's Technical Services at (800) 544-8881.

SECTION 2 - ANALYTE BACKGROUND

PCBs

Polychlorinated biphenyls (PCBs) are a class of 209 discrete chemical compounds (congeners) in which 1-10 chlorine atoms are attached to biphenyl. They were produced commercially as complex mixtures for a variety of uses, especially dielectric fluids in capacitors and transformers. PCBs were marketed under the tradename Aroclor®, from 1930 to 1977, for use in transformers, capacitors, printing inks, paints, dedusting agents, pesticides and other applications. Their chemical and physical stability and their electrical insulating properties led to their commercial utility. Since PCBs do not readily degrade in the environment and are lipophilic, they persist and tend to bioaccumulate. PCBs have been shown to be ubiquitous environmental pollutants occurring in most human and animal adipose samples, milk, sediment, and numerous other matrices.

In 1968, PCB-contaminated cooking oil was shown to cause an epidemic of severe acne among residents of a western province in Japan. Eventually, over 1000 people in this outbreak were diagnosed as having Yusho ("oil disease" in Japanese) in which acne is accompanied by other features including liver and nerve abnormalities in those who ingested the oil and low birth weights and hyperpigmentation in children born to exposed mothers. However, the toxicological data is confounded by the presence of other toxic compounds in the oil including polychlorinated dibenzofurans (PCDFs) that result from contamination and degradation of PCB. A similar incident occurred in Taiwan in 1979. Over 2000 people, mainly school children near a food processing plant, were poisoned and diagnosed with Yucheng ("oil disease" in Chinese).

After the Yusho poisoning, concern about the widespread environmental occurrence and health risk posed by PCBs resulted in regulation of the manufacture, processing, distribution, and use of PCBs in the United States under the Toxic Substance Control Act (TSCA). The U.S. Environmental Protection Agency (EPA) was given responsibility for enforcement of this act.

The EPA subsequently promulgated a series of rules, according to which: the discharge of PCB-containing effluents and the production of PCBs in the U.S. is prohibited; disposal of materials contaminated with PCBs is regulated; and use of PCB-containing materials still in service is restricted. PCBs are classified as a probable human carcinogen, Group B2, by the EPA (chemicals for which there is sufficient evidence of carcinogenicity in animals and inadequate data in humans). The EPA has established a Maximum Contaminant Level Goal (MCLG) of zero in drinking water. The Maximum Contaminant Level (MCL) and Practical Quantitation Limit (PQL) for drinking water have been set at 0.5 ppb. In soil the EPA established 500 ppm as an incineration limit and 50 ppm as the TSCA hazardous waste regulatory limit/landfill limit. Regulatory limits for soil remediation vary according to state and site, but, in general, they are 5 or 10 ppm for industrial restricted access areas and 1 or 2 ppm for residential access areas.

BTEX (PETROLEUM HYDROCARBONS)

Volatile aromatic hydrocarbons, which include BTEX (benzene, toluene, ethylbenzene and xylenes), are prevalent contaminants at hazardous wastes sites across the United States as a result of spillage of hydrocarbon fuels and leaking underground storage tanks. It has been estimated that 2 to 5 million underground storage tanks containing petroleum hydrocarbons or chemicals are present in the United States. Suspected contamination sites must be evaluated to map out a contamination plume, to determine if remediation is needed or to determine the effectiveness of remediation effort. Identifying the presence of hydrocarbon contamination is a high priority and may result in sites being listed on the National Priorities List (NPL) established by the Comprehensive Environmental Response, Compensation and Liability Act (CERCLA or Superfund). Exposure via drinking water, especially groundwater, is the stated purpose of much of the hydrocarbon and underground storage tank regulations.

The collection and analysis of soil and groundwater samples have become the most widely practiced methods of site assessment and has gained wide acceptance of regulatory agencies. Petroleum hydrocarbon chemicals can occur in the liquid, dissolved, adsorbed, or vapor phase. Chromatographic effects such as differential multiphase partitioning, selective solubilization and other "weathering" effects are characteristic of hydrocarbon contamination. So, while accurate and precise characterization of a contaminated region of interest is desired, it may be difficult due to inherent uncertainty in measuring and interpreting results obtained for volatile organic compounds (VOCs).

The aromatic hydrocarbons have relatively high environmental mobility and propensity to partition in water. Hydrocarbons may migrate long distances along subsurface structures such as sewer lines and other utility trenches. In addition, a number of mechanisms and factors affect the behavior of these compounds in unsaturated soil. Such factors include transport of chemical compounds with the fluid or air flow, gas and liquid diffusion, hydrodynamic dispersion, sorption, volatilization/dissolution, abiotic degradation and biodegradation. But soil sampling is still commonly viewed as a definitive method of characterizing the magnitude and extent of contamination despite the high degree of variability in results due to spatial and temporal variation in soil properties and the small fraction of the total volume of soil at a site that is sampled.

Current EPA field sampling guidelines (SW-846) for the determination of VOCs in soil specify that subsamples be shipped to the laboratory in bottles that have been filled to capacity (i.e. no headspace) and stored at 4°C for no more than 14 days.

TNT

TNT is the common name for 2,4,6-trinitrotoluene, the most widely used military high-explosive. It has found wide application in shells, bombs, grenades, demolition and propellant compositions. TNT is among the least impact and friction sensitive of the high explosives but can be exploded on impact by a detonator mechanism. It is used as a standard explosive against which all other military high explosives are rated. The presence of TNT and other explosives in water and soil is one of the most serious environmental problems facing the military sites where munitions were manufactured, stored or used.

TNT is considered highly toxic, mutagenic and carcinogenic in bacterial and animal tests (USEPA, 1989). TNT is released to the environment as a result of open detonation and burning techniques used in the disposal of munitions. Historically, large quantities of aqueous TNT effluents are discharged at production facilities and ammunition load, assemble and pack plants. As much as one half million gallons of TNT wastewater are generated per day by a single TNT production facility (Hartley et al., 1981). "Red water" and "pink water" found at manufacturing, assembly and packing sites are due to TNT and other nitrated compounds and are classified as hazardous waste by the EPA and cannot be discharged into streams.

TNT has been identified in at least 19 of 1,300 hazardous waste sites that have been proposed for inclusion on the EPA National Priorities List (NPL). The U.S. EPA has assigned TNT a carcinogenic classification of C, which indicates that it is a possible human carcinogen. The Drinking Water Equivalent Level (DWEL), a lifetime exposure at which adverse health effects would not be expected to occur, is 20 ppb. The Lifetime Health Advisory for TNT in drinking water has been set at 2 ppb (USEPA, 1989). Soil clean-up levels vary from site to site but generally will be in the range of 1 to 30 parts per million

SECTION 3 - QUALITY CONTROL AND SAMPLING PLANS

QUALITY ASSURANCE / QUALITY CONTROL (QA/QC) PROGRAM

A QA/QC program develops information to evaluate the accuracy and precision of analytical data in order to establish the quality of the data. It can also provide an indication of the need for corrective action and determine the effect of such actions. The U.S. EPA in *Test Methods for Evaluating Solid Wastes* (SW-846) has recommended that a QA/QC program be structured in two major sections. The first, a program plan, serves as an operational charter for the laboratory and sets up basic laboratory policies. The second section, a project specific plan, differs from the program plan in that specific details of a particular sampling / analysis program are addressed. The essential elements of a QA Project Plan are:

- Title Page
- Table of Contents
- Project Description
- Project Organization and Responsibility
- QA Objectives
- Sampling Procedures
- Sample Custody
- Calibration Procedures and Frequency
- Analytical Procedures
- Data Reduction, Validation, and Reporting
- Internal Quality Control Checks
- Performance and System Audits
- Preventive Maintenance
- Specific Routine Procedures Used to Assess Data Precision, Accuracy, and Completeness
- Corrective Action
- Quality Assurance Reports to Management

In the U.S. EPA's *Quality Assurance/Quality Control Guidance for Removal Activities* from the Office of Emergency and Remedial Response, the QA Project Plan is further subdivided into a generic "Branch QA Project Plan" and a site-specific "Sampling QA/QC Plan." The Sampling QA/QC Plan (OSWER Directive 9360.4-01 Guidance) should contain the following sections:

- Title Page
- Background
- Data Use Objectives
- Quality Assurance Objectives
- Approach and Sampling Methodologies
- Project Organization and Responsibilities
- Quality Assurance Requirements
- Deliverables
- Data Validation

As immunoassays are applied in these programs, careful consideration should be given to the characteristics of the method. As stated in OSWER Directive 9360.4-01 Guidance, "the quality of the data is determined by its accuracy and precision against prescribed requirements or specifications, and by its usefulness in assisting the user to make a decision or answer a question with confidence." Immunoassay methods have performance characteristics that allow them to be effectively employed in many projects.

QA/QC METHODOLOGY

DATA USE AND DATA QUALITY (QA) OBJECTIVES:

The purpose of the data collection should be clearly understood before sampling begins. The intended use affects the choice of analytical method as well as the QA objective. Immunochemical methods are used commonly for QA1 and QA2 but they can be used, in some circumstances, to achieve QA3 objectives. The EPA has described three primary data quality levels.

spaced samples. This allows a more detailed determination of contaminant location. A particularly efficient approach is to collect initial samples from relatively large grids or strata, and then, when the initial results are available, sample areas of greatest interest in smaller grids or strata to obtain a more detailed assessment. This iterative process can be employed to obtain detailed information over large areas.

The availability of rapid results from immunoassays makes it useful in monitoring the effectiveness of remediation techniques to reduce or eliminate a contamination. The sampling plan in these applications can employ samples taken more frequently, approaching a "real time" monitoring of the remediation process to determine if the contaminant concentration in soil or water samples exceeds site-specific action limits.

Sampling Equipment & Storage. The choice of sampling equipment and sample containers will depend upon the contaminant of interest and site considerations. In making decisions about sampling equipment, the user needs to consider: 1) potential interactions between the sampling equipment or container with the analytes of interest including losses by adsorption, volatilization, or contamination caused by leaching from the containers or sampling devices; 2) cleaning procedures to minimize sample contamination and cross contamination between samples; and 3) analyte-specific properties that may dictate the use of optimum equipment or techniques such as minimizing agitation for volatile compounds.

Once the sample has been collected it must be stored to maintain the chemical and physical properties that it possessed at the time of collection. Recommendations of sample containers, sample preservation, and sample holding times are available from regulators such as the U.S. EPA in SW-846.

To assist the user, Ohmicron provides collection devices and sample containers optimized for use with the RaPID Assay system. The Soil Collection Kit includes a collection device and glass vials with Teflon lined caps for storage of the extract. The performance of the collection device and the stability of the extract have been characterized by Ohmicron for each analyte and reported in their respective Sample Extraction Kit package inserts. Other information about storage and preparation of the sample can be found in the Sample Extraction Kit package insert. Also provided in the Soil Collection Kit are labels for the collection devices and extraction collection vials to aid in chain of custody documentation.

QUALITY ASSURANCE REQUIREMENTS:

Level QA1. The requirements for Level QA1 can be readily met using the RaPID Assay system. The following requirements apply:

A. **Sample documentation.** The sample location, depth and matrix can be documented along with sample collection time, date and field analysis time and date.

B. **Instrument calibration data or a performance check of a test method.** *Ohmicron recommends that the Positive* Control provided with each RaPID Assay kit be included in an analytical batch. Results obtained for the Positive Control should be within a previously established range. Guidance on an acceptable range is provided in the RaPID Assay kit package insert in the Quality Control section. Proficiency Samples are also available from Ohmicron and can be used as controls at other analyte concentrations.

C. **Detection limit should be determined, unless inappropriate.** Detection limits are discussed in the "Selecting a Cutoff Concentration" section of this guide. Experimental determination of detection limits can be performed if desired. In SW-846, method detection limit is defined as the analyte concentration three standard deviations of above the average noise level. A similar estimate can be made using an immunochemical method by estimating the mean and standard deviation at zero analyte concentration. Using these estimates the

recommended that one performance evaluation sample be run daily.

G. **Other control measures for consideration.** Although not required in EPA Level QA2 plans, Ohmicron recommends:

- Matrix spikes be included. To perform a matrix spike, add a predetermined quantity of stock solution of the analyte of interest to a sample matrix prior to sample extraction. Contact Ohmicron Technical Services for further information.
- The raw data (i.e. the printout from the RPA-I or RPA-III) and calculations used for final results be documented.
- The three standard levels and the zero provided with each RaPID Assay kit be run in duplicate in each analytical batch.
- An uncontaminated sample from each site matrix be analyzed each day to document possible matrix interference.
- Sample duplicates be performed on at least one out of every twenty samples to document method repeatability.

DATA VALIDATION:

Level QA1. QA1 data need only be evaluated for calibration and detection limits criterion.

Level QA2. The results of 10% of the samples in the analytical package should be evaluated for all of the documentation elements. The holding times, blanks, and detection limits should also be reviewed.

OTHER QA/QC CONSIDERATIONS:

- Carefully and thoroughly read the entire package insert provided with the kits.
- Use replicate analyses when results indicate concentrations near the action level.
- Do not use kits past their expiration date.
- Do not mix reagents from one kit with another kit.
- Kits should be stored as indicated in instructions (generally 2-8°C for RaPID Assay kits and 2-30°C for RaPID Prep Extraction kits) and extended transit time at ambient conditions avoided.
- Appropriate training of operators is critical in obtaining reliable results. Materials (e.g. videotape, proficiency samples, written information) and assistance are available from Ohmicron.
- Additional information on quality control of immunoassays can be obtained from Ohmicron Technical Report T00029.

SECTION 4 - SELECTING A CUTOFF CONCENTRATION

GENERAL

Depending on the project, either quantitative or qualitative results may be required from an analytical method. Results from the RaPID Assay system may be interpreted as relative concentrations for purposes of measuring the extent of a contamination. More commonly, the concentration of contaminant in a sample is evaluated relative to an action or regulatory level. In other words, the sample is defined as above (positive) or below (negative) the action level.

The action level for projects vary with analyte, local regulations and the intended use of the site. The RaPID Assay System, because it reports quantitative values, allows the user to select a cutoff concentration appropriate for the particular project and to modify the dilution scheme and optimize the method for that particular concentration. This section provides guidance on factors to consider when selecting the appropriate cutoff for a project. The next section provides instructions on how to perform the optimum dilution of sample to detect the analyte at the cutoff concentration.

ACTION LEVELS

In most projects an Action Level is determined from regulatory and safety considerations. The concentrations of chemical contaminants cannot be equal to or exceed this level. Since there are a number of factors that affect a result from an analytical method, the highest concentration that would be judged as non-hazardous (negative) would be somewhat less than the Action Level. The difference between this level, which will be referred to as the "Cutoff" and the Action Level will be affected by a number of factors.

FACTORS AFFECTING THE CUTOFF

REACTIVITY:

All immunoassays rely on antibodies as the critical analytical reagent. Antibodies, large protein molecules known as immunoglobulins, are produced by the immune system of animals in response to foreign substances (antigens). Antibodies have the ability to specifically and tightly bind antigens. These are the properties that are exploited in an immunoassay.

Specificity describes the ability of an analytical method to distinguish between true (or specific) and interferences (or non-specific) results. Immunoassays can be developed to have different levels of specificity depending on the intended application of the immunoassay. Immunoassays can be very specific, detecting only one chemical compound, or they can be made less specific so that related compounds can be detected. The level at which related compounds are detected in an immunoassay is referred to as "cross-reactivity."

The characteristic specificity of an immunoassay is an important factor in the determination of a cutoff concentration. An immunochemical method developed for a class of compounds (e.g., PCBs) may be more sensitive to some members of the class (e.g., Aroclor 1254) than others. If a site has been characterized as being contaminated with predominantly one member of the class, this should be taken into account when determining the cutoff concentration. In the RaPID Assay package insert for each method, data are provided that characterize the reactivity of the method. These data represent the results obtained under a particular set of conditions and should be used as estimates of reactivity relative to the kit calibrators. It should be recognized that as conditions change (temperature, timing, etc.) the apparent reactivity of a class of compounds can change. Sometimes these changes can be significant (50% or more). Therefore, whenever possible, the assay should be directly calibrated with the compound of interest for greatest analytical accuracy.

The issue of reactivity becomes more complicated when dealing with mixtures of reactive components. This is exemplified by the detection of gasoline contamination using the Total BTEX immunoassay. Gasoline contains

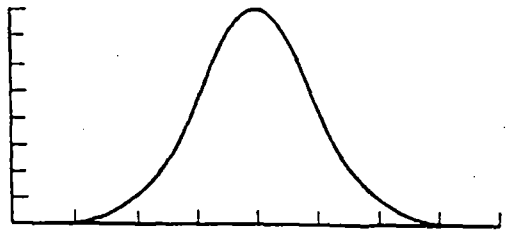


Figure 1 - Normal Distribution Frequency Plot

If the results are normally distributed and the variability of the method has been characterized, a user can predict how often (frequency) a result will occur above or below a particular known analyte concentration. When choosing a cutoff value it is important to understand the probability of positive and negative results at various concentrations relative to the action level. If one chooses a cutoff concentration too close to the action level, incidence of false negative results will be high. On the other hand, if the cutoff is much lower than the action level the frequency of false negatives will decrease but the incidence of false positives (positive results for concentrations below the action level) will increase.

To assist the user in determining the appropriate *Analytical Confidence Factor*, Ohmicron has developed tables for each analyte and sample matrix that estimate the frequency of positive and negative results at various sample concentrations. These estimates have been statistically derived from the precision that has been observed for most methods at approximately the midpoint of the calibration curve (except Total BTEX which is calculated at the lowest concentration standard, Standard 1). This is generally the most precise region of immunoassay results. If a different cutoff concentration is chosen, the tables provided may no longer be appropriate. Consult Ohmicron's Technical Services for assistance.

CHOOSING A CUTOFF

A number of approaches can be taken to determining the concentration at which immunoassay results will be considered positive. In this section three approaches will be presented in order of analytical preference. Some approaches may not be feasible due to the availability of analysis or materials.

METHOD 1 - USING A SITE SAMPLE:

In many cases initial site assessment activities will include analysis of one or more samples likely to be contaminated at concentrations above the established action level by an accepted traditional chromatographic method. Assuming the sample is representative of the site contamination and the quantitative analysis can be confidently performed (QA Level 3), this sample can be used to establish a cutoff concentration for the immunoassay. The preferred approach is to analyze the sample following the immunoassay kit protocol, including further dilution of the extract if necessary. The extract from such a sample can then be diluted to the cutoff concentration using the units assigned in the quantitative analysis and run as a single point calibrator in each assay run. Samples yielding results higher than the diluted calibrator should be considered positive and those giving a lower result, negative. Using this technique the immunoassay results are adjusted for extraction efficiency and contaminant reactivity on a run by run basis.

An example of this method of standardization might be a site to be assessed for gasoline contamination. In this example, a soil sample from the site had been analyzed using EPA Method 8020 and a result of 230 ppm BTEX (total) was obtained. The site action level of 10 ppm had been established so a 1:23 further dilution of the sample extract prepared according to the immunoassay extraction method could be used as a calibrator for the Total BTEX immunoassay. In this case, sample extracts resulting in absorbance values greater than the absorbance value obtained for this calibrator would be considered negative while those giving lower absorbance values would be considered positive for the contamination.

Alternatively, the appropriately diluted sample extract can be assayed by the immunoassay and a corresponding immunoassay concentration unit value established. Continuing the example given above, the sample extract after the additional

WORKSHEET

Required Action Level	_____	*(A)*
Reactivity Factor (from Table 1)	_____	*(B)*
Extraction Recovery Factor	_____	*(C)*
Analytical Confidence Factor (from Table 2)	_____	*(D)*
Cutoff Concentration A × B × C × D	_____	*(E)*

Example -

To optimize the PCB RaPID Assay system for detection Aroclor 1248 at 10 ppm in soil:

Required Action Level	10 ppm *(A)*	An *Action Level* of 10 ppm of Aroclor 1248 is assumed for this example. See *Action Level* in text above.
Reactivity Factor (from Table 1)	0.85 *(B)*	Since the PCB to be detected in this example is Aroclor 1248, the *Reactivity Factor* obtained from Table 1 of the PCB RaPID Assay Performance Characteristics is 0.85.
Extraction Recovery Factor	0.85 *(C)*	The *Extraction Recovery Factor* of 0.85 is obtained from the RaPID Prep Sample Extraction kit package insert. Preferably, this factor would be obtained from a spiked matrix determination.
Analytical Confidence Factor (from Table 2)	0.80 *(D)*	By examining Table 2 for PCB in Soil it can be observed that an *Analytical Confidence Factor* of 0.8 is estimated to yield 96.1% negative results at a PCB concentration of 5 ppm Aroclor 1248 (0.5 × *Action Level*[10 ppm Aroclor 1248]). In this example it is judged that a 3.9% "false positive" rate would be acceptable for samples at 5 ppm. It is also noted that at 10 ppm (*Action Level*) the estimated rate of positive results is 88% while at 20 ppm (2.0 × *Action Level*) the incidence for false negative results is estimated to be <0.1%.
Cutoff Concentration	5 ppm *(E)*	Performing the calculation A×B×C×D the result is 5.8 ppm. In this example the *Cutoff Concentration* used to classify the PCB assay results (after correction for dilution) was rounded down to 5 ppm to be conservative.

PCB RaPID ASSAY CHARACTERISTICS

Table 1 - Reactivity Factors for PCB RaPID Assay

Compound	*Reactivity Factor*
Aroclor 1016	0.14
Aroclor 1232	0.19
Aroclor 1242	0.41
Aroclor 1248	0.85
Aroclor 1254	1.00
Aroclor 1260	1.56
Aroclor 1262	0.76
Aroclor 1268	0.16

Table 2 - Analytical Confidence Factor Data

PCB in Water

Analytical Confidence Factor	@ 0.5 × Action Level		@ 1.0 × Action Level		@ 2.0 × Action Level	
	% Negative	% Positive	% Negative	% Positive	% Negative	% Positive
0.9	>99.9	<0.1	20.3	79.8	<0.1	>99.9
0.8	99.4	0.6	4.8	95.2	<0.1	>99.9
0.7	95.2	4.8	0.6	99.4	<0.1	>99.9
0.6	79.8	20.3	<0.1	>99.9	<0.1	>99.9
0.5	50.0	50.0	<0.1	>99.9	<0.1	>99.9

PCB in Soil

Analytical Confidence Factor	@ 0.5 × Action Level		@ 1.0 × Action Level		@ 2.0 × Action Level	
	% Negative	% Positive	% Negative	% Positive	% Negative	% Positive
0.9	99.1	0.9	27.8	72.2	<0.1	>99.9
0.8	96.1	3.9	12.0	88.0	<0.1	>99.9
0.7	88.0	12.0	3.9	96.1	<0.1	>99.9
0.6	72.2	27.8	0.9	99.1	<0.1	>99.9
0.5	50.0	50.0	0.2	99.8	<0.1	>99.9

Note - The shaded data achieves false positive rates of around 10% or less at 0.5 × Action Level and less than 5% false negative rates at 2.0 × Action Level.

Total BTEX RaPID ASSAY CHARACTERISTICS

If the *Cutoff Concentration* cannot be determined by Method 1 or 2 described earlier in this section, it is recommended that the Total BTEX kit be used as instructed in the package inserts and that samples yielding results greater than the lowest concentration standard (Standard 1) be classified as positives. Using this approach, the data in the following table reflects the concentrations of each reactive compound or mixture that would be equivalent to Standard 1 (Cutoff) and the lowest concentration that would be reliably (greater than 95% positive rate) detected (Action Level).

Compound	Water		Soil	
	Cutoff (ppm)	Action Level (ppm)	Cutoff (ppm)	Action Level (ppm)
Total BTEX*	0.09	0.14	0.9	1.4
m-Xylene	0.10	0.16	1.0	1.6
p-Xylene	0.34	0.51	3.4	5.1
o-Xylene	0.54	0.80	5.4	8.0
Ethylbenzene	1.10	1.52	11.0	15.2
Toluene	1.02	1.46	10.2	14.6
Benzene	11.1	14.84	111.0	148.4
Naphthalene	0.08	0.11	0.80	1.1
1,2,4-Trimethylbenzene	0.11	0.17	1.10	1.7
Anthracene	0.36	0.77	3.6	7.7
Styrene	0.38	0.56	3.8	5.6
Phenanthrene	0.20	0.28	2.0	2.8
1,3,5-Trimethylbenzene	0.37	0.57	3.7	5.7
Acenaphthene	0.51	0.74	5.1	7.4
n-Propylbenzene	0.91	1.29	9.1	12.9
Creosote	0.35	0.51	3.5	5.1
Gasolines	3.22	5.43	32.2	54.3
Mineral Spirits	2.86	4.18	28.6	41.8
Diesel Fuel	2.86	3.90	28.6	39.0
Kerosene	3.40	4.85	34.0	48.5
Jet A Fuel	5.75	7.84	57.5	78.4

*Total BTEX - equivalent parts of benzene, toluene, ethylbenzene and *m*-, *o*- and *p*-xylene (i.e. 1 ppm Total BTEX is composed of 1 ppm each of benzene, toluene, ethylbenzene and *m*-, *o*- and *p*-xylene). Alternatively, results can be expressed as the sum of the components by multiplying the value shown by 6.

SECTION 5 - SELECTING THE RANGE (DILUTIONS)

GENERAL

Ohmicron supplies RaPID Assay methods along with the appropriate reagents and equipment to prepare the sample for analysis. This system has been optimized to provide reliable results over a fixed range of concentrations that will often correspond to project needs. In addition, since the RaPID Assay methods present results in quantitative terms, the user has the flexibility to adjust the assay range to that which best suits a particular project. This can be accomplished by utilizing the range of results defined by standard curve or by further diluting the sample into the range. This section provides a systematic approach to determining the recommended dilution for samples at a variety of concentrations. The resulting dilution will position the critical concentration at an optimum position in the assay standard curve. A worksheet for performing the calculations is provided to aid the user.

GENERAL PROCEDURE

DEFINITIONS:

Cutoff Concentration: the concentration of analyte that will be used to classify samples as positive or negative for an Action Level. This concentration should be expressed in the same units as the RaPID Assay *Standard 2 Concentration*. When following the procedure recommended below, the assay performance will be adjusted to provide greatest precision on samples at this concentration. See Section 4: Selecting a Cutoff Concentration for guidance.

Standard 2 Concentration: the concentration of analyte in the Standard 2 provided in the RaPID Assay kit. There are three standards provided in each RaPID Assay kit. The *Standard 2 Concentration* is in the most sensitive region of the standard curve. For purposes of these calculations, the *Standard 2 Concentration* should be expressed in the same units as the *Cutoff Concentration*. *Standard 2 Concentrations* for the RaPID Assay kits are shown in Table 1 on the worksheet found at the end of this section.

Target Dilution Factor: the total dilution required to bring a sample at the *Cutoff Concentration* to a level at which the method is most precise.

Kit Sample Dilution: the total dilution obtained by following the dilution scheme provided with the RaPID Assay method and associated sample preparation techniques. For example when analyzing soil samples according the instructions provided with the PCB RaPID Assay, the RaPID Prep Soil Collection kit, and the PCB RaPID Prep Sample kit, the total dilution of the soil sample is 2000. A complete list of *Kit Sample Dilutions* is provided in Table 1 on the worksheet found at the end of this section.

Working Sample: the sample obtained after completing the sample preparation steps, if any, indicated in the Ohmicron kit method. This is the sample to be added to the assay tube. In the case of soil samples, the *Working Sample* is the diluted soil extract. For water samples, it may be the water sample itself or a dilution of the sample, i.e., the 1:2 methanol dilution (1 part methanol plus 1 part sample) of the water in the PCB method.

PROTOCOL:

1. Determine the *Cutoff Concentration* for the sample(s) to be analyzed for the project.
2. Obtain the *Standard 2 Concentration* of the appropriate RaPID Assay kit from Table 1 on the worksheet.
3. Calculate the *Target Dilution Factor* by dividing the *Cutoff Concentration* by the *Standard 2 Concentration*.
4. Obtain the *Kit Sample Dilution* from Table 1 of the worksheet.

WORKSHEET

Cutoff Concentration _____ (*A*)

Standard 2 Concentration (from Table 1) _____ (*B*)

Target Dilution Factor (*A/B*) _____ (*C*)

Kit Sample Dilution (from Table 1) _____ (*D*)

Ratio Target to Kit (*C/D*) _____ (*E*)

Volume of *Working Sample* (from Table 2) _____

Volume of Sample Diluent (from Table 2) _____

Table 1 - Ohmicron Kit Information

Analyte	*Standard 2 Concentration* (ppb)	Sample Matrix	*Kit Sample Dilution*
PCP	2 ppb	Water Soil	1 1000
PCB	1 ppb	Water Soil	2 2000
PAH	10 ppb	Water Soil	1.33 100
Total BTEX	0.35 ppm	Water Soil	1 10
TNT	1.0 ppb	Water Soil	1 1000

Table 2 - Dilution Schemes

*E**	Volume of *Working Sample*	Volume of Sample Diluent (mL)
1 to 10	0.5 mL (500 µL)	(*E* × 0.5)-0.5
10 to 100	0.05 mL (50 µL)	*E* × 0.05

*If *E* is between 100 - 1000 refer to Calculations Section.

Example -

To optimize the PCB RaPID Assay system for a 40 ppm in soil cutoff (Step numbers refer to the Protocol step numbers given earlier in the General Procedure section):

1) *Cutoff Concentration* 40,000 ppb(*A*)
2) *Standard 2 Concentration* 1 ppb (*B*)
3) *Target Dilution Factor* (*A/B*) 40,000 (*C*)
4) *Kit Sample Dilution* 2,000 (*D*)
5) Ratio Target to Kit (*C/D*) 20 (*E*)

Volume of *Working Sample* 50 µL

Volume of Sample Diluent (*E* × 0.05) 1.0 mL

7) If diluted sample from RaPID Assay is 2.3 ppb (*F*)
8) The concentration of sample in original matrix is (*F* × *C*) 92,000 ppb(*G*)
9) Compare (*G*) to *Cutoff Concentration* (*A*); greater is positive, less negative — Positive for Action Level

SECTION 6- RPA-I™ ANALYZER

The RPA-I Analyzer is a laboratory benchtop-based, single wavelength, dual beam, microprocessor-controlled analyzer. It can read the absorbances of calibrators and samples, perform mathematical computations, and report raw absorbances and sample concentrations with statistics. For a complete and detailed description of the RPA-I, please refer to the *RPA-I RaPID Analyzer Operations Manual* (Ohmicron Part No. A00046).

ENVIRONMENT

- 5° C to 33° C
- 10% to 85% humidity
- Flat, level surface away from strong sources of electromagnetic interference.
- No direct sunlight or drafts.
- Removed from sources of direct heat and moisture.
- Ventilation space at least 6 inches on sides and back.

UNPACKING AND INSTALLATION

1. Inspect the carton for visible signs of damage and note the condition of the SHOCK-WATCH indicator on the side of the carton. If damage has occurred, or a part is missing, immediately contact Ohmicron.

2. Open the carton and remove the brown rectangular box from the grey packing material. (Save all boxes.) This box contains the power transformer, roll of paper, and Program Cartridge. Refer to Figure 1 for identification of shipping carton contents.

3. Lift off the gray packing material to reveal the photometer. Remove it from the carton.

4. Insert the Program Cartridge (with the white label facing up) into the Program Cartridge Holder found on the rear panel of the instrument. Push in until the white label is no longer visible (Refer to Figure 2).

5. With the power **OFF** to the instrument, (bottom of the white toggle power switch should be depressed) insert the round end of the Power Transformer (notched end facing up) into the AC Power Connector found on the rear panel of the instrument. Plug the square end of the power cable into a grounded AC outlet.

6. The instrument is activated by depressing the top of the white toggle power switch. The instrument will perform a "Self Test." During this short test, the various electronic components of the RPA-I are automatically analyzed. This includes checks of EPROM and RAM memory. If there are any abnormalities in these areas, the RPA-I will alert the operator with an "ERROR" message. If all the parameters are satisfactory, the "Select Command" prompt will appear and the operator may continue.

SHORT OPERATING PROCEDURE FOR THE RPA-I

ALLOW THE RPA-I TO WARM UP FOR 30 MINUTES PRIOR TO USE. Avoid analyzing samples with air bubbles, foam, scratches, or foreign matter. The RPA-I performs a self test first. If all parameters are satisfactory, the "Select Command" prompt will appear. If there are abnormalities, an "Error" message will appear.

The RPA-I reports all results on a thermal paper printout. The unit is turned off by switching the power switch in the rear of the unit to the off position.

INSTRUMENT DISPLAY	OPERATOR RESPONSE
SELECT COMMAND	Press RUN
RUN PROTOCOL: Aldicarb, Atrazine, Alachlor, etc.	Scroll using the YES [↑] or NO [↓] until the desired protocol appears. Press ENTER.
SPL. REPLICATES: (1-5)	Press 1 (Press 2 if analyzing samples in duplicate, etc.). Press ENTER.
BLANK TUBE INSERT TUBE	Insert tube with 1 mL of washing solution/buffer
EVALUATING TUBE REMOVE TUBE (Beep)	Remove tube
CAL. #1 REP. #1 INSERT TUBE	Insert first standard replicate (0 ppb calibrator/tube #1).
EVALUATING TUBE REMOVE TUBE (Beep)	Remove Tube
Follow the prompts on the instrument display.	Note: Tube order is important here. The RPA-I expects to see the standards/calibrators in ascending order in duplicate, starting with 0 ppb.

After all the standards (calibrators) have been evaluated, the instrument will display:

PRINTING DATA	Data will print.
LISTING XFORM DATA	
PRINTING CURVE	Curve will print only if programmed to print (See Section 3 Special Functions - Instrument Functions: Print Curve).
CTRL. #1 REP. #1 INSERT TUBE	Insert Control Tube.
EVALUATING TUBE REMOVE TUBE (Beep)	Remove Tube.
EDIT CALIBRATORS YES/NO	Press NO if it is not necessary to edit the calibrators, press YES to edit (See Section 3 Run).
SPL. #1 REP. #1 INSERT TUBE	Insert first Sample Tube.
EVALUATING TUBE REMOVE TUBE (Beep)	Remove Tube.

Follow the prompts on the instrument display. After all the samples have been evaluated, press STOP.

```
Control Data :
--------------

Ctrl#    Abs      Conc
-------------------------
  1    Ø.272     3.Ø5        ⬅

ID:_____________________

Samples Data :
--------------

Spl#     Abs      Conc      %CV
-------------------------------
  1    Ø.482     Ø.83
       Ø.46Ø     Ø.94
Mean   Ø.471     Ø.89      6.2⬅

ID:_____________________

  2    Ø.36Ø     1.7Ø
       Ø.368     1.61
Mean   Ø.364     1.66      2.5

ID:_____________________

  3    Ø.925     Ø.Ø3nd
       Ø.93Ø     Ø.Ø3nd
Mean   Ø.928     Ø.Ø3nd    4.Ø

ID:_____________________

  4    Ø.991     Ø.Ø1nd      ⬅
       Ø.998     Ø.ØØnd
Mean   Ø.995     Ø.Ø1nd15.6*⬅

ID:_____________________

  5    Ø.927     Ø.Ø3
       Ø.921     Ø.Ø3
Mean   Ø.924     Ø.Ø3      4.6

ID:_____________________

  6    Ø.23Ø     4.2Ø
       Ø.233     4.1Ø
Mean   Ø.232     4.15      1.2

ID:_____________________

  7    1.Ø38       nd        ⬅
       1.Ø36       nd
Mean   1.Ø37       nd

ID:_____________________
END OF RUN
Ø4-12-93 12:39:24
```

Control Data

Displays absorbance and concentration of control sample. This concentration should be compared to the reported range located on the control vial label to assure the quality of the run.

Sample Data

6.2 = this %CV is calculated using the sample concentrations

"nd" indicates concentration below the "Normal Range Low" value entered during the protocol setup. This value is the least detectable dose (LDD) for RaPID Assay protocols.
"*" indicates the %CV exceeds the parameter setting limit. This is common when the concentration is very low.

An "nd" without a concentration indicates the absorbance measured is greater than the absorbance of the 0 ppb standard therefore a concentration cannot be calculated.

SECTION 7 - RPA-III™ ANALYZER

The RPA-III RaPID Analyzer is a hand-held microprocessor-based unit with fully interchangeable filters for wavelength selection. The instrument features a liquid crystal display and is powered through a cable connected to an electrical outlet. By itself, the RPA-III does not provide a printout of results, nor does it perform mathematical functions. For a complete and detailed description of the RPA-III, please refer to the *RPA-III RaPID Analyzer Operations Manual* (Ohmicron Part No. A00101).

UNPACKING AND INSTALLATION

1. Inspect the package for visible signs of damage and note the condition of the carton. If damage has occurred, or a part is missing, immediately contact Ohmicron.
2. Open the carton and remove the photometer and power cord from the packing material.
3. Plug the square end of the power cord into a grounded 110 v AC outlet. Insert the other end into the back of the RPA-III.
4. The unit is turned off by unplugging the power cable from the unit.

SHORT OPERATING PROCEDURE FOR THE RPA-III

Before reading tubes, allow five minutes after powering the RPA-III for warm up. Avoid analyzing samples with air bubbles, foam, scratches, or foreign matter.

INSTRUMENT DISPLAY	OPERATOR RESPONSE
STANDARDIZE? Y/N	Press the **Zero/No** button.
ZERO BLANK	Insert the blank tube containing at least 1 mL of Washing Solution/Buffer and press the **Zero/No** button.
READ	The RPA-III reads the blank tube and zeros the instrument (NOTE: for optimum performance, re-zero the unit after every ten readings. To re-zero, insert the blank and press the **Zero/No** button twice.)
00 0.000 ABS.	The unit displays the reading number and the absorbance. Remove tube.
READ SAMPLE	Insert the first tube and press the **Read/Yes** button.
01 X.XXX ABS.	The tube is read and the absorbance is displayed. RECORD THE ABSORBANCE VALUE.
02 X.XXX ABS.	Repeat for all standards, control, and samples.

CALCULATIONS

Using the graph paper provided with the RaPID Assay Kit, draw the standard curve by plotting the B/B_0 ratios versus concentrations using the absorbance data obtained above. Graph papers are specific for each method. Use only the graph paper supplied with each kit.

The mean absorbance value for the 0 standard is the B_0. The mean absorbance value for the other calibrators is the B value. Divide the absorbance of the standard, control or sample by the zero absorbance and multiply by 100 to obtain the % B/B_0. Draw the best straight line through all standard $\%B/B_0$ points ($\%B/B_0$ - y-axis, concentration - x-axis). Using the $\%B/B_0$ of the sample interpolate the concentration using the standard curve.

SECTION 8 - RPA-III™ FIELD KIT

The RPA-III RaPID Field Kit consists of the RPA-III Analyzer; a hand-held microprocessor-based unit with liquid crystal display, and a portable case which contains a rechargeable battery, printer and tube cover. The Field kit also contains a power cord for battery charging and optional AC operation. The RPA-III Field Kit is intended for use outside of the laboratory environment.

For a complete and detailed description of the RPA-III, please refer to the *RPA-III RaPID Analyzer* Operations Manual (Ohmicron Part No. A00101).

UNPACKING AND INSTALLATION

1. Seat the RPA-III firmly in the case making sure the battery/printer connection is inserted into the photometer.

2. Assuming the battery is charged, turn on both the printer and the RPA-III via the two power toggle switches.

Refer to the RPA-III Operating Manual for complete and detailed descriptions of operations.

SHORT OPERATING PROCEDURE FOR THE RPA-III FIELD SYSTEM

Allow the RPA-III to warm up for five minutes after switching it on before reading tubes. Avoid analyzing samples with air bubbles, foam, scratches, or foreign matter.

INSTRUMENT DISPLAY	OPERATOR RESPONSE
STANDARDIZE? Y/N	Press the **Zero/No** button.
ZERO BLANK	Insert the blank tube containing at least 1 mL of Washing Solution. Place the tube cover over the tube and seat it firmly on the reader. Press the **Zero/No** button.
READ	The RPA-III reads the blank tube and zeros the instrument (NOTE: for optimum performance, re-zero the unit after every ten readings. To re-zero, insert the blank and press the **Zero/No** button twice.)
00 0.000 ABS.	The unit displays the reading number and the absorbance. Remove tube.
READ SAMPLE	Insert the first tube. Place the tube cover over the tube. Press the **Read/Yes** button. The absorbance is displayed and printed.
01 X.XXX ABS.	The tube is read and the absorbance is displayed. RECORD THE ABSORBANCE VALUE.
02 X.XXX ABS.	Insert the next tube. Place the tube cover over the tube and press the **Read/Yes** button. The absorbance is displayed and printed.

CALCULATIONS

Refer to the appropriate *RPA-III Field Protocol* found in the *RPA-III RaPID Analyzer* Operations Manual for directions on *Calculations and Interpretation of Results* for qualitative or semi-quantitative results.

SECTION 9 - OTHER SPECTROPHOTOMETERS

REQUIREMENTS

This section describes the requirements for use of RaPID Assays with tube readers other than the RPA series photometers. These requirements are:

Absorbance Range: 0 - 2.0 AU

Wavelength: 450 nm.

Detection volume: 1 mL in cuvettes.

Drift: $\leq$ 0.005 AU per hour at 0 AU.

Linearity: $\leq$ 0.005 A or 2% difference from calculated regression line. Correlation coefficient, r = 0.9995 or better.

Results reporting: to a display or printer.

Sipper cell: the operator must first establish pump cycle times that allow for 1 mL to be drawn up from the tube and provide delivery to the flow cell to be read. Once this has been established, a return cycle of two times the pick-up volume should be returned to avoid cross-over contamination.

Throughput: ability to read the tubes within *15 minutes* after the addition of stop solution to the assay tube.

CALCULATIONS

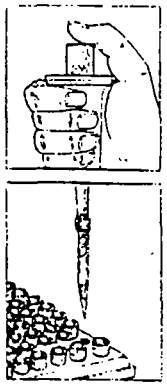
Using the graph paper provided with the RaPID Assay Kit, draw the standard curve by plotting the B/B_0 ratios versus concentrations using the absorbance data obtained above. Graph papers are specific for each method. Use only the graph paper supplied with each kit.

The mean absorbance value for the 0 standard is the B_0. The mean absorbance value for the other calibrators is the B value. Divide the absorbance of the standard, control or sample by the zero absorbance and multiply by 100 to obtain the % B/B_0. Draw the best straight line through all standard $\%B/B_0$ points ($\%B/B_0$ - y-axis, concentration - x-axis). Using the $\%B/B_0$ of the sample interpolate the concentration using the standard curve.

SECTION 10 - FIXED VOLUME PIPETTES

ASSEMBLY

Secure a tip on the pipette nose by pressing the nose cone **firmly** into a tip contained in the pipette tip rack.



USE

GENERAL:

The piston stroke is divided into three sections: measuring, blow-out, and tip ejection.

First stop: required volume is defined and dispensed

Second stop: dispense any residual liquid

Third stop: tip ejection

Practice several pipette transfers with water using the following procedure:

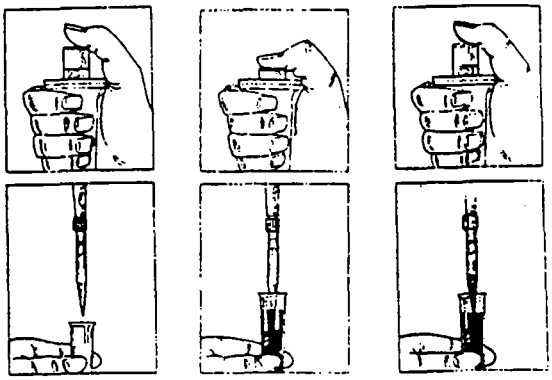
FILLING:

1. Keep pipette almost vertical.
2. Press button down to the first stop. Immerse tip 2-3 mm into the liquid.
3. **Slowly** allow the button to glide back (never let it snap back). Avoid drawing air bubbles.
4. Slide tip out along the inside of the vessel.

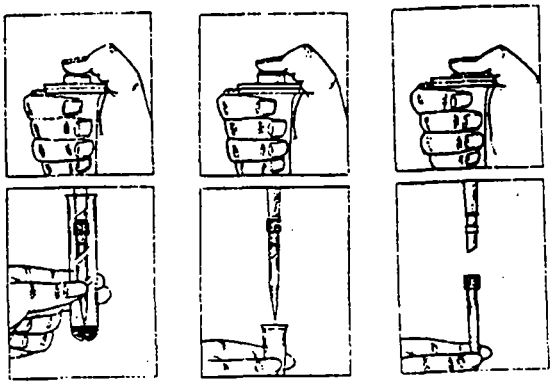
DISPENSING:

5. Insert pipette almost to the bottom of the test tube. Touch the tip to the side of the test tube about 5 mm above the dispensed liquid. Press button down to the **second stop**.
6. Remove pipette from the test tube.
7. Press button down to the third stop and discard the tip.

IMPORTANT: A new tip should be used for <u>each</u> standard or sample.



1 2 3&4

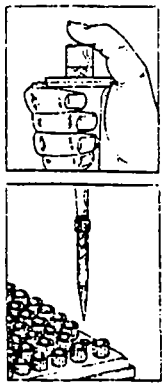


5 6 7

SECTION 11 - TRI-VOLUME PIPETTES

ASSEMBLY

Secure a tip on the pipette nose by pressing the nose cone **firmly** into a tip contained in the pipette tip rack.



USE

The tri-volume pipette will deliver the volume displayed opposite the red mark.

To select the volume to be delivered, press the button down to the horizontal mark (middle of the numbers) in the volume ring (e.g. 100--200--250) and turn to the right or left. The button must be returned to the fully extended position (numbers visible) to lock the required volume into place.

GENERAL:

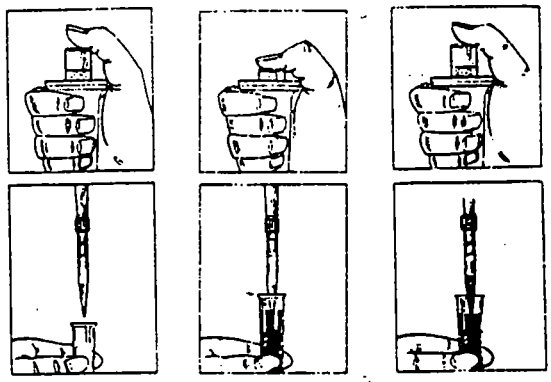
The piston stroke is divided into three sections: measuring, blow-out, and tip ejection.

First stop: required volume is defined and dispensed.

Second stop: dispense any residual liquid.

Third stop: tip ejection

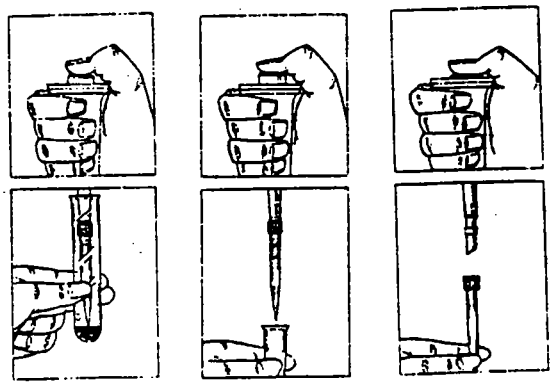
Practice several pipette transfers with water using the following procedure:



1 2 3&4

FILLING:

1. Keep pipette almost vertical.
2. Press button down to the first stop. Immerse tip 2-3 mm into the liquid.
3. **Slowly** allow the button to glide back (never let it snap back). Avoid drawing air bubbles.
4. Slide tip out along the inside of the vessel.



5 6 7

DISPENSING:

5. Insert pipette almost to the bottom of the test tube. Press button down to the **second stop**.
6. Remove pipette from the test tube.
7. Press button down to the third stop and discard the tip.

IMPORTANT: A new tip should be used for <u>each</u> standard or sample.

SECTION 12 - REPEATER PIPETTES

ASSEMBLY

DESIGN PRINCIPLE:

1. Volume Selection Dial: To determine the pipette volume, the dial setting (1-5) is multiplied by the minimum pipette volume of the reservoir. See example under Reservoir/Use of the Repeater Pipette.

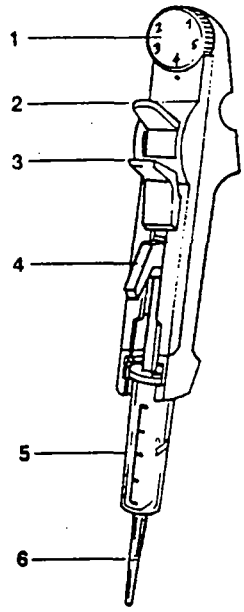
2. Pipette Lever: To deliver the selected volume, press this lever down until it stops.

3. Filling Lever: To fill the reservoir, slide this lever upward.

4. Locking Clamp: The locking clamp serves to firmly clamp the reservoir into the pipette.

5. Reservoir

6. Reservoir Cone



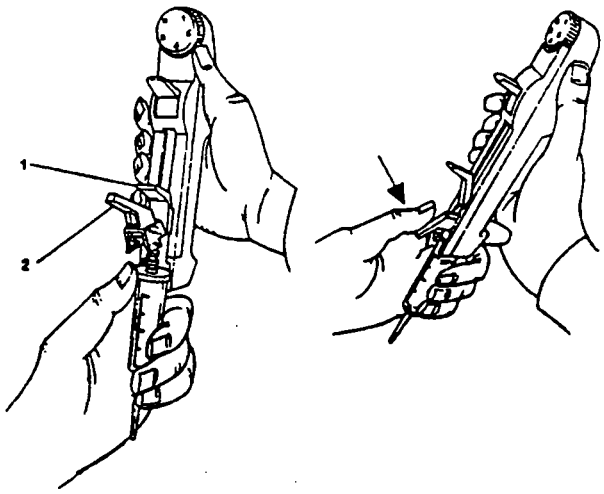
INSERTING A RESERVOIR:

1. Slide the filling lever (1) down until it stops.

2. Raise the locking clamp (2) upward.

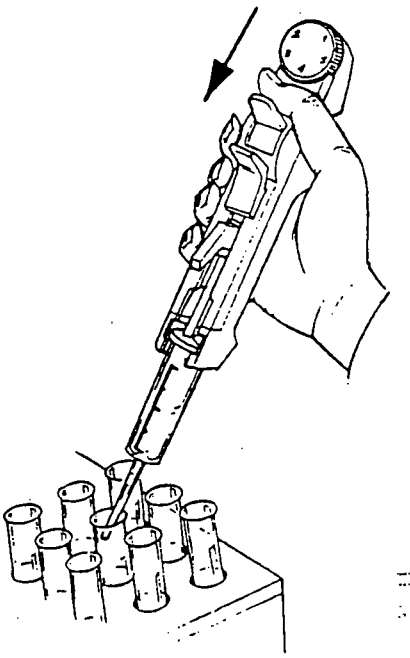
3. Insert the reservoir <u>until it clicks</u> into position. **Be sure the reservoir plunger is fully inserted into the barrel and the filling lever is completely down before attaching the reservoir to the pipette.**

4. Lower the locking clamp to secure the reservoir in place.



PIPETTING:

A set of five labeled 12.5 mL (1=250 µL) reservoirs is needed for each RaPID Assay. They should be labeled with the assay name and the following: "conjugate 250 µL," "particles 500 µL," "wash 1 mL," "color 500 µL," and "stop 500 µL."



1. Check the volume selection dial for the appropriate setting.
   Enzyme conjugate ➡ set dial at 1
   Magnetic particles ➡ set dial at 2
   Wash solution ➡ set dial at 4
   Color reagent ➡ set dial at 2
   Stop reagent ➡ set dial at 2

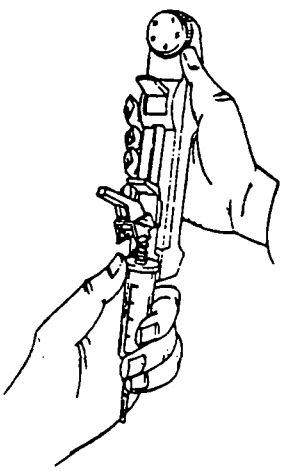
2. Hold the repeater at an angle so the tip is about one half inch below the tube rim without touching the rim or tube wall with the reservoir cone and aim the cone to deliver the liquid down the inside wall of the test tube.

3. Dispense the liquid by completely depressing the pipetting lever with a smooth, continuous motion until it stops.

4. Allow the pipetting lever to return to its starting position and repeat delivery into the next tube.

5. After the pipetting is completed, return the unused reagent to its container by holding the repeater unit over the container and pressing the filling lever down until it stops.

6. Prior to storage of the reservoirs, rinse the dedicated reservoirs twice with distilled water (keeping the reservoir in the pipette) by filling with 12.5 mL of distilled water each time and discarding the contents into a sink. Store syringes assembled (plunger inserted into barrel). Keep washed assembled syringes separated from each other. (Hint: An empty tray from the fixed volume pipette tips makes a handy storage rack). Reservoirs should be changed periodically (after 5-10 uses) since precision deteriorates with use.



REMOVING THE RESERVOIR:

Once the filling lever (1) is completely down, raise the locking clamp (2) upward and remove the reservoir.

SECTION 13 - MAGNETIC RACK

USE

The magnetic rack is composed of two parts: the top rack that firmly holds the test tubes in place and the bottom base which contains the magnets.

ASSEMBLY

Place the rack on top of the base making sure they ecurely and the test tubes are pushed y will go into the base.

USE OF THE ASSEMBLED RACK

For separation steps (washing and decanting) - to pull the magnetic particles to the sides of the tubes allowing the unbound components to be discarded.

DISASSEMBLY

Separate the top rack from the bottom base.

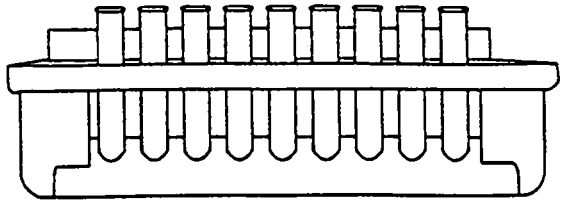
USE OF THE DISASSEMBLED RACK

For incubation steps - to allow the magnetic particles to remain suspended throughout the solution.

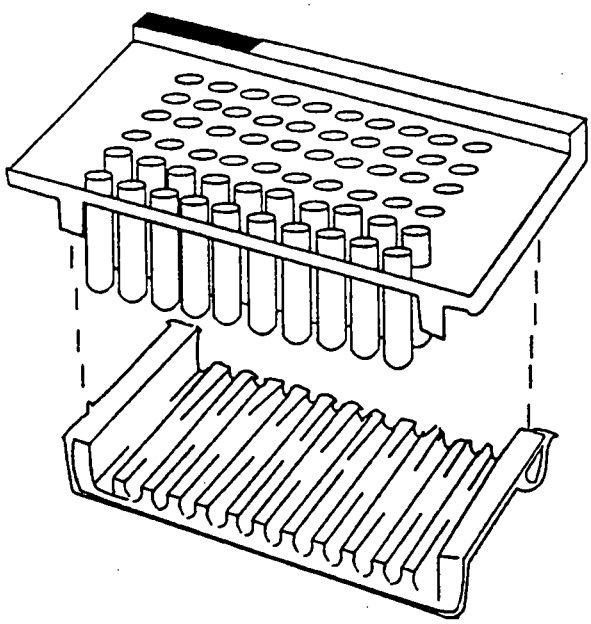
CARE

Do not submerge base in water. Clean with warm water and mild detergents.

CAUTION: Exposure to extreme heat or organic solvents could effect product's performance. Do not under any circumstances attempt to disassemble base to observe magnets. Magnets will be violently attracted to each other.



Assembled View



Disassembled View

SECTION 14 - SOIL EXTRACTION

GENERAL

The RaPID Assay kits are combined with the RaPID Prep™ Soil Collection Kit and the appropriate Sample Extraction Kits to provide an integrated system for soil extraction and analysis. The system allows for reliable, convenient and cost effective determinations at the field testing or remediation site.

Before performing your first analysis, read entire package insert found in each box.

REQUIRED MATERIALS

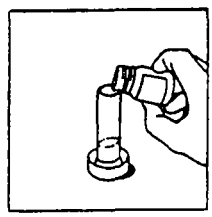
- RaPID Prep Soil Collection Kit
- Appropriate RaPID Prep Sample Extraction Kit
- Stopwatch or clock with second hand
- Permanent marking pen
- Protective gloves
- Digital balance (optional)

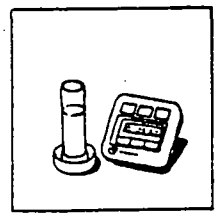
SAMPLING

Using the Soil Sample Collection Device from the Soil Collection Kit, collect 10 g of soil by weight or volume as directed in the package insert.

EXTRACTION

1. Add entire contents of the Extraction Solution to the Collection Device.
2. Replace screw cap.
3. Process by carefully following the package insert.
4. Let stand for 5 minutes.





FILTRATION

1. Remove screw cap and attach Filter Cap.
2. Attach plunger rod to device.
3. Remove luer cap and filter extract into Extract Collection Vial.

SECTION 15 - START-UP

MATERIALS NECESSARY TO PERFORM A RAPID ASSAY

1. RaPID Assay test kit with assay protocol, flowchart and package insert.
2. RPA-I, RPA-III with operating instructions (or equivalent spectrophotometer capable of reading 450 nm in a 1 mL sample size).
3. Magnetic rack separation unit.
4. Precision pipettes and appropriate tips, capable of delivering 100 µL, 200 µL, or 250 µL depending on method.
5. Repeating pipette, with appropriate reservoirs, capable of delivering 250 µL, 500 µL, and 1000 µL.
6. Thermolyne Maxi Mix™, Vortex Genie™ (or equivalent vortex mixer).
7. Disposable serological 5 or 10 mL glass or plastic pipettes for dispensing equal amounts of Chromogen and Hydrogen Peroxide to form the color reagent.
8. Pipette filler/bulb (for use with above disposable pipettes).
9. 20 mL (or larger) glass or plastic beaker for preparation of color reagent. Be sure the beaker has been thoroughly cleaned and rinsed. If contaminants are present, the reagent will develop a blue color and should be discarded.
10. Blotting paper for decanting steps (usually use a stack of 5 kitchen type paper towels).
11. Permanent marking pens for labeling tubes.
12. Instructional video (optional).
13. Laboratory timer or wrist watch.

ASSAY TIPS

Gently blot. Avoid **banging** the rack during the decanting steps.

Decant with the magnetic base joined to the top rack.

Wait a full two minutes for each separation step.

Bring samples and reagents fully to room temperature.

Vortex when specified <u>without foaming.</u>

Pay attention to the number of washes (2) and the amount of wash solution added (1 mL).

Add color reagent <u>without</u> the magnetic base joined to the top rack.

Remove as much liquid as possible from the test tubes by slowly decanting. A few droplets will remain in the tube.

Assure precise pipetting techniques.

Swirl the magnetic particles, without foaming, prior to addition.

SECTION 16 - ASSAY WORKSHEET

PURPOSE OF WORKSHEET

To aid in your evaluation of the assay kit, this familiarization run (a typical calibration curve plus proficiency samples) should be performed. Operators should repeat this run until the %CV of absorbances are consistently better than 10%. Experienced operators will obtain %CV's approaching 5% or better. The concentration values obtained on the control and the proficiency samples should be within the stated ranges.

FAMILIARIZATION RUN

Include in the run each standard in duplicate, a single control, and each proficiency sample in duplicate. Following the instructions in the kit package insert and found elsewhere in this manual, perform the assay.

RESULTS

RPA-I:

1. Observe the absorbance, the absorbance %CV, and the predicted result for each standard from the RPA-I printout (see Section 1 for a description of the printout).
2. Calculate the mean, SD, and %CV for the results from the standards and proficiency samples using a statisical calculator or the formulas given below the worksheet. Record in the following worksheet. Note that the %CV on the results may be significantly different than the absorbance %CV.
3. Compare to the above guidelines.

RPA-III OR OTHER PHOTOMETERS:

1. Record the absorbance value for each standard or sample in the worksheet which follows.
2. Calculate the mean, standard deviation (SD), and %CV on the absorbances for each standard and sample using a statisical calculator or the formulas given below the worksheet.
3. Using the graph paper provided with the kit, prepare a standard curve as described in the "Results" section of the kit package insert under "Manual Calculations". Read all samples and calibrators as individual points from the standard curve. Use the worksheet which follows to record the results.
4. Calculate and record the mean, SD, and %CV for the results using a statisical calculator or the formulas given below the worksheet.
5. Compare to the above guidelines.

INTERPRETATION

If you run two curves and do not achieve acceptable %CV's on the absorbances call Ohmicron Technical Support (1-800-544-8881) to discuss your results.

SECTION 17 - TROUBLESHOOTING

Symptom	Cause	Corrective Action
Increased Absorbance	Long incubation.	Adhere to incubation times.
	Washed tubes only once.	Be sure to wash tubes twice.
	Washed tubes with less than 1 mL of Washing Solution.	Check pipette for delivery of 1 mL of Washing Solution.
	Warm reagents.	Bring reagents to room temperature.
Decreased Absorbance	Banging rack during decanting.	Gently blot tubes, don't bang.
	Decanting without top rack joined to magnetic base.	Join rack and base prior to decanting.
	Short incubation.	Adhere to incubation times.
	Did not wait 2 minutes between washings.	Allow particles to separate between washings.
	Cold reagents.	Bring reagents to room temperature.
	Did not vortex after addition of color reagent or particles.	Vortex tubes after the addition of color reagent and particles.
	Addition of color reagent while top rack is joined to magnetic base.	Separate rack from base before adding color reagent.
Higher than expected results	Presence of cross reactants, particulate matter, or other interferences in the sample.	For particulate matter, filter samples with a 0.2 µm filter and re-assay. For cross reactants and possible interfering substances, dilute sample and reassay.
	Inaccurate standard curve.	Re-run standard curve.
	Expired reagents and/or kit.	Discard and replace with a fresh kit.
	Drift in sample results from beginning to end of run.	Add all reagents in a consistent manner to entire rack within one minute.
	Sample dilution inadequately mixed.	Be sure to throughly mix the diluted extract by inverting several times.

Symptom	Cause	Corrective Action
Increased % CV's (cont.)	Excessive wash solution remaining in tubes after decanting (may appear as bubbles).	After decanting, while holding top rack and bottom base together, allow tubes to drain in an inverted position for a few minutes. Also obtain more absorbent toweling.
	Imprecise addition of reagents.	Replace reservoir. Pipettes may need maintenance.
	Neglecting to vortex during the wash steps in the PCB procedure.	In the PCB RaPID Assay after adding the Washing Solution to each tube, vortex each tube for 1-2 seconds.

SECTION 18 - QUICK GUIDE

GENERAL

This section contains materials that are commonly used by experienced users. Prior to referring to this section, follow the general instructions found in Section 1 - Introduction for use of this manual. This section includes the following:

- Worksheet - Selecting a Cutoff Concentration
- Pentachlorophenol RaPID Assay Characteristics
- PCB RaPID Assay Characteristics
- PAHs RaPID Assay Characteristics
- Total BTEX RaPID Assay Characteristics
- TNT RaPID Assay Characteristics
- Worksheet - Selecting the Range (Dilutions)
- Short Operating Procedure for the RPA-I

PENTACHLOROPHENOL RaPID ASSAY CHARACTERISTICS

Table 1 - Reactivity Factors for Pentachlorophenol RaPID Assay

Compound	*Reactivity Factor*
Pentachlorophenol	1.00
2,3,5,6-Tetrachlorophenol	0.54
2,3,4,6-Tetrachlorophenol	0.15

Table 2 - Analytical Confidence Factor Data

Pentachlorophenol in Water

Analytical Confidence Factor	@ 0.5 × Action Level		@ 1.0 × Action Level		@ 2.0 × Action Level	
	% Negative	% Positive	% Negative	% Positive	% Negative	% Positive
0.9	99.4	0.6	26.6	73.4	<0.1	>99.9
0.8	97.0	3.0	10.6	89.4	<0.1	>99.9
0.7	89.4	10.6	3.0	97.0	<0.1	>99.9
0.6	73.4	26.6	0.6	99.4	<0.1	>99.9
0.5	50.0	50.0	<0.1	>99.9	<0.1	>99.9

Pentachlorophenol in Soil

Analytical Confidence Factor	@ 0.5 × Action Level		@ 1.0 × Action Level		@ 2.0 × Action Level	
	% Negative	% Positive	% Negative	% Positive	% Negative	% Positive
0.9	95.9	4.1	33.3	66.7	<0.1	>99.9
0.8	90.4	9.6	19.2	80.8	<0.1	>99.9
0.7	80.8	19.2	9.6	90.4	<0.1	>99.9
0.6	66.7	33.3	4.1	95.9	<0.1	>99.9
0.5	50.0	50.0	1.5	98.5	<0.1	>99.9

Note - The shaded data achieves false positive rates of around 10% or less at 0.5 × Action Level and less than 5% false negative rates at 2.0 × Action Level.

PAHs RaPID ASSAY CHARACTERISTICS

Table 1 - Reactivity Factors for PAHs RaPID Assay

Compound	*Reactivity Factor*
Phenanthrene	1.00
Fluoranthene	3.51
Benzo(*a*)pyrene	2.39
Pyrene	2.14
Chrysene	2.11
Anthracene	1.50
Indeno(1,2,3-*c*,*d*) pyrene	0.61
1,2-Benzanthracene	0.58
Fluorene	0.47
Benzo(*b*)fluoranthene	0.30
Creosote	0.99
Fuel Oil #6	0.31
Heating Oil	0.06

Table 2 - Analytical Confidence Factor Data

PAHs in Water

Analytical Confidence Factor	@ 0.5 × Action Level		@ 1.0 × Action Level		@ 2.0 × Action Level	
	% Negative	% Positive	% Negative	% Positive	% Negative	% Positive
0.9	>99.9	<0.1	13.3	86.7	<0.1	>99.9
0.8	>99.9	<0.1	1.3	98.7	<0.1	>99.9
0.7	98.7	1.3	<0.1	>99.9	<0.1	>99.9
0.6	86.6	13.4	<0.1	>99.9	<0.1	>99.9
0.5	50.0	50.0	<0.1	>99.9	<0.1	>99.9

PAHs in Soil

Analytical Confidence Factor	@ 0.5 × Action Level		@ 1.0 × Action Level		@ 2.0 × Action Level	
	% Negative	% Positive	% Negative	% Positive	% Negative	% Positive.
0.9	99.1	0.9	27.8	72.2	<0.1	>99.9
0.8	96.1	3.9	12.0	88.0	<0.1	>99.9
0.7	88.0	12.0	3.9	96.1	<0.1	>99.9
0.6	72.2	27.8	0.9	99.1	<0.1	>99.9
0.5	50.0	50.0	0.2	99.8	<0.1	>99.9

Note - The shaded data achieves false positive rates of around 10% or less at 0.5 × Action Level and less than 5% false negative rates at 2.0 × Action Level.

TNT RaPID ASSAY CHARACTERISTICS

Table 1 - Reactivity Factors for TNT RaPID Assay

Compound	*Reactivity Factor*
TNT (2,4,6-Trinitrotoluene)	1.00
1,3,5-Trinitrobenzene	0.66

Table 2 - Analytical Confidence Factor Data

TNT in Water

Analytical Confidence Factor	@ 0.5 × Action Level		@ 1.0 × Action Level		@ 2.0 × Action Level	
	% Negative	% Positive	% Negative	% Positive	% Negative	% Positive
0.9	>99.9	<0.1	18.7	81.3	<0.1	>99.9
0.8	99.6	0.4	3.7	96.3	<0.1	>99.9
0.7	96.3	3.7	0.4	99.6	<0.1	>99.9
0.6	81.3	18.7	<0.1	>99.9	<0.1	>99.9
0.5	50.0	50.0	<0.1	>99.9	<0.1	>99.9

TNT in Soil

Analytical Confidence Factor	@ 0.5 × Action Level		@ 1.0 × Action Level		@ 2.0 × Action Level	
	% Negative	% Positive	% Negative	% Positive	% Negative	% Positive
0.9	>99.9	<0.1	18.4	81.6	<0.1	>99.9
0.8	99.6	0.4	3.6	96.4	<0.1	>99.9
0.7	96.4	3.6	0.3	99.7	<0.1	>99.9
0.6	81.6	18.4	<0.1	>99.9	<0.1	>99.9
0.5	50.0	50.0	<0.1	>99.9	<0.1	>99.9

Note - The shaded data achieves false positive rates of around 10% or less at 0.5 × Action Level and less than 5% false negative rates at 2.0 × Action Level.

SHORT OPERATING PROCEDURE FOR THE RPA-I

ALLOW THE RPA-I TO WARM UP FOR 30 MINUTES PRIOR TO USE. Avoid analyzing samples with air bubbles, foam, scratches, or foreign matter. The RPA-I performs a self test first. If all parameters are satisfactory, the "Select Command" prompt will appear. If there are abnormalities, an "Error" message will appear.

The RPA-I reports all results on a thermal paper printout. The unit is turned off by switching the power switch in the rear of the unit to the off position.

INSTRUMENT DISPLAY	OPERATOR RESPONSE
SELECT COMMAND	Press RUN
RUN PROTOCOL: Aldicarb, Atrazine, Alachlor, etc.	Scroll using the YES [↑] or NO [↓] until the desired protocol appears. Press ENTER.
SPL. REPLICATES: (1-5)	Press 1 (Press 2 if analyzing samples in duplicate, etc.). Press ENTER.
BLANK TUBE INSERT TUBE	Insert tube with 1 mL of washing solution/buffer
EVALUATING TUBE REMOVE TUBE (Beep)	Remove tube
CAL. #1 REP. #1 INSERT TUBE	Insert first standard replicate (0 ppb calibrator/tube #1).
EVALUATING TUBE REMOVE TUBE (Beep)	Remove Tube
Follow the prompts on the instrument display.	Note: Tube order is important here. The RPA-I expects to see the standards/calibrators in ascending order in duplicate, starting with 0 ppb.

After all the standards (calibrators) have been evaluated, the instrument will display:

PRINTING DATA	Data will print.
LISTING XFORM DATA	
PRINTING CURVE	Curve will print only if programmed to print (See Section 3 Special Functions - Instrument Functions: Print Curve).
CTRL. #1 REP. #1 INSERT TUBE	Insert Control Tube.
EVALUATING TUBE REMOVE TUBE (Beep)	Remove Tube.
EDIT CALIBRATORS YES/NO	Press NO if it is not necessary to edit the calibrators, press YES to edit (See Section 3 Run).
SPL. #1 REP. #1 INSERT TUBE	Insert first Sample Tube.
EVALUATING TUBE REMOVE TUBE (Beep)	Remove Tube.

Follow the prompts on the instrument display. After all the samples have been evaluated, press STOP.

RAPID PREP™ FLOWCHART: PCB SOIL EXTRACTION PROTOCOL

1 *SAMPLING*

Using the Soil Sample Collection Device from the Soil Collection Kit, collect soil by weight (10 g) or volume as directed (see package insert for discussion).

2 *EXTRACTION*

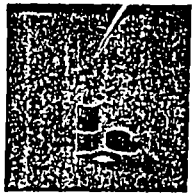
Add 20 mL (entire contents) of PCB Extraction Solution to the Collection Device.
Replace cap (without filter).
Shake for 1 minute.
Let stand for 5 minutes.

3 *FILTRATION*

Remove screw cap and attach Filter Cap. Attach plunger rod to device.
Remove luer cap and filter extract into Extract Collection Vial.

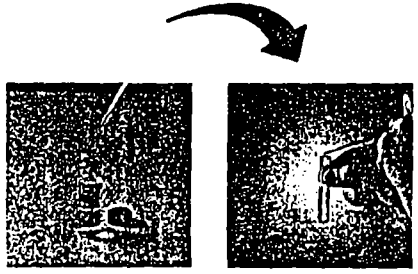
4a *DILUTION SCHEME*

For samples 500 ppb to 10 ppm:
Add 25 µL of filtered extract to a vial of PCB Extract Diluent (25 mL).
Cap and mix by by inverting several times.
Factor = 2000.



4b *DILUTION SCHEME*

For samples 50 ppm to 1000 ppm:
Add 25 µL of filtered extract to a vial of PCB Extract Diluent (25 mL).
Mix.
Add 25 µL of diluted extract (from 1st dilution) to 2.5 mL of PCB RaPID Assay Diluent.
Mix thoroughly.
Factor = 200,000.



5 *ASSAY*

Analyze diluted extract as "sample" according to the PCB RaPID Assay procedure (see reverse).

Multiply results of extracted soil samples by appropriate factor.

For Ordering or Technical Assistance Contact:
Ohmicron Environmental Diagnostics, Inc.
800 544-8881 215 860-5115
FAX 215 860-5213

PCB Rapid Assay Kits
Part # A00133 30 Tests
 # A00134 100 Tests
RaPID Prep Soil Collection Kit A00127

PCB FLOWCHART: SOIL PROTOCOL

1

Remove upper rack from magnetic base.

Label test tubes for Standards, Control, and Samples.

Tube #	Content
1, 2	Diluent/Zero Standard, 0 ppb
3, 4	Standard 1 0.25 ppb
5, 6	Standard 2 1.0 ppb
7, 8	Standard 3 5.0 ppb
9	Control
10	Sample 1
11	Sample 2

Add 200 µL of either Standards, Control or Diluted Soil Extract to the **bottom** of each test tube by inserting the pipet tip all the way into the tube without touching the sides or the bottom of the tube.

2

Add 250 µL of PCB Enzyme Conjugate down the inside wall of each tube by aiming the pipet tip 1/4" to 1/2" below the tube rim without touching the rim or tube wall with the pipet tip; deliver liquid gently.

3

Add 500 µL of **thoroughly** mixed PCB Antibody Coupled Magnetic Particles down the inside wall of each tube by using the technique described in Box 2. *Vortex* for 1 to 2 seconds (at low speed to minimize foaming).

4

Incubate 15 minutes at room temperature (15°-30°C).

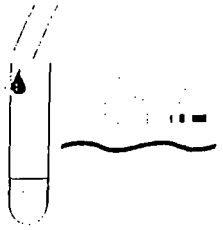
5

Combine the upper rack with the magnetic base; **press all tubes into base;** allow 2 minutes for the particles to separate.

6

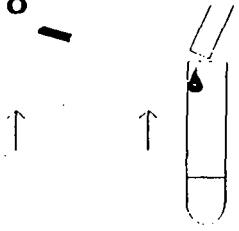
Do **not** separate upper rack from lower base. Using a smooth motion, *invert* the combined rack assembly over a sink and pour out the tube contents: keep inverted and **gently** blot the test tube rims on several layers of paper toweling.

7



Add 1 mL of Washing Solution down the inside wall of each tube. Vortex each tube. *Wait 2 minutes.* Using a smooth motion, invert the combined rack assembly over a sink and pour out the tube contents: keep inverted and **gently** blot the test tube rims on several layers of paper toweling.
Repeat this step.

8

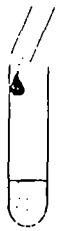


Lift the upper rack (with its tubes) off the magnetic base; *add* 500 µL of Color Reagent down the inside wall of each tube by using the technique described in Box 2. *Vortex* for 1 to 2 seconds (at low speed to minimize foaming).

9

Incubate for 20 minutes at room temperature (15°- 30° C). During this period, add 1 mL of Washing Solution into a clean tube for use as an instrument blank in Step 10.

10



Add 500µL of Stopping Solution down the inside wall of each tube by using the technique previously described. *Read* results at 450nm within 15 minutes after adding the Stopping Solution. *Multiply* results of extracted soil samples by the appropriate factor.
[Safety Caution: Stopping Solution contains 0.5% sulfuric acid.]

For Ordering or Technical Assistance Contact:
Ohmicron Environmental Diagnostics, Inc.
800.544.8881 215.860-5115

PCB Rapid Assay Kits
Part # A00133 Tests
 # A00134 Tests

2. QUANTERRA SOP FOR TO-13 SVOCs ANALYSIS

UNCONTROLLED COPY

Quanterra Environmental Services

Controlled Copy
Copy No._____

SOP No. SAC-OP-0006
Revision No. 1
Revision Date: 3/11/96
Page: 1 of 18

OPERATION-SPECIFIC STANDARD OPERATING PROCEDURE

TITLE: Preparation and Extraction of Semi-Volatiles on PUF(PolyUrethane Foam)/XAD-2 Resin Samples For GC/MS Analysis

(SUPERSEDES: LM-CAL-7013)

Prepared by: Brian Thompson and Steve Valmores

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Approved by: _____
Laboratory Director, Bruce Warden

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1. SCOPE AND APPLICATION

1.1. This document describes the pre-cleaning and preparation procedure for the determination of base/neutral and acids involving a combination quartz filter/adsorbent cartridge with subsequent analysis by GC/MS. A list of analytes and their CAS registration numbers are listed in Appendix I.

2. SUMMARY OF METHOD

2.1. Filters and adsorbent cartridges are cleaned in solvent, dried, assembled and shipped to the field. During sampling, the amount of air sampled through the filter is recorded and the filter and cartridge are sent to the laboratory for analysis. The filter and adsorbent cartridge is extracted using soxhlet extraction techniques. The extract is then concentrated by Kuderna-Danish and adjusted to the final volume of extract for analysis by GC/MS.

3. DEFINITIONS

3.1. Refer to the Glossary of the Quanterra QAMP.

3.2. Batch: A QC batch is a set of up to 20 field samples of similar matrix that behave similarly and are processed using the same procedures, reagents, and standards within the same time period.

3.2.1. For method TO-13, the QC batch is defined at the preparation stage.

3.3. PUF Cartridge: Air sampling cartridge consisting of a glass cylinder in which a cylindrical polyurethane foam adsorbent filter is inserted. These are typically used in high volume samplers.

3.4. XAD-2 resin: A porous polymeric adsorbent resin typically used for semivolatile air sampling. The resin is placed in a cartridge and used in a high volume air sampler.

4. INTERFERENCES

4.1. Method interferences may be caused by contaminants in solvents, reagents, glassware, and other sample processing hardware.

4.2. Matrix interferences can be caused by contaminants that are coextracted from the sample.

4.3. Heat, ozone, NO_2 and ultraviolet light may cause sample degradation.

5. SAFETY

5.1. Procedures shall be carried out in a manner that protects the health and safety of all Quanterra associates.

5.2. Eye protection that satisfied ANSI Z87.1 (as per the Chemical Hygiene Plan), laboratory coat, and chemically resistant gloves must be worn while samples, standards, solvents, and reagents are being handled. Disposable gloves that have been contaminated will be removed and discarded; other gloves will be cleaned immediately.

5.2.1. Neoprene, natural rubber, and butyl gloves provide varying degrees of protection against those chemicals listed. Refer to permeation/degradation charts for the actual data.

5.3. The health and safety hazards of many of the chemicals used in this procedure have not been fully defined. Additional health and safety information can be obtained from the Material Safety Data Sheets (MSDS) maintained in the laboratory.

5.3.1. Chemicals that have been classified as carcinogens, or potential carcinogens, under OSHA include: methylene chloride

5.3.2. Chemicals that have been classified as flammable, under OSHA include: acetone and hexane

5.4. Exposure to chemicals must be maintained as low as reasonably achievable, therefore, unless they are known to be non-hazardous, all samples must be opened, transferred and prepared in a fume hood, or under other means of mechanical ventilation. Solvent and waste containers will be kept closed unless transfers are being made.

5.5. The preparation of standards, reagents and glassware cleaning procedures that involved organic solvents such as methylene chloride will be conducted in a fume hood with the sash closed as far as the operation will permit.

5.6. All work must be stopped in the event of a known or potential compromise to the health and safety of a Quanterra associate. The situation must be reported immediately to a laboratory supervisor.

6. EQUIPMENT AND SUPPLIES

6.1. Soxhlet apparatus

6.2. Filtering funnels - 100 mm top diameter

6.3. Disposable 1 mL pipettes

6.4. Concentrator tubes, Kuderna-Danish - 10 mL graduated

6.5. Evaporative flasks, Kuderna-Danish - 1000 mL. Attach to concentrator tube with clamp

6.6. Vials - 2 mL capacity with Teflon-lined screw cap

6.7. Quartz Fiber filter (10.16 cm, round)

6.8. Boiling chips - methylene chloride rinsed

6.9. Glass sampling cartridge with Nickel or Stainless Steel screen (Mesh 200/200)

6.10. PolyUrethane Foam (PUF) plugs, (Density 0.0225g/ cm^3)

6.11. XAD-2 resin (20/60 mesh)

6.12. Cotton gloves

6.13. Water bath - heated with a concentric ring cover capable of temperature control

6.14. Top loading balance capable of weighing accurately to 0.01 g

6.15. Nitrogen evaporation device

6.16. Aluminum foil, methylene chloride rinsed.

6.17. Pyrex dish

6.18. Chemically resistant gloves

7. REAGENTS AND STANDARDS

7.1. Methylene chloride - pesticide grade or equivalent

7.2. Hexane - pesticide grade or equivalent.

7.3. Acetone - pesticide grade or equivalent

7.4. Sodium sulfate (ACS), anhydrous, granular

7.5. Surrogate spiking solution. See Attached Appendix II.

7.6. Duplicate Control Sample (DCS) spiking solution . See Attached Appendix II.

7.7. Pre-spike solution (by client request).

8. SAMPLE COLLECTION, PRESERVATION AND STORAGE

8.1. The samples should be returned to the laboratory, packed in blue ice to ensure that the assembled cartridge and filter are chilled to 4 ± 2°C. The samples should be received by the laboratory within two days of sample collection.

8.2. The samples are logged in the laboratory logbook or appropriate data tracking system according to sample location and filter/adsorbent cartridge number identification.

8.3. The samples must be kept refrigerated at 4 ± 2°C, exposure of samples to fluorescent light must be minimized.

8.4. Sample extracts must be stored in sealed air-tight vials.

8.5. Samples, sample extracts, and standards must be stored separately.

8.6. The extraction shall be started within 7 days from sample collection. Extracts must be analyzed within 30 days following the start of the extraction.

9. QUALITY CONTROL

9.1 One method blank must be extracted with every process batch of similar matrix, not to exceed twenty (20) samples. The method blank is a PUF/XAD-2 cartridge and quartz fiber filter processed in the same manner and at the same time as the associated samples. Corrective actions must be documented on a Nonconformance Memo, then implemented when target analytes are detected in the method blank above the reporting limit or when surrogate recoveries are outside control limits. See Quanterra Inc. QA Policy 003 for specific acceptance criteria.

9.2 A pair of Duplicate Control Samples (DCS) must be extracted with every process batch of similar matrix, not to exceed twenty (20) samples. The DCS is a media-free solvent aliquot spiked with surrogate solution and DCS spike solution. See Appendix II for spike levels. The DCS pair must be processed in the same manner and at the same time as the associated samples. Corrective actions must be documented on a Nonconformance Memo, then implemented when recoveries of any spiked analyte is outside control limits provided on the LIMS or by the client. See Quanterra Inc. QA Policy 003 for specific acceptance criteria.

9.3 All samples in the process batch, including the Method Blank and the DCS are spiked with appropriate surrogate solution. See Appendix II for spike levels.

9.4 In the event that the PUF/XAD-2 cartridges were pre-spiked with surrogate prior to shipment to the field, by client request only, the method blank is also spiked with the pre-spike solution. See Appendix II.

10. CALIBRATION AND STANDARDIZATION

10.1. The analytical balance used must be calibrated and documented in a logbook for each day of use.

10.2. Standard and equipment calibration must be performed according to the frequency and procedures described in the SW846 8270 SOP "GCMS Analysis of Semi-volatile Organics", LM-CAL-3004, revision 4.0, SOP "GCMS Analysis Based on Method 8270B, SW846", CORP-MS-0001, revision 1.0, and the Quanterra Incorporated Quality Assurance Management Plan.

11. PROCEDURE

11.1. One time procedural variations are allowed only if deemed necessary in the professional judgment of supervision to accommodate variation in sample matrix, radioactivity, chemistry, sample size, or other parameters. Any variation in procedure shall be completely documented using a Nonconformance Memo and is approved by a Technical Specialist and QA Manager. If contractually required, the client shall be notified. The Nonconformance Memo shall be filed in the project file.

11.2. Any unauthorized deviations from this procedure must also be documents as a nonconformance, with a cause and corrective action described. The Nonconformance Memo shall be filed in the project file.

11.3. Preparation of Sample Filter and Adsorbent Cartridge

11.3.1. The sampling device consists of a glass cartridge fitted with a nickel or stainless steel screen to retain the PUF /XAD-2 resin. A quartz fiber filter (10.16 cm, round) accompanies the PUF/XAD-2 cartridge to the field.

11.3.2. Quartz Fiber Filter Preparation

11.3.2.1. For clean-up of the quartz fiber filter, a batch of quartz fiber filters is placed in a soxhlet apparatus and extracted at least once with methylene chloride for a minimum of 16 hours at an extraction rate of approximately 4 cycles per hour.

11.3.2.2. At the end of the soxhlet extraction, the quartz fiber filter is removed from the soxhlet apparatus, placed in a fume hood to dry for a minimum of six hours before use.

11.3.3. PUF Adsorbent Preparation

11.3.3.1. The PUF adsorbent is composed of 1 inch and 2 inch inserts cut from sheet stock and should fit with slight compression in the glass cartridge, supported by wire screen.

11.3.3.2. For initial clean-up of the PUF adsorbent, a batch of PUF plugs is placed in a soxhlet apparatus and extracted with methylene chloride for a minimum of 4 hours at an extraction rate of approximately 4 cycles per hour.

11.3.3.3. At the end of the initial soxhlet extraction, the spent methylene chloride is discarded per Attachment C of the Chemical Hygiene Plan, Section WS002, Table 1, and replaced with fresh methylene chloride. The PUF adsorbent is extracted at least twice for a minimum of 16 hours each at an extraction rate of approximately 4 cycles per hour.

11.3.3.4. The extracted PUF plugs are placed in solvent rinsed Pyrex dishes and dried in a fume hood for a minimum of 4 hours.

11.3.4. XAD-2 Resin Preparation

11.3.4.1. For initial clean-up of the XAD-2 resin, a batch of XAD-2 is placed in a soxhlet apparatus and extracted with methylene chloride for a minimum of 4 hours at an extraction rate of approximately 4 cycles per hour.

11.3.4.2. At the end of the initial soxhlet extraction, the spent methylene chloride is discarded per Attachment C of the Chemical Hygiene Plan, Section WS002, Table 1, and replaced with fresh methylene chloride. The XAD-2 resin is extracted at least once for a minimum of 16 hours at an extraction rate of approximately 4 cycles per hour.

11.3.4.3. The XAD-2 resin is removed from the soxhlet apparatus, placed in a solvent rinsed Pyrex dish and dried at room temperature for a minimum of 16 hours.

11.3.5. PUF/XAD-2 Sampling Cartridge Preparation

11.3.5.1. **Cotton gloves must be worn while handling glass cartridges to prevent contamination. Chemically resistant gloves must be worn to prevent exposure to samples or chemicals.**

11.3.5.2. Prior to assembly of the PUF/XAD-2 sampling cartridges, the glass cartridges containing nickel or stainless steel screen are rinsed with acetone, hexane, and methylene chloride and allowed to dry for a minimum of 1 hour. A soxhlet-extracted, dried 1 inch PUF is placed on top of the screen in the glass sampling cartridge.

11.3.5.3. Approximately 15 grams of 20/60 mesh XAD-2 resin is placed into the sampling cartridge on top of the PUF.

11.3.5.4. The 2 inch PUF adsorbent is placed on top of the XAD-2 resin bed.

11.3.5.5. The glass module containing the PUF/XAD-2 adsorbent is wrapped with methylene chloride-rinsed aluminum foil, labeled, placed in a ziplock bag , then placed in a bubble wrap plastic bag and tightly sealed with tape for shipment.

11.4. Certification

11.4.1. At least one PUF/XAD-2 sample and filter from each batch must be analyzed as a media blank before the batch is considered for field use. A batch is considered a group of up to 20 assembled PUF/XAD-2 sample cartridges with quartz fiber filters

11.4.2. Each batch of cleaned and certified cartridges including QC samples should be shipped to the field, and sampled within the same delivery group (SDG) to the extent possible.

11.4.3. An assembled cartridge consists of the PUF/XAD-2 and filter. All these components may be extracted and analyzed together. No target compounds should be present above the reporting limit in an acceptable "pre-cleaned" blank. Surrogate compound recoveries should be within acceptable limits. If target compounds are present, the associated batch of media will be rinsed again and re-analyzed.

11.5. Pre-spike samples (optional, upon client request).

11.5.1. Prior to field deployment, a surrogate compound ($^{13}C_6$-Hexachlorobenzene or any other suitable isotopically labeled surrogate) may be added to the PUF/XAD-2 cartridge. The surrogate compound should be added to each cartridge. See Appendix II.

11.6. Storage of Spike Certified Adsorbent Cartridge

11.6.1. The PUF/XAD-2 sample cartridges must be stored in air-tight glass or metal containers, protected from light and refrigerated at 4 $\pm$2 °C from the time of

preparation. The cartridges must be wrapped in aluminum foil for protection from sunlight and external contamination.

11.6.2. The PUF/XAD-2 sample cartridges should not be stored for more than thirty days prior to sampling. The expiration date must be included on the cartridge label.

11.6.3. Label each lot as (date prepared; client; series #). Log into the PUF preparation tracking log.

11.7. Extraction Procedure

11.7.1. Soxhlet Extraction Procedure

11.7.1.1. Assemble the soxhlet apparatus. The soxhlet apparatus consists of a triple solvent rinsed 1 liter round bottom flask containing 2 to 3 methylene chloride-rinsed boiling chips, with a solvent rinsed large extractor body attached.

11.7.1.2. Add 800 mL of methylene chloride to the soxhlet apparatus..

11.7.1.3. Using cotton gloves, transfer the PUF/XAD-2 sample cartridge and filter to the soxhlet apparatus. The PUF/XAD-2 sample cartridge and filter are analyzed together.

11.7.1.4. Add a certified clean PUF/XAD-2 sample cartridge to be used as a Method Blank.

11.7.1.5. Add the surrogate solution to all samples, including the Method Blank and DCS. (See Appendix II for spike level.) Add spike solution to the DCS. (See Appendix II for spike level.) ***Note:*** ***Add the pre-spike solution to the method blank if samples were spiked prior to field deployment.***

11.7.1.6. Reflux the sample for a minimum of 16 hours at an extraction rate of approximately 4 cycles per hour.

11.7.1.7. The heating elements are turned off, and the samples are allowed to cool approximately one hour.

11.7.1.8. Remove the condenser and rinse the extractor with 10-20 mLs of methylene chloride. The soxhlet apparatus is tilted to allow the solvent to drain into the round bottom flask. The extractor body is removed and the lower joint is rinsed with 2-3 mLs methylene chloride into the round bottom flask. The PUF/XAD-2 cartridge is carefully removed and the extractor body is rinsed twice with 5-10 mLs of methylene chloride into the round bottom flask. The extracts are now ready for concentration by K-D.

11.7.2. Concentration Procedure

11.7.2.1. Assemble a Kuderna-Danish concentrator by attaching a 10 mL concentrator tube to a 1000 mL evaporative flask. Add 2 -3 methylene chloride-rinsed boiling chips.

11.7.2.2. Transfer the extract by pouring the extract through funnel and collect the extract in the K-D concentrator. Rinse the round bottom flask 2-3 times with 5-10 mLs methylene chloride to complete the quantitative transfer.

Note: If one or more of the sample extracts appears to have water present, ALL the extracts (including the Method Blank and DCS) must be poured through a drying funnel containing methylene chloride-rinsed anhydrous, granular sodium sulfate, plugged with methylene chloride-rinsed glass wool.

11.7.2.3. Pour the sample through the funnel, rinse the round bottom flask 3 times with methylene chloride and pour the rinse through the funnel. If sodium sulfate was used in the funnel, rinse the sodium sulfate two (2) times with methylene chloride.

11.7.2.4. Attach a three-ball Snyder column to the evaporative flask. Pre-wet the Snyder column by adding about 1 mL of the extraction solvent to the top of the column. Place the K-D apparatus in a hot water bath (80°C to 85°C) so that the concentrator tube is partially immersed in the hot water, and the entire lower rounded surface of the flask is bathed with hot vapor. Adjust the vertical position of the apparatus and the water temperature as required to complete the concentration in one hour. At the proper rate of distillation, the balls of the column will

actively chatter but the chambers will not flood with condensed solvent. When the apparent volume of liquid reaches 6 mL, remove the K-D apparatus from the water bath, dry the bottom of the evaporative flask, K-D tube and joint with 5-10 mLs acetone. Add 1-2 mLs of methylene chloride to the Snyder column to rinse the inside of the flask into the K-D tube. Allow to drain and cool. Remove the Snyder column. Remove the K-D tube and rinse the joint of the flask with 0.5-1.0 mLs of methylene chloride into the K-D tube.

11.7.2.5. Quantitatively transfer the extracts from the K-D tubes to 16 mL culture tubes using a disposable pipette. Rinse the sides and bottom of the K-D tube 2-3 times with ~ 0.5 mL methylene chloride to complete the transfer.

11.7.2.5.1. Nitrogen Blowdown - evaporate the solvent volume of the extracts under a gentle stream of nitrogen. Concentrate to approximately 3 mLs, cap with Teflon lined screw caps, and gently invert the extract several times to rinse the sides of the culture tube.

11.8. Remove the caps and continue to blow down the extract to slightly below 1 mL. Do not allow the extract to go to dryness. Carefully transfer the extracts to a Teflon-sealed 2 mL screw cap autovial. Complete the quantitative transfer by rinsing the bottom and sides of the internal wall of the culture tube with small amounts of methylene chloride. Adjust the final volume to 1.0 mL. Extracts are now ready for analysis by GC/MS.

12. METHOD PERFORMANCE

12.1. The group/team leader has the responsibility to ensure that this procedure is performed by an associate who has been properly trained in its use and has the required expertise.

13. POLLUTION PREVENTION

13.1. No solvents of any kind or in any amount are to be disposed of in the sinks or evaporated in the hoods.

14. WASTE MANAGEMENT

14.1. Waste management in the procedure must be segregated and disposed according to the facility hazardous waste procedures. The Environmental Health and Safety Coordinator or Hazardous Materials Technician should be contacted if additional information is required.

14.2. All waste must be disposed of according to the facility hazardous waste management procedures, Attachment C of the Chemical Hygiene Plan, Section WS002, Table 1.

14.3. Samples and other solutions containing high concentrations of toxic materials must be disposed of according to the facility hazardous waste management procedures, Attachment C of the Chemical Hygience Plan, Section WS003, Disposal of Samples After Analysis.

15. REFERENCES

15.1. Compendium Method TO-13 - The Determination of Benzo(a)pyrene and other Polynuclear Aromatic Hydrocarbons in Ambient Air Using Gas Chromatographic and High Performance Liquid Chromatographic Analysis.

15.2. Method 8270, SW846, Third Edition, USEPA Office of Solid Waste and Emergency Response, Washington D.C., 20460, September 1986.

15.3. SW846, Test Methods for Evaluating Solid Waste, Third Edition, Update II, October 1994, Semivolatile Organic Compounds by Gas Chromatography/Mass Spectrometry (GC/MS): Capillary Column Technique, Method 8270B.

15.4. USEPA Contract Laboratory Program, Statement Of Work for Organic Analysis, OLM0X.X.

15.5. "GCMS Analysis of Semi-volatile Organics", LM-CAL-3004, revision 4.0.

15.6. "GCMS Analysis Based on Method 8270B, SW846", CORP-MS-0001, revision 1.0.

16. MISCELLANEOUS

16.1. Deviations from reference method.

16.1.1. TO-13 was published for the analysis of PAH's only, other semivolatiles can be analyzed at reporting limits similar to the PAH's reporting limits.

16.1.2. The quartz filter, XAD-2 resin and PUF adsorbent are cleaned separately in batches, assembled as a sample cartridge, extracted, and analyzed for contaminants of interest. TO-13 lists the individual extraction, clean-up and analysis of the PUF adsorbent, XAD-2 resin, and quartz fiber filter.

16.1.3. TO-13 states glass fiber filters are used for sampling. This SOP references the option to use quartz fiber filters for sampling.

16.1.4. TO-13 states drying XAD-2 resin in a vacuum oven with ultra-pure nitrogen gas stream at room temperature for 2-4 hours. This SOP references XAD-2 resin to be dried in a fume hood for a minimum of 16 hours before use.

16.1.5. TO-13 uses hexane-rinsed aluminum foil. This SOP uses methylene chloride-rinsed aluminum foil.

16.1.6. Section 11.4 is not referenced in TO-13.

16.1.7. Section 11.5 is not referenced in TO-13 and is optional, upon client request.

16.1.8. Section 11.7.1 soxhlet procedure does not include a pre-cleaning extraction of 800 mL methylene chloride of the soxhlet apparatus. The extraction solvent is methylene chloride instead of the 10% ether/hexane extraction solvent referenced in TO-13.

16.1.9. TO-13 lists a concentration step which involves a solvent exchange to cyclohexane and to a final extract volume of 1 mL in cyclohexane. This SOP extraction and concentration steps are processed with methylene chloride. The final sample extract is at 1.0 mL in methylene chloride.

16.2. List of other SOPs cross-referenced in SOP.

16.2.1. SAC-QA-0041 Calibration and Calibration Check of Balances, Rev. 1.1.

16.2.2. LP-CAL-0001 Laboratory Documentation Practices, Rev. 3.0.

16.2.3. QA Policy 003 Quanterra Quality Control Program, Rev. 0.

16.2.4. QA Policy 008 Data Recording Requirements, Rev. 0.

16.2.5. LP-CAL-0004 Sample Receipt and Login, Rev. 4.0.

16.2.6. LM-CAL-3004, "GCMS Analysis of Semi-volatile Organics", Rev. 4.0.

16.2.7. CORP-MS-0001, "GCMS Analysis Based on Method 8270B, SW846", Rev. 1.0.

16.2.8. Chemical Hygiene Plan, Attachment C, Hazardous Waste Management Procedures, Revision 0.

APPENDIX I

Method Analytes and CAS Registry Numbers

Analyte	CAS #	Analyte	CAS #
Phenol	108-95-2	Acenaphthene	83-32-9
bis-(2-Chloroethyl)ether	111-44-4	2,4-Dinitrophenol	51-28-5
2-Chlorophenol	95-57-8	4-Nitrophenol	100-02-7
1,3-Dichlorobenzene	541-73-1	Dibenzofuran	132-64-9
1,4-Dichlorobenzene	106-46-7	2,4-Dinitrotoluene	121-14-2
Benzyl alcohol	100-51-6	2,6-Dinitrotoluene	606-20-2
1,2-Dichlorobenzene	95-50-1	Diethyl phthalate	84-66-2
2-Methylphenol	95-48-7	4-Chlorophenyl phenyl ether	7005-72-3
bis(2-Chloroisopropyl) ether	108-60-1	Fluorene	86-73-7
4-Methylphenol	106-44-5	4-Nitroaniline	100-01-6
n-Nitroso-di-n-propylamine	621-64-7	4,6-Dinitro-2-methylphenol	534-52-1
Hexachloroethane	67-72-1	N-Nitrosodiphenylamine	86-30-6
Nitrobenzene	98-95-3	4-Bromophenyl phenyl ether	101-55-3
Isophorone	78-59-1	Hexachlorobenzene	118-74-1
2-Nitrophenol	88-75-5	Pentachlorophenol	87-86-5
2,4-Dimethylphenol	105-67-9	Phenanthrene	85-01-8
Benzoic acid	65-85-0	Anthracene	120-12-7
bis(2-Chloroethoxy)methane	111-91-1	Di-n-butyl phthalate	84-74-2
2,4-Dichlorophenol	120-83-2	Fluoranthene	206-44-0
1,2,4-Trichlorobenzene	120-82-1	Pyrene	129-00-0
Naphthalene	91-20-3	Butyl benzyl phthalate	85-68-7
4-Chloroaniline	106-47-8	3,3'-Dichlorobenzidine	91-94-1
Hexachlorobutadiene	87-68-3	Benzo(a)anthracene	56-55-3
4-Chloro-3-methylphenol	59-50-7	bis(2-Ethylhexyl)phthalate	117-81-7
2-Methylnaphthalene	91-57-6	Chrysene	218-01-9
Hexachlorocyclopentadiene	77-47-4	Di-n-octyl phthalate	117-84-0
2,4,6-Trichlorophenol	88-06-2	Benzo(b)fluoranthene	205-99-2
2,4,5-Trichlorophenol	95-95-4	Benzo(k)fluoranthene	207-08-9
2-Chloronaphthalene	91-58-7	Benzo(a)pyrene	50-32-8
2-Nitroaniline	88-74-4	Indeno(1,2,3-c,d)pyrene	193-39-5
Dimethyl phthalate	99-65-0	Dibenzo(a,h)anthracene	53-70-3
Acenaphthylene	208-96-8	Benzo(g,h,i)perylene	191-24-2
3-Nitroaniline	99-09-2		

APPENDIX II

SURROGATE AND DUPLICATE CONTROL SAMPLE SPIKES

SURROGATE SPIKING

Surrogates are added to all the samples, including the MB (which is also the SCS), and the DCS. Add 0.5 mL of the ABN surrogate solution to each sample regardless of size, to yield spike levels shown below.

Analyte	SW846 (ug/sample)	CLP SOW 3/90 (ug/sample)
2-Fluorophenol	100	75
Phenol-d5	100	75
2,4,6-Tribromophenol	100	75
2-Fluorobiphenyl	50	50
Terphenyl-d14	50	50
Nitrobenzene-d5	50	50
2-Chlorophenol	NA	75
1,2-Dichlorobenzene-d4	NA	50

NA= Not Applicable

Pre-Spike Surrogate (by client request only)

Analyte	Amount (ug/sample)
$^{13}C_6$ Hexachlorobenzene*	50

* or any other suitable isotopically labeled surrogate

DCS SPIKING

Spike the DCS with 0.5 mL of the ABN DCS spiking solution to yield the spike levels shown below.

Analyte	SW846 (ug/sample)	CLP SOW 3/90 (ug/sample)
Pentachlorophenol	100	75
Phenol	100	75
2-Chlorophenol	100	75
4-Chloro-3-methylphenol	100	75
4-Nitrophenol	100	75
1,2,4-Trichlorobenzene	50	50
Acenaphthene	50	50
2,4-Dinitrotoluene	50	50
Pyrene	50	50
n-Nitroso-di-n-propylamine	50	50
1,4-Dichlorobenzene	50	50

3. QUANTERRA SOP FOR TO-14 VOCs ANALYSIS

Quanterra
Environmental Services

Controlled Copy
Copy No._____

SOP No. COI-MS-0003
Revision No. 0
Revision Date: 04/09/96

OPERATION-SPECIFIC STANDARD OPERATING PROCEDURE

TITLE: DETERMINATION OF VOLATILE ORGANICS (VOCS) IN AMBIENT AIR BY GC/MS - SCAN MODE USING EPA METHOD TO-14

(SUPERSEDES: LM-ATL-7001)

Prepared by: Mark Johnson

Reviewed by: _____
GC/MS Chemist, Mark Johnson

Approved by: _____
Quality Assurance Manager, Sevdâ Aleckson

Approved by: _____
Environmental Health and Safety Coordinator, Manny Velazquez

Approved by: _____
Laboratory Manager, Val Mallari

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1. SCOPE AND APPLICATION

1.1. Analytes: VOCs with molecular weights in the general range of 40-200 g/mol suitable for GC/MS analysis. Target analytes are listed in Table 1.

1.2. Method detection limits (see current MDL study).

1.3. Reporting limits: in the general range of 2-20 ppbv (See Table 1).

1.4. Applicable matrices - air, vapor

1.5. Dynamic range (See Table 1)

PROPRIETARY

1.6. Approximate analytical time

1 min. - cool down of cryotrap
1 min. - flush of inlet system with internal standard
1 min. - collection of 100 ml internal standard on trap
1 min. - flush of inlet system on trap
5 min. - collection of 500 ml of sample/standard on trap
1 min. - flush of trap with HP Helium and GC oven cool down
24 min. - GC run time

When running multiple samples, steps can be overlapped to reduce run time to approximately 25 min.

2. SUMMARY OF METHOD

2.1. An air sample is metered through a mass flow controller and concentrated onto a cryogenically cooled trap. After 100 ml of internal standard and 500 ml (nominal) of the sample has been trapped, a valve is switched and the trap is heated to purge the trap's contents onto the gas chromatography column. The target compounds are analyzed with a mass spectrometer operated in the scan mode.

3. DEFINITIONS

3.1. Standard pressure is defined as 1.0 atmosphere, 14.6 psia, 0" Hg, and 0 psig, assuming Quanterra Air Toxics Lab, City of Industry elevation and average barometric pressure.

3.2. Standard molar volume is defined as 24.5 l/mol at room temperature.

PROPRIETARY

4. INTERFERENCES

4.1. Gas regulators are cleaned by the manufacturer using Freon 113, which is one of the target compounds. Before using ultra high purity (UHP) Nitrogen (N_2), Hydrocarbon (HC) free air, Internal Standard (I.S.), or a target compound standard mix, each regulator should be purged a minimum of three times with the appropriate gas.

4.2. Contamination may occur in sample or standard results if canisters are not properly cleaned prior to use. Canisters should not be used for the collection of samples until a batch blank analysis indicates that no target compounds are present above 0.2 ppbv, or a level previously agreed upon by Quanterra and Client. All other sampling equipment including pumps, flow controllers and filters must be thoroughly cleaned to ensure that the filling apparatus will not contaminate samples.

4.3. High levels of CO_2 and/or moisture may limit the amount of sample that can be concentrated due to plugging of the trap. High levels of CO_2 may also over-pressurize the instrument's vacuum system requiring delay of scan start time or the reduction of amount of sample trapped. High levels of CO_2 have been shown to shift the retention time of the first several target compounds earlier.

5. SAFETY

5.1. Procedures shall be carried out in a manner that protects the health and safety of all Quanterra associates.

5.2. Eye protection that satisfied ANSI Z87.1 (as per the Chemical Hygiene Plan), laboratory coat, and appropriate gloves should be worn while samples, standards, solvents, and reagents are being handled. Disposable gloves that have been contaminated will be removed and discarded; other gloves will be cleaned immediately.

5.3. The health and safety hazards of many of the chemicals used in this procedure have not been fully defined. Additional health and safety information can be obtained from the Material Safety Data Sheets (MSDS) maintained in the laboratory.

5.4. Exposure to chemicals must be maintained **as low as reasonably achievable**, therefore, unless they are known to be non-hazardous, all samples must be opened, transferred and prepared in a fume hood, or under other means of mechanical ventilation. Solvent and waste containers will be kept closed unless transfers are being made.

5.5. The preparation of standards and reagents will be conducted in a fume hood with the sash closed as far as the operation will permit.

5.6. At no time should SUMMA canisters be pressurized over 40 psig.

5.7. All work must be stopped in the event of a known or potential compromise to the health and safety of a Quanterra associate. The situation must be reported **immediately** to a laboratory supervisor.

6. EQUIPMENT AND SUPPLIES

6.1. Gas chromatograph - capable of subambient temperature programming for the oven and with the jet separator option (Hewlett Packard 5890).

6.2. Mass-selective detector - equipped with computer and appropriate software (Hewlett Packard 5970B with HP-1000 RTE-A data system).

6.3. Cryogenic trap with temperature control assembly (Nutech 8533 and 3538). See Figure 1.

6.4. Electronic mass flow controller - for maintaining constant sample flow through Nutech concentrators (Unit Instruments)

6.5. Chromatographic grade stainless steel or nickel tubing and stainless steel plumbing fittings.

6.6. Chromatographic column - DB-624 0.53 ID, 30 meter length (J&W Scientific).

6.7. Stainless steel vacuum/pressure gauge capable of measuring from 30" of mercury (Hg) to 40 psig (Span Instruments) or equivalent.

6.8. High precision vacuum gauge - for making daily standards. (Wallace & Tiernan Pennwalt)

6.9. Pressure regulators for carrier gas and standards - 2 stage, stainless steel diaphragm (single stage acceptable for standards).

6.10. SUMMA passivated canisters 6 L or 15 L (Scientific Instrumentation Specialists, Anderson Instruments) or equivalent.

6.11. 7 micron filters (Nupro), or equivalent.

6.12. Vacuum flow regulators capable of flow settings to give required time sample flows (Valin or equivalent).

6.13. Diaphragm type vacuum pump to draw sample through mass flow controller.

7. REAGENTS AND STANDARDS

7.1. 4-Bromofluorobenzene, 25 ng/uL in methanol (for tuning of mass spectrometer)

7.2. High purity air and helium for making dilutions and for carrier gas.

7.3. Pressurized air source for valve actuation.

7.4. Gas calibration stock standards are at a nominal concentration of 1 ppmv (CS_2 is not as stable and so the concentration is 5 ppmv). Standards are prepared in a balance gas of nitrogen and are analytically certified by the supplier (Scott-Marrin, Scott Specialty, or equivalent). To facilitate certification by vendor, the standards were divided into several cylinders. (See current stock cylinder standard certifications)

7.5. Internal standard mix of Bromochloromethane, 1,4-Difluorobenzene, and Chlorobenzene-d5 at 250 ppbv each in a balance gas of nitrogen (Scott-Marrin or equivalent).

8. SAMPLE COLLECTION, PRESERVATION AND STORAGE

8.1. Samples should be collected in precleaned and batch analyzed SUMMA passivated canisters. A 7 micron filter should be placed on the inlet of the can to protect the valve from particulates. Canisters should never be pressurized over 40 psig.

8.2. The absolute pressure of the canister should be recorded before and after sample collection. See Section 11.1 Sample preparation.

8.3. Samples must be kept within the temperature range of 20 to 30 °C.

8.4. Tedlar bag samples should be protected from sunlight.

8.5. Canisters samples should be analyzed within 14 days of collection.

8.6. Tedlar Bag samples should be analyzed within 3 days of collection.

9. QUALITY CONTROL

9.1. For every 12 hour shift the mass spectrometer must meet the tuning criteria described in Section 11.2.

9.2. After tuning, a single point check standard must be analyzed. Ninety percent of the target compound response factors must be within ± 30 percent difference of the initial multipoint calibration curve average response factors. If the check standard fails to meet this criterion, the system and standard conditions should be evaluated and the standard reanalyzed. If the reanalysis fails to meet linearity criteria a new five point calibration curve must be run.

9.3. A laboratory control sample (LCS) must be analyzed daily or every 20 samples, whichever is more frequent. This sample will consist of target VOCs prepared in a separate canister from a second source. Five compounds will be used to assess control for the LCS: methylene chloride, 1,1-dichloroethene, trichloroethene, toluene and 1,1-2,2-tetrachloroethane. The percent recovery for the five control compounds must be within a window of 80-115% or a window established using historical lab data. QC sample limits may change once established using historical lab data.

9.4. For each lot of 20 samples analyzed, a duplicate control sample (DCS) must be analyzed. The DCS sample is identical to the LCS in composition and source. The same LCS percent recovery criterion must be met. In addition, the relative percent difference (RPD) for the LCS and DCS must be ≤ 10%. If either of the control samples fail the criteria the system should be checked and the LCS that failed reanalyzed. Samples will not be considered reportable until the DCS criteria are met.

9.5. A method blank must be analyzed every 12 hour shift before samples. The blank results must indicate that there are no target compounds present above the reporting limits (RL). The method blank is prepared by adding zero grade air to a SUMMA canister. Internal standards are added to the trap and the blank is processed exactly as a sample or standard.

9.6. If any of the above criteria are not met, corrective actions must be implemented before analyses can proceed.

9.7. Internal Standards and their associated key ions are noted in Table 2. The internal standard areas are monitored for each 12 hour shift by comparing the areas of the internal standards in each sample with the areas of the internal standards in the daily continuing calibration standard. Sample areas are considered acceptable if they fall between 50% and 150% of the daily standard areas. Any sample outside this criteria should be reanalyzed. Samples failing upon repeat analysis should be documented on the analysis benchsheet and in the report narrative. The internal standard area of Bromochloromethane should always be greater than 50,000 area counts to reliably achieve standard reporting limits

10. CALIBRATION AND STANDARDIZATION

10.1. A low level static dilution of the stock standard gas mixtures is made in a 6 or 15 liter SUMMA canister. The high precision vacuum gauge is flushed with HC free air and attached to the top valve of the clean, evacuated canister. After recording the absolute pressure, 2.00 psi of each of the 4 standard mixtures and 1.00 psi of CS2 are added to the canister (each regulator and the transfer line should be flushed several times before transfer of standard to the canister). Close the canister valves and replace the high precision gauge with a vacuum/pressure gauge of known accuracy. Pressurize the can with HC free air to 30 psig. This will yield a standard with a nominal concentration of 45 ppbv for most compounds. Actual concentrations of each compound as certified should be used in all calculations.

10.2. A high level static dilution of the stock standard gas mixtures is made in a 6 or 15 liter SUMMA canister. The high precision vacuum gauge is flushed with HC free air and attached to the top valve of the clean, evacuated canister. After recording the absolute pressure, 8.00 psi of each of the 4 standard mixtures and 2.00 psi of CS2 are added to the canister (each regulator and the transfer line should be flushed several times before transfer of standard to the canister). Close the canister valves and replace the high precision gauge with a vacuum/pressure gauge of known accuracy. Pressurize the can with HC free air to 30 psig. This will yield a standard with a nominal concentration of 179 ppbv for most compounds. Actual concentrations of each compound as certified should be used in all calculations.

10.3. An initial, minimum 5 point, curve is run in the linear working range of the system for each compound. The nominal concentration of the standards are 2.2, 4.5, 11, 45, 144, 287, and 448 ppbv. More than 5 levels are run in order to maximize each compound's linear working range and to verify reporting limits. These concentrations are achieved by varying the amount of standard trapped on the instrument and/or the concentration of the standard being analyzed. A percent relative standard deviation (%RSD) is calculated for each target analyte in that analytes dynamic range as indicated in Table 1 using the calculation in section 14.4. 90% of the target compounds must be less than 30% RSD to accept the curve for analysis. Refer to section 11 for RSD calculation. All compounds should have a response factor greater than 0.05.

10.4. During every 12 hours of operation, after tuning criteria is verified, a one point midrange standard (500 mL of 44 ppbv) is run to verify linearity with the initial calibration. A percent difference is calculated between the response factors (RFs) from the continuing calibration standard and the average RFs from the initial curve for each target analyte using the calculation in section 11. 90% of the target compound response factors must be within 30% difference of the initial multipoint curve average

Response Factor, or a new initial calibration must be run. All compounds should have a response factor greater than 0.05.

10.5. The daily, one point check standard response factors (RFs) are used to quantitate the results of the samples for the 12 hour shift.

11. PROCEDURE

11.1. Sample Preparation

11.1.1. The initial pressure of the sample canister is checked and recorded by attaching a vacuum/pressure gauge to the canister. The gauge should be rinsed before use with zero grade nitrogen by physically holding against the air outlet and flushing for a few seconds. If the pressure is less than approximately 6 psig, pressurize the canister to 10 psig with zero grade nitrogen and record the final pressure. Pressures should be recorded in the sample pressurizing log and on the individual canister field sheets.

11.1.2. If the canister pressure is increased, a dilution factor (DF) is calculated and applied to results:

$$DF = \frac{Y_a}{X_a}$$

Where:

X_a = *absolute canister pressure before dilution (initial pressure)*
Y_a = *absolute canister pressure after dilution (final pressure)*

11.2. Initial/Daily GC/MS Tuning

11.2.1. After a successful autotune as per manufacture recommendations each instrument is manually tuned using PFTBA so that m/z 69 is 100%, m/z 131 is approximately 34%, and m/z 213 is approximately 36%. The width and axis parameters are set using the routines in the software. This initial tune should remain stable for extended periods of time, and retuning with PFTBA should not be necessary every day

11.2.2. At the beginning of each 12 hour shift, prior to any analytical runs, the GC/MS system must be verified that acceptable tune performance criteria are achieved. If any of the key ions fail the abundance criteria listed in Section 11.2.4, the system is considered out of tune and any subsequent sample/standard analysis should be considered unacceptable

11.2.3. For shift tune verification the cryotrap is not used and should be left at 150C. Alternatively the trap can be cold or cooling down but should be out of line with the column. The GC program (see Table 2) is initiated by using the BAMON commands. The GC programs are named "GCBFB1" and "GCBFB2" for systems A and B respectively. Tune files are typically "MTBFB1" and "MTBFB2." Once the data system has initiated the run and GC oven has stabilized, the remote start light will turn on and the system is ready for injection.

2 uL of a 25 ng/uL 4-Bromofluorobenzene (BFB) standard is injected into the injection port of the concentrator unit and the remote start button is activated.

11.2.4. Once the tuning run is complete (~ 6 minutes), run the program TUNVOA, data file." This is a program that will evaluate the tuning analysis and print out the required information automatically. If the BFB tuning criteria cannot be met on the first injection, retuning the instrument with PFTBA and or system maintenance may be required prior to re-injection of BFB.

Mass	Ion Abundance Criteria
50	15 to 40% of mass 95
75	30 to 60% of mass 95
95	Base Peak, 100% Relative Abundance
96	5 to 9% of mass 95
173	<2% of mass 174
174	>50% of mass 95
175	5 to 9% of mass 174
176	>95% but <101% of mass 174
177	5 to 9% of mass 176

11.3. One time procedural variations are allowed only if deemed necessary and appropriate by the supervisor to accommodate variation in sample matrix, radioactivity, chemistry, sample size, or other parameters. Any variation in procedure shall be completely documented using a Nonconformance Memo including approval by a Technical Specialist and QA Manager. If it is determined that the quality of the sample data is significantly affected and/or if contractually required, the client shall be notified. The Nonconformance Memo shall be filed in the project file.

11.4. Any unauthorized deviations from this procedure must also be documented as a nonconformance, with a cause and corrective action described.

12. ANALYSIS

12.1. The continuing standard and the QA/QC samples are analyzed the same as samples. After the one-point continuing calibration standard is analyzed an evaluated (section 9.2), duplicate laboratory control samples (DCS) are analyzed and evaluated (See sections 9.3 and 9.4).

12.2. Before sample analysis a zero grade nitrogen method blank is analyzed and evaluated (see section 9.5), The method blank consists of a SUMMA canister that is filled with zero grade nitrogen.

12.3. At the beginning of a sample or standard run the sample valve is in the purge position and the trap valve is in the column position. The trap is cooled to <150C. The internal standard canister attached and approximately 100 ml is flushed through (this can be concurrent with the trap cool down). After flushing the trap valve is placed in the sample position and the trap temperature is checked to be at -150C or less.

Trapping is started by switching the sample valve to sample position and activating a timer. When 100 ml has been trapped (1 minutes at 100 ml/min) the sample valve is switched to purge and the internal standard canister is closed and removed.

12.4. The sample/standard canister is then attached and approximately 100 ml is flushed through. Trapping is again started by switching the sample valve to sample and starting a timer. After the appropriate amount of time (5 minutes for 500 ml at 100 ml/min) the sample valve is switched back to purge, the canister is closed and the data system is set up.

12.5. Typically, the "Name" field in the data system set up screen contains the Quanterra sample number and the name of the client. The "Misc." section contains the volume of the sample that was trapped, the client sample ID, the canister number (or a note of "Tedlar bag"), and the canister or Tedlar bag dilution factor (final/initial pressure, if applicable).

12.6. The GC system methods are "GCNEW1: and "GCNEW2" (see Table 3). When the GC reaches ready at the programmed start temperature, and after waiting at least 30 seconds after trapping, the trap valve is switched to column, and then the cryotrap is switched to heat and the run is started simultaneously.

12.7. A canister filled with zero grade air is attached to the sample line in between samples. The line is allowed to flush until it is time for the next sample.

12.8. Trapped sample volumes are adjusted for pressurization to give an undiluted volume of 500 ml.

13. DATA INTERPRETATION

13.1. Qualitative Analyses

13.1.1. Two criteria must be satisfied to verify positive identification. (1) elution of sample component at the same GC relative or absolute retention time as those of the standard component; and (2) correspondence of the sample component and the standard component mass spectra.

13.1.1.1. The sample component relative retention time (RRT) must compare within ± 0.06 RRT units of the RRT of the standard component. As an option, RT must compare within 0.5 minutes of the standard component absolute retention time (RT). For reference, the RT standard must be run within the same 12 hour shift as the sample.

13.1.1.2. (1) All ions present in the standard mass spectra at a relative intensity greater than 10% (most abundant ion in the spectrum equals 100%) must be present in the sample spectrum. (2) The relative intensities of ions specified in (1) must agree within plus or minus 20% between the standard reference and sample spectra. (Example: For an ion with an abundance of 50% in the reference spectra, the corresponding sample abundance must be between 30 and 70 percent. Standard reference mass spectra must be obtained on each individual system.

13.1.1.3. If a compound cannot be verified by all of the criteria in the above paragraphs but in the technical judgment of the analytical chemist the identification is correct, then the compound may be reported.

13.1.2. For samples containing components not associated with the calibration standards, a library search may be made for the purpose of tentative identification. The necessity to perform this type of identification will be determined by the type of analysis being conducted. normally, compounds will be given general names consisting of major functional groups and number of carbon atoms unless a retention time reference is available. Guidelines for making tentative identification are:

13.1.2.1. Relative intensities of major ions in the reference spectrum (ions > 10% of the most abundant ion) should be present in the sample spectrum.

13.1.2.2. The relative intensities of the major ions should agree within ± 20%. (Example: For an ion with an abundance of 50% in the standard spectrum, the corresponding sample ion abundance must be between 30 and 70%).

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

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

13.1.2.5. Ions present in the reference spectrum but not in the sample spectrum should be reviewed for possible subtraction from the sample spectrum because of background contamination or coeluting peaks. Data system library reduction programs can sometimes create these discrepancies.

13.1.2.6. Only peaks having a total ion current greater than 10% of the nearest eluting Internal Standard total ion current will be evaluated for reporting.

13.1.2.7. Semiquantitative results will be calculated for tentatively identified compounds using total ion current areas and assuming a relative response factor of 1.0 of the nearest internal standard.

Computer generated library search routines should not use normalization routines that would misrepresent the library or unknown spectra when compared to each other. Only after visual comparison of sample with the nearest library searches will the mass spectral interpretation specialist assign a tentative identification.

13.2. Quantitative Analysis:

13.2.1. When a compound has been identified, the quantification of that compound will be based on the integrated abundance from the EICP of the primary characteristic ion. Quantification will take place using the internal standard technique. A summary table of primary and secondary ions for target compounds and internal standards is listed in Table 4. The calculation for quantitation of sample is found in section 14.

14. DATA ANALYSIS AND CALCULATIONS

14.1. The HP data system is set up to automatically quantitate the sample results based on a 500 ml sample size. The default result units are in ppbv.

14.2. If a sample size other than 500 ml was used and/or a canister sample was pressurized, the result must be adjusted as shown below:

$$Final\ result\ ppbv = raw\ result\ ppbv\ X \frac{500\,mL}{sample\ volume\ injected}\ X \frac{final\ psia}{initial\ psia}$$

14.3. Calculation for Relative Response Factor (RRF):

$$RRF = \frac{Area\ cpd\ in\ Std.}{Area\ I.S.}\ X \frac{Conc.\ I.S.}{Conc.\ cpd\ in\ Std.}$$

The area of the primary quantitation ion is used in the calculation.
I.S.: Internal Standard

14.4. Calculation for Percent Relative Standard Deviation (%RSD):

$$\%RSD = \frac{Std.\ Dev.\ of\ RRFs}{Mean\ of\ RRFs}\ X\ 100$$

14.5. Calculation for Percent Difference (%D):

$$\%D = \frac{Average\ RRF\ from\ initial\ curve\ RRF\ cpd.}{Average\ RRF\ from\ initial\ curve}\ X\ 100$$

14.6. Calculation for Determining Concentration of Compounds:

$$Conc.\ Cpd(ppbv) = \frac{Area\ cpd\ in\ sample}{Area\ I.S.\ in\ sample}\ X \frac{Conc.\ I.S.}{RRF\ daily\ Std.}\ X\ Dil.\ Factor\ (See\ 14.2)$$

The area of the primary quantitation ion is used in the calculation.
I.S.: Internal Standard

14.7. Calculation for Percent Recovery (%Rec):

$$\%\,Rec = \frac{Amount\ cpd\ recovered}{Amount\ cpd.\ spiked}\ X\ 100\%$$

14.8. Calculation for Relative Percent Difference (RPD):

$$RPD = \frac{Value\ A - Value\ B}{Average\ of\ Values}\ X\ 100$$

15. REPORTING

15.1. Standard reporting units are ppbv. If results are to be reported in ng/L or ug/m^3 use the following equation:

$$result\ ppbv\ X \frac{Molecular\ weight\ of\ compound}{24.5} = results\ ng/L\ or\ ug/m^3$$

Note: 24.5 is the molar volume of ideal gas at 25° Centigrade and 1 atm.

15.2. Reporting limits

15.2.1. See Table 1. All reporting limits and MDLs must be derived on GC/MS systems at the Quanterra Air Toxics laboratory and are periodically updated.

15.2.2. "J" values (results below the normal reporting limit but above the MDL) are issued on request only.

15.3. Significant figures

15.3.1. All results should be reported to two significant figures.

15.4. No conversion of the analytical results to standard conditions is made.

16. METHOD PERFORMANCE

17. POLLUTION PREVENTION

18. WASTE MANAGEMENT

19. REFERENCES

19.1. Method Source

19.1.1. "EPA Compendium Method TO-14. The Determination of Volatile Organic Compounds (VOCs) in Ambient Air using SUMMA Passivated Canisters Sampling and Gas Chromatographic Analysis."

19.2. Deviations from Method

19.2.1. Nitrogen is used for dilution/pressurization purposes.

19.2.2. TO-14 recommends the use of a 0.32 mm column coupled directly to the MSD. With the HP system, the MSD can only handle flow of 1 ml/min or less. The 0.32 mm column provides ~ 3 ml/min. Quanterra uses a 0.53 mm column through a jet separator.

19.2.3. TO-14 describes an inlet system that uses a vacuum to pull the sample through the trap. Quanterra optionally uses the pressure of the sample canister to drive the sample through the trap.

19.2.4. TO-14 describes the use of a Nafion dryer to remove excess moisture from air matrices. Quanterra does not use a Nafion dryer since polar compounds may be lost during this removal step.

19.2.5. TO-14 describes the use of SUMMA passivated steel canisters for sampling and analysis. No mention is made of Tedlar sampling bags. Quanterra analyzes samples in Tedlar bags for VOCs using the same procedures described herein. A modification to the method is noted on the final report.

20. MISCELLANEOUS (TABLES, APPENDICES, ETC.)

Table 1 VOC Target Compounds

Compound	R.T. (approx.)	Report Limits (ppbv)	Dynamic Range (ppbv)
Dichlorodifluoromethane (Freon 12)	2.05	2.0	2-450
Chloromethane	2.62	4.0	4-450
1,2-Dichloro-1,1,2,2-tetrafluoroethane (Freon 114)	2.66	2.0	2-450
Vinyl Chloride	2.89	2.0	2-450
Bromomethane	3.34	2.0	2-450
Chloroethane	3.52	4.0	4-450
Trichlorofluoromethane (11)	3.85	2.0	2-450
1,1-Dichloroethene	4.44	2.0	2-450
Carbon Disulfide	4.54	10.0	10-360
1,1,2-Trichloro-1,2,2-trifluoroethane (Freon 113)	4.52	2.0	2-450

Compound	R.T. (approx.)	Report Limits (ppbv)	Dynamic Range (ppbv)
Hexachlorobutadiene	21.52	2.0	4-450

Compound	R.T. (approx.)	Report Limits (ppbv)	Dynamic Range (ppbv)
Acetone	4.62	10.0	10-450
Methylene Chloride	5.07	2.0	2-450
trans-1,2-Dichloroethene	5.38	2.0	2-450
1,1-Dichloroethane	5.91	2.0	2-450
Vinyl Acetate	6.13	10.0	10-450
cis-1,2-Dichloroethene	6.70	2.0	2-450
2-Butanone	6.84	10.0	10-450
Chloroform	7.23	2.0	2-450
1,1,1-Trichloroethane	7.33	2.0	2-450
Carbon Tetrachloride	7.56	2.0	2-450
Benzene	7.88	2.0	2-450
1,2-Dichloroethane	7.97	2.0	2-450
Trichloroethene	8.95	2.0	2-450
1,2-Dichloropropane	9.31	2.0	2-450
Bromodichloromethane	9.88	2.0	2-450
cis-1,3-Dichloropropene	10.70	2.0	2-450
4-Methyl-2-pentanone	11.11	10.0	10-450
Toluene	11.21	2.0	2-450
trans-1,3-Dichloropropene	11.82	2.0	2-450
1,1,2-Trichloroethane	12.12	2.0	2-450
Tetrachloroethene	12.19	2.0	2-450
2-Hexanone	12.78	30.0	30-450
Dibromochloromethane	12.80	2.0	2-450
1,2-Dibromoethane	12.90	2.0	2-450
Chlorobenzene	13.98	2.0	2-450
Ethylbenzene	14.33	2.0	2-450
1,4-and 1,3-(p,m)Xylene	14.61	2.0	2-900
1,2-(ortho)Xylene	15.43	2.0	2-450
Styrene	15.49	2.0	2-450
Bromoform	15.78	2.0	2-450
1,1,2,2-Tetrachloroethane	17.18	2.0	2-450
Benzyl Chloride	17.30	10.0	10-450
4-Ethyltoluene	17.55	2.0	2-450
1,3,5-Trimethylbenzene	17.73	2.0	2-450
1,2,4-Trimethylbenzene	18.55	2.0	2-450
1,3-Dichlorobenzene	19.02	2.0	2-450
1,4-Dichlorobenzene	19.26	2.0	2-450
1,2-Dichlorobenzene	19.83	2.0	2-450
1,2,4-Trichlorobenzene	21.32	20.0	20-450

TABLE 2 BFB GC Method

Method file: GCBFB1(2)

METHOD FILE LIST

Method file:	GCBFB1 GCBFB2	GC Type: 5890 Column: Cap	Run type: SCAN,GC,E1 Splitless: Yes	
Temperature:	Inj.P 90.0	Intfc 250.0	Source 0.0	
GC/DIP		LEVEL A	LEVEL B	POST RUN
Temp 1	30.0	100.0	0.0	0.0
Time 1	1.0	3.0	0.0	0.0
Rate	35.0	0.0	0.0	
Temp 2	100.0	0.0	0.0	
Time	15.0	0.0	0.0	

Oven equilibration Time. 0.10 min

Run time: 6.00
Scan Start time 2.50

Scan Parameters:	Mass Range:	34 to 260	
	Multiplier voltage:	varies	Number of A/D samples: 8
	GC Peak threshold:	20000 counts	
	Threshold:	100 counts	

TABLE 3 GC Analytical Method

Method file: GCNEW1(2)

METHOD FILE LIST

Method file:	GCSYS1 GCSYS2	GC Type: 5890 Column: Cap	Run type: SCAN,GC,E1 Splitless: Yes

Temperature:	Inj.P	Intfc	Source
	90.0	250.0	0.0

GC/DIP		LEVEL A	LEVEL B	POST RUN
Temp 1	-50.0	10.0	100.0	0.0
Time 1	1.5	0.0	0.0	0.0
Rate	50.0	5.5	40.0	
Temp 2	10.0	100.0	160.0	
Time	0.0	0 0	3.4	

Oven equilibration Time. 0.10 min

Run time: 24.00
Scan Start time 1.60

Scan Parameters:			
	Mass Range:	34 to 260	
	Multiplier voltage:	varies	Number of A/D samples: 8
	GC Peak threshold:	20000 counts	
	Threshold:	10 counts	

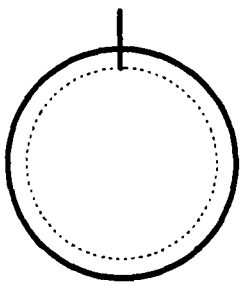
TABLE 4 VOC Key Ions

Compound	Primary	Secondary		
Bromochloromethane I.S. #1	49	130	128	
Dichlorodifluoromethane (Freon 12)	85	87,	50	
Chloromethane	50	52		
1,2-Dichloro-1,1,2,2-tetrafluoroethane (Freon 114)	85	135	87	
Vinyl Chloride	62	64		
Bromomethane	94	96,	79	
Chloroethane	64	66,	49	
Trichlorofluoromethane (11)	101	103	66	
1,1-Dichloroethene	61	96,	63,	98
Carbon Disulfide	76	78,	44	
1,1,2-Trichloro-1,2,2-trifluoroethane (Freon 113)	151	101	103	85
Acetone	43	58		
Methylene Chloride	84	49,	86	
trans-1,2-Dichloroethene	96	61,	98,	63
1,1-Dichloroethane	63	65,	83	
Vinyl Acetate	43	44,	86,	42
cis-1,2-Dichloroethene	96	61,	98,	63
2-Butanone	72	57,	43	
Chloroform	83	85,	47	
1,1,1-Trichloroethane	97	99,	61	
Carbon Tetrachloride	117	119	121	82
1,4-Difluorobenzene I.S. #2	114	63,	88	
Benzene	78	50,	52,	77
1,2-Dichloroethane	62	64,	49,	98
Trichloroethene	130	95,	132	97
1,2-Dichloropropane	63	62,	41,	39
Bromodichloromethane	83	85,	129	
cis-1,3-Dichloropropene	75	77,	39	
4-Methyl-2-pentanone	43	58,	57,	85
Toluene	91	65,	92	
Chlorobenzene-d5 I.S. #3	117	52,	54,	82
trans-1,3-Dichloropropene	75	77,	39	
1,1,2-Trichloroethane	97	83,	85,	61
Tetrachloroethene	164	129	131	166
2-Hexanone	58	43	57	
Dibromochloromethane	129	127	208	131
1,2-Dibromoethane	107	109	188	
Chlorobenzene	112	77,	114	

TABLE 4 VOC Key Ions

Compound	Primary	Secondary		
Ethylbenzene	91	106		
1,4-and 1,3-(p,m)Xylene	91	106	105	
1,2-(ortho)Xylene	91	106	105	
Styrene	104	78	103	51
Bromoform	173	171	175	93
1,1,2,2-Tetrachloroethane	83	85,	133	131
Benzyl Chloride	91	126	63	
4-Ethyltoluene	105	120	77	
1,3,5-Trimethylbenzene	105	120	77	
1,2,4-Trimethylbenzene	105	120	77	
1,3-Dichlorobenzene	146	148	111	75
1,4-Dichlorobenzene	146	148	111	75
1,2-Dichlorobenzene	146	148	111	75
1,2,4-Trichlorobenzene	180	182	109	145
Hexachlorobutadiene	225	227	223	

FIGURE 1. Nutech 8533 Flow Diagram



4. IEA SOP FOR LOG IN OF COMMERCIAL SAMPLES

1.0 APPROVALS

The signatures of the following individuals indicate that this SOP is complete and meets the requirements specified in corporate document # QAS00200.NET.

Laboratory Director

Quality Assurance Manager

UNCONTROLLED COPY
PROPRIETARY DOCUMENT

2.0 SCOPE AND APPLICATION

2.1 The objective of this document is to describe the flow of commercial samples through the Sample Management laboratory.

2.2 Areas to be addressed include a description of the login process and the paperwork involved.

3.0 SAFETY

3.1 Safety glasses, labcoats, and vinyl gloves should always be worn when handling samples.

3.2 Sample coolers should be opened under the hood over the workbench at the receiving dock door.

3.3 Extreme care should be used when handling samples with strong odors or samples that are noted by the client to be hazardous. Such sample containers should be placed in the exhaust hood of the Sample Management area for the duration of the login process.

4.0 APPARATUS AND MATERIALS

4.1 Sample Storage Refrigerators: R1, R6, R18, R23, R24, R26, R28

4.2 Computer with SAM program

4.3 Label Printer/Labels

4.4 Full Page Printer

4.5 pH paper

4.6 Capillary tubes

4.7 Preservatives (see Attachment IV)

4.7.1 Preservatives are prepared and provided by the Inorganic/Metals laboratory and requested for replenishment by Sample Management personnel, as required.

4.8 uR Meter

4.9 Infrared temperature detector gun

5.0 PROCEDURE

5.1 All coolers are scanned for radioactivity with a uR Meter prior to acceptance from a courier. See the SOP for Radiation Screening, IEA Doc# RAS02901.NC for details on the use of the uR Meter. Coolers that exceed the acceptable limit (0.5 uR/h) are not accepted and must not be received into the facility. The cooler and its contents are not signed for and are returned to the courier.

5.1.1 Notify the Department Supervisor of any cooler(s) that must be returned to a client for failed radiation screening.

5.2 Accept the cooler from the courier by recording the time of receipt and signing the Chain-of-Custody. Check the cooler for any custody seals or additional documentation that may have been submitted (i.e., letter, task order form, regulatory report form) and include with the records to be added to the project folder once login is complete.

5.2.1 Check the COC form any special instructions or information (i.e., field notes, drinking water or NPDES sample notification, RUSH request).

5.2.2 Check the temperature of the samples within the cooler(s) with the Thermo Hunter IR Gun and note the temperature on the COC. If the temperature is not in the range of 4 $\pm$ 2°C, notify the Project Manager by completing a Discrepancy Form (Attachment I).

NOTE: A single Discrepancy Form should be completed for a project with all discrepancies noted described for the Project Manager to handle at once, wherever possible.

5.2.2.1 If the client sends a temperature bottle in the cooler(s) with the sample, the temperature in this bottle should be measured first, using the IR Gun unless specific instructions require the use of an NIST traceable calibrated thermometer.

5.2.3 Confirm that the temperature was checked by recording the temperature on the COC. Confirm other conditions of sample receipt by checking the applicable blocks under the "Remarks on Sample Receipt" section of the COC.

5.2.4 If a limited protocol holding time is noted (see Attachment IV), a Discrepancy Form is completed and forwarded to the Project Manager who will notify the client and/or get approval for a rush preparation/analysis

5.2.4.1 A limited protocol holding time is defined as less than 3 days remaining on a published holding time.

For parameters that have normal protocol holding times of 48 hours or less, it is not necessary to process Discrepancies or rush approvals.

5.3 Check the COC for an IEA workquote number. Find the quote in SAM by typing in QW and the quote number (i.e., QW 9109100).

5.3.1 If a workquote does not exist for the project, complete a discrepancy form, requesting that a workquote be created by the appropriate Project Manager.

5.4 Press F5 to find the analyses that were originally quoted. Compare the quote in SAM with the parameters indicated on the COC. If differences are noted, complete a Discrepancy form and forward it to the appropriate Project Manager for quote generation.

5.5 Press F5 again to determine if a Project Memo exists. Read the "Sample Management" section of the Memo for any special instructions.

5.6 Determine the turnaround time from the quote and prioritize the samples by placing the appropriately colored sticker on the cooler.

Red	Rush turnaround
Purple	Limited Protocol (Holding time)
Green	5 day TPH-GC
Yellow	10 day turnaround
Orange	15 day (or longer) turnaround

A combination of stickers may be used to reflect what is indicated on the COC.

5.7 Write the date of receipt and the analyses required on the sticker for quick reference.

5.8 Arrange the coolers in order of priority for login as follows:

1 RUSHES
2 Limited Protocol
3 5-day TPH-GC
4 10-day Turnaround
5 $\geq$ 15 day Turnaround

5.9 Sample Management will contact the department(s) that have limited protocol samples. Samples having limited protocols should be recorded in the Limited Protocol Logbook (Attachment II).

5.10 Record the project in the Daily Receipt Log (Attachment III). For clients that have multiple locations, record the location also.

5.11 Remove the samples from the cooler for login. Arrange the samples on the countertop in the order indicated on the COC. If at any time during the login process a discrepancy is noted or if there are legibility problems associated with the COC or labels, a Discrepancy form should be completed and forwarded to the appropriate Project Manager in that instance.

5.12 Observe the samples for the presence of custody seals. If custody seals are present and they are intact, note this on the COC. If custody seals are not intact, note this on the COC, complete a Discrepancy form and forward it to the appropriate Project Manager.

5.13 Observe the containers for cracks or breaks. Note the condition of the samples as received intact or identify the number of sample(s) and sample IDs that were broken on the COC. Complete a Discrepancy form and forward it to the appropriate Project Manager for any samples that were not received intact.

5.14 If a sample received requires analysis from different departments, determine if sufficient sample was shipped. If not, a Discrepancy form is completed and forwarded to the appropriate Project Manager.

5.15 If a sample requires both volatile and non-volatile analyses from the same container, the sample is stored in the appropriate volatiles refrigerator. All samples scheduled for storage in the volatiles refrigerators are required to be delivered to the assigned refrigerator by the person performing the login of the project.

5.16 Check the Preservation Table (Attachment IV) for correct container sizes and preservation.

5.16.1 Check the pH of non-volatile samples by removing a small portion from the sample bottle with a capillary tube and placing a drop of sample onto wide range pH paper. **NEVER PUT pH PAPER IN THE SAMPLE CONTAINER.** If the pH indicates the need for pH adjustment to meet preservation requirements, forward a Discrepency form to the Project Manager. The client will be contacted to determine if pH adjustment will occur in Sample Management.

5.16.2 If pH adjustment is required, remove the Sample to the Sample Management hood and add the appropriate preservative (using Attachment IV, Preservation Table).

5.17 Enter the client/workquote information from the COC in the SAM system. While in the workquote, verify that the COC/client information is correct and add any other pertinent information including: Client Project Manager, PO number, Client work ID, specific client sample IDs and date collected, etc. If any discrepancy is noted between the workquote and the COC, use the COC as the guide. Complete a Discrepancy form and forward to the appropriate Project Manager to verify the change and notify the client, as necessary.

5.18 Press F5 to the go the Quote Analytical Screen. Verify that all parameters listed on the COC appear on the screen. Parameter test and/or job codes can be added as needed.

5.19 To receive the codes, enter the correct number of samples in the "Rec" field, press F8 and then Y. This transfers the job and test codes to the workorder.

5.20 Press F5 and choose the "go to ord" option from the menu. The number for the project will automatically be chosen and will appear in the "CAT" field. This number must be noted on the COC. In the "Contact" field, press ALT C to enter "Client Relat".

5.21 Enter the appropriate report due date, matrix, and receipt date. Enter the lab due date in the "Date Due" field and the number of days that the sample will be kept after the report is mailed in the "Keep for" field. The keep date is typically 15 days for standard Level I, II and III projects. Commercial CLP project samples are held for 30 days, unless otherwise specified by the Project Manager/memo.

5.22 The "Disp" field is used to define the method of disposal of any unused remaining raw sample after analysis. Use the following codes when completing this field:

5.22.1 "D" indicates that the samples should be disposed of normally.

5.22.2 "S" indicates that the samples should be disposed of as hazardous.

5.22.3 "R" indicates that the samples will be returned to the client. When the samples are to be returned to the client, the cover of the project folder is stamped "Samples to be Returned" and the original COC will be placed in a folder bin identified for this purpose in Sample Management. The COC will be returned with the samples to the client.

5.22.4 "A" indicates that disposal is pending the clients' decision.

5.22.5 See the SOP for Sample Storage Purge, IEA Doc# SMS01100.NC for details of sample removal/disposal procedures.

5.23 Press Crtl F5 to go to the AUTOFRAC Screen. Verify that the "Samps/Frac" fields are correct. A base sample ID (such as an MW precursor) is entered, if applicable, in the "Default ID" field. Enter the sampling dates in the "data Collected default" field. Verify that the sample matrices are correct. Press F5. The Sample ID, Date Collected, and Category will be completed based on the information that was entered earlier (see Section 5.17). Any changes that need to be made may be entered here.

5.24 Arrow down to the test and job codes to verify that they have been entered correctly. Press ALT F5. The "Fracs" field at the top of the screen will be filled in. Verify that the number in the "Fracs" field is the total number of bottles received.

5.25 Page down to the "Location by Fraction Type" field. Locations for the actual sample bottles are determined by the requested parameters. Refrigerator assignments are as follows:

R28:	GC/MS Volatiles
R26:	Dedicated Client Volatiles*
R23:	Total/Fecal Coliform Samples
R18:	Semivolatiles, Inorganics, TPH, CLP, Metals Soil Samples
R6:	GC Volatile
R1:	GC/MS Volatile
R24:	Dedicated Client Volatiles*
Shelf Room:	Metals (Waters)

Three 40 mL vials are provided to clients for GC and GC/MS water volatile samples. All three vials are placed in a tray or box in R1 (GC/MS), R6 (GC), R28 (GCMS), R28 or R26, as appropriate.

*Only samples vials received from the client to which the refrigerator has been assigned may be stored here.

5.26 If the project is a CLP project received as a Sample Delivery Group (SDG), follow the procedures identified the the SOP for CLP Inorganic Sample Receipt, IEA Doc# SMS00900.NC and/or the SOP for CLP Organic Sample Receipt, IEA Doc# SMS00206.NC.

5.27 After all samples have been logged in, generate a bar code label for the project folder cover and labels for each of the sample bottles received. Do so by typing labl_bar and the workorder #/F for the bar code and labl_tst and the workorder #/F for the bottle labels. Attach the appropriate label to its corresponding sample bottle. Be sure that the SAM generated bottle labels match the parameter written on the sample bottle. Make sure that the sample ID from the SAM label matches the sample bottle ID.

Attach the bar code label to the outside of the project folder.

5.28 Generate the Data for Project Sheet, Department Sign-Off Page and the Project Memo (if applicable). Do so by going to the J(1): prompt. Press C and Enter to change the printer selection from the label printer to the laser printer. Type Project and the project Number. Attach the Data for Project Sheet, the Project Memo and any other documentation received with the project to the left inside flap of the project folder and the Department Sign-Off Sheet to the right flap of the project folder.

5.29 Hand write the project number and the lab due date on a file folder label and attach the label to the folder tab.

5.30 Attach any color code stickers necessary to the appropriate location on the folder.

Red:	Rush project
Red on top of the folder tab label:	5 day TPH
Orange on top of the folder tab label:	Radiological parameters

5.31 If the project is a Level III or CLP QC type or has specific requirement for internal COCs in the memo, generate the internal COC for each sample through the SAM system, using the COCs command followed by the complete workorder number.

5.32 Complete the Login Checklist (Attachment V) to verify that the login procedure has been completed and that it is correct.

5.33 A completed folder consists of:

- Inside Left Project Folder Flap:
  - Data for Project Sheet
  - Error Sheet
  - Project Memo (If applicable)
  - Original COC
  - Sample Login Checklist
  - Relevant Paperwork (i.e., Discrepancy, letter, form)
- Inside Right Project Folder Flap:
  - Department Sign-Off Sheet
  - Airbill (if available)
- 3 unattached copies of the COC

5.34 Place the samples on a cart. The Sample Custodian will distribute them to the appropriate refrigerator/storage area. EXCEPTION: All volatile samples are delivered to the appropriate volatiles storage refrigerator by the person who logged them in.

5.34.1 Samples that are scheduled for analyses to be performed by the radiological facility will be transferred under Chain of Custody, using the Lab Courier Delivery Form (see the SOP for Chain of Custody, IEA Doc# QAS01301.NC).

5.34.2 When samples are needed for analysis in the immediate facility, each lab will request the samples by completing a Sample Request/Delivery Form (Attachment VI) and submitting it to the Sample Custodian. The Sample Custodian will pull the samples requested and notify the laboratory to pick up the samples from the Sample Management area.

5.35 In the event that a discrepancy is submitted to a Project Manager, the samples should be replaced in the cooler(s) in which they arrived and/or stored in R18 to maintain appropriate temperature for storage of samples.

6.0 SUPPLEMENTAL DOCUMENTS

6.1 SOP for Chain of Custody, IEA Doc# QAS01301.NC.

6.2 SOP for IR Temperature Gun, IEA Doc# SMS01000.NC.

6.3 SOP for Sample Storage Purge, IEA Doc# SMS01100.NC.

6.4 SOP for Radiation Screening, IEA Doc# RAS02901.NC.

6.5 SOP for Project Transaction, IEA Doc# GES00500.NC.

7.0 REFERENCES

7.1 Employee Chemical Safety Handbook, IEA Doc# SFS00300.NC.

7.2 Laboratory Health & Safety Plan, IEA Doc# SFS00200.NC.

8.0 SUBSTANTIVE REVISIONS

8.1 Section 3.2 added. Section 4.5, 4.6, 4.7, 4.8 and 4.9 added. Section 5.0 Revised to be more explicit in the login procedure. Sections 6.1 through 6.5 added. Sections 7.1 and 7.2 added.

Attachment I

IEA Corporation

CLIENT PROJECT DISCREPANCY REPORT

Project No. _____ Client _____

Project Manager _____ Phone No. (____) ____ - _____

Discrepancy

1.

2.

3.

4.

Samples Received:	1) Out of Protocol	Yes	No
	2) Chilled*	Yes	No
	3) Other	Yes	No
	4) Matrix	Soil	Water

Comments:

Initiated By: _____ Date: __/__/__ Time: __:__

*Not Required for Metals

Resolution:

1.

2.

3.

4.

Client Contacted	Yes	No	Date:	__/__/__
Analyze Samples	Yes	No	Time:	__:__
Will Resample	Yes	No	Day Zero:	_____
Send Transpak	Yes	No		

Resolved By: _____ **Date:** __/__/__ **Time:** :

Secondary Review By: _____ **Date:** __/__/__

Attachment II

LIMITED PROTOCOL

Date:_____

SAMPLING DATE	SAMPLING TIME	BOD	COLOR	MBAS	O-PO_4	NO_3	TURB	SM	BAC-T	CR^{+6}	CLIENT	LAB NOTIFIED BY	TIME NOTIFIED	ANALYST NOTIFIED

OTHER SAMPLES WITH LESS THAN THREE PROTOCOL DAYS REMAINING

CLIENT	SAMPLING DATE	SAMPLING TIME	3030 C	OTHER PARAMETERS	# OF SAMPLES	LAB NOTIFIED BY	TIME NOTIFIED	ANALYST NOTIFIED

Secondary Review:_____Date:_____

Attachment III

DAILY RECEIPT LOG

DATE:_____

IEA REP	COMPANY NAME	COURIER	PKGS.	AIR BILL#

Secondary Review By:_____ Date:_____

IEA, Inc. Form# SML01200.NC

IEA Logbook# SM6*

Attachment IV

Metals in Water					
Parameter[3]	Technique	Method	Holding Time	Container	Preservation
Aluminum	flame	202.1	6 Months	500 ml P,G	HNO3 to pH < 2
	furnace	202.2	6 Months	500 ml P,G	HNO3 to pH < 2
	ICP	200.7	6 Months	500 ml P,G	HNO3 to pH < 2
	flame	7020	6 Months	500 ml P,G	HNO3 to pH < 2
	ICP	6010	6 Months	500 ml P,G	HNO3 to pH < 2
Antimony	flame	204.1	6 Months	500 ml P,G	HNO3 to pH < 2
	furnace	204.2	6 Months	500 ml P,G	HNO3 to pH < 2
	ICP	200.7	6 Months	500 ml P,G	HNO3 to pH < 2
	flame	7040	6 Months	500 ml P,G	HNO3 to pH < 2
	furnace	7041	6 Months	500 ml P,G	HNO3 to pH < 2
	ICP	6010	6 Months	500 ml P,G	HNO3 to pH < 2
Arsenic	furnace	206.2	6 Months	500 ml P,G	HNO3 to pH < 2
	AA, hydride	206.3	6 Months	500 ml P,G	HNO3 to pH < 2
	ICP	200.7	6 Months	500 ml P,G	HNO3 to pH < 2
	AA, hydride	7061	6 Months	500 ml P,G	HNO3 to pH < 2
	furnace	7060	6 Months	500 ml P,G	HNO3 to pH < 2
	ICP	6010	6 Months	500 ml P,G	HNO3 to pH < 2
Barium	flame	208.1	6 Months	500 ml P,G	HNO3 to pH < 2
	furnace	208.2	6 Months	500 ml P,G	HNO3 to pH < 2
	ICP	200.7	6 Months	500 ml P,G	HNO3 to pH < 2
	flame	7080	6 Months	500 ml P,G	HNO3 to pH < 2
	furnace	7081	6 Months	500 ml P,G	HNO3 to pH < 2
	ICP	6010	6 Months	500 ml P,G	HNO3 to pH < 2
Beryllium	flame	210.1	6 Months	500 ml P,G	HNO3 to pH < 2
	furnace	210.2	6 Months	500 ml P,G	HNO3 to pH < 2
	ICP	200.7	6 Months	500 ml P,G	HNO3 to pH < 2
	flame	7090	6 Months	500 ml P,G	HNO3 to pH < 2
	furnace	7091	6 Months	500 ml P,G	HNO3 to pH < 2
	ICP	6010	6 Months	500 ml P,G	HNO3 to pH < 2
Boron	colorimetric	212.3	6 Months	500 ml P,G	HNO3 to pH < 2
	ICP	6010	6 Months	500 ml P,G	HNO3 to pH < 2

Metals in Water-Continued...					
Parameter [1]	Technique	Method	Holding Time	Container	Preservation
Cadmium	flame	213.1	6 Months	500 ml P,G	HNO3 to pH<2
	furnace	213.2	6 Months	500 ml P,G	HNO3 to pH<2
	ICP	200.7	6 Months	500 ml P,G	HNO3 to pH<2
	flame	7130	6 Months	500 ml P,G	HNO3 to pH<2
	furnace	7131	6 Months	500 ml P,G	HNO3 to pH<2
	ICP	6010	6 Months	500 ml P,G	HNO3 to pH<2
Calcium	ICP	200.7	6 Months	500 ml P,G	HNO3 to pH<2
	flame	215.1	6 Months	500 ml P,G	HNO3 to pH<2
	titrimetric	215.2	6 Months	500 ml P,G	HNO3 to pH<2
	flame	7140	6 Months	500 ml P,G	HNO3 to pH<2
	ICP	6010	6 Months	500 ml P,G	HNO3 to pH<2
Chromium	flame	218.1	6 Months	500 ml P,G	HNO3 to pH<2
	furnace	218.2	6 Months	500 ml P,G	HNO3 to pH<2
	ICP	200.7	6 Months	500 ml P,G	HNO3 to pH<2
	flame	7190	6 Months	500 ml P,G	HNO3 to pH<2
	furnace	7191	6 Months	500 ml P,G	HNO3 to pH<2
	ICP	6010	6 Months	500 ml P,G	HNO3 to pH<2
Chromium Hexavalent	Coprecipitation	7195	24 Hours	500 ml P,G	Cool, 4 C.
	colorimetric	7196	24 Hours	500 ml P,G	Cool, 4 C.
	flame	7197	24 Hours	500 ml P,G	Cool, 4 C.
	DPP	7198	24 Hours	500 ml P,G	Cool, 4 C.
Cobalt	flame	219.1	6 Months	500 ml P,G	HNO3 to pH<2
	furnace	219.2	6 Months	500 ml P,G	HNO3 to pH<2
	ICP	200.7	6 Months	500 ml P,G	HNO3 to pH<2
	flame	7200	6 Months	500 ml P,G	HNO3 to pH<2
	furnace	7201	6 Months	500 ml P,G	HNO3 to pH<2
	ICP	6010	6 Months	500 ml P,G	HNO3 to pH<2

Metals in Water-Continued...					
Parameter [3]	Technique	Method	Holding Time	Container	Preservation
Copper	ICP	200.7	6 Months	500 ml P,G	HNO3 to pH < 2
	flame	7210	6 Months	500 ml P,G	HNO3 to pH < 2
	furnace	7211	6 Months	500 ml P,G	HNO3 to pH < 2
	ICP	6010	6 Months	500 ml P,G	HNO3 to pH < 2
Iron	flame	236.1	6 Months	500 ml P,G	HNO3 to pH < 2
	furnace	236.2	6 Months	500 ml P,G	HNO3 to pH < 2
	ICP	200.7	6 Months	500 ml P,G	HNO3 to pH < 2
	flame	7380	6 Months	500 ml P,G	HNO3 to pH < 2
	furnace	7381	6 Months	500 ml P,G	HNO3 to pH < 2
	ICP	6010	6 Months	500 ml P,G	HNO3 to pH < 2
Lead	flame	239.1	6 Months	500 ml P,G	HNO3 to pH < 2
	furnace	239.2	6 Months	500 ml P,G	HNO3 to pH < 2
	ICP	200.7	6 Months	500 ml P,G	HNO3 to pH < 2
	flame	7420	6 Months	500 ml P,G	HNO3 to pH < 2
	furnace	7421	6 Months	500 ml P,G	HNO3 to pH < 2
	ICP	6010	6 Months	500 ml P,G	HNO3 to pH < 2
Magnesium	flame	242.1	6 Months	500 ml P,G	HNO3 to pH < 2
	furnace	239.2	6 Months	500 ml P,G	HNO3 to pH < 2
	flame	7450	6 Months	500 ml P,G	HNO3 to pH < 2
	furnace	7421	6 Months	500 ml P,G	HNO3 to pH < 2
Manganese	flame	243.1	6 Months	500 ml P,G	HNO3 to pH < 2
	furnace	243.2	6 Months	500 ml P,G	HNO3 to pH < 2
	ICP	200.7	6 Months	500 ml P,G	HNO3 to pH < 2
	flame	7460	6 Months	500 ml P,G	HNO3 to pH < 2
	furnace	7461	6 Months	500 ml P,G	HNO3 to pH < 2
	ICP	6010	6 Months	500 ml P,G	HNO3 to pH < 2
Mercury	cold vapor-manual	245.1	28 Days	500 ml P,G	HNO3 to pH < 2
	cold vapor-automated	245.2	28 Days	500 ml P,G	HNO3 to pH < 2
	cold vapor-manual	7470	28 Days	500 ml P,G	HNO3 to pH < 2

Metals in Water-Continued...					
Parameter [5]	Technique	Method	Holding Time	Container	Preservation
Molybdenum	flame	246.1	6 Months	500 ml P,G	HNO3 to pH<2
	furnace	246.2	6 Months	500 ml P,G	HNO3 to pH<2
	ICP	200.7	6 Months	500 ml P,G	HNO3 to pH<2
	flame	7480	6 Months	500 ml P,G	HNO3 to pH<2
	furnace	7481	6 Months	500 ml P,G	HNO3 to pH<2
	ICP	6010	6 Months	500 ml P,G	HNO3 to pH<2
Nickel	flame	249.1	6 Months	500 ml P,G	HNO3 to pH<2
	furnace	249.2	6 Months	500 ml P,G	HNO3 to pH<2
	ICP	200.7	6 Months	500 ml P,G	HNO3 to pH<2
	flame	7520	6 Months	500 ml P,G	HNO3 to pH<2
	furnace	7521	6 Months	500 ml P,G	HNO3 to pH<2
	ICP	6010	6 Months	500 ml P,G	HNO3 to pH<2
Potassium	flame	258.1	6 Months	500 ml P,G	HNO3 to pH<2
	ICP	200.7	6 Months	500 ml P,G	HNO3 to pH<2
	flame	7610	6 Months	500 ml P,G	HNO3 to pH<2
	ICP	6010	6 Months	500 ml P,G	HNO3 to pH<2
Selenium	ICP	200.7	6 Months	500 ml P,G	HNO3 to pH<2
	furnace	270.2	6 Months	500 ml P,G	HNO3 to pH<2
	AA, hydride	270.3	6 Months	500 ml P,G	HNO3 to pH<2
	ICP	6010	6 Months	500 ml P,G	HNO3 to pH<2
	furnace	7740	6 Months	500 ml P,G	HNO3 to pH<2
	AA, hydride	7741	6 Months	500 ml P,G	HNO3 to pH<2
Silica	ICP	200.7	6 Months	500 ml P,G	HNO3 to pH<2
Silver	flame	272.1	6 Months	500 ml P,G	HNO3 to pH<2
	furnace	272.2	6 Months	500 ml P,G	HNO3 to pH<2
	ICP	200.7	6 Months	500 ml P,G	HNO3 to pH<2
	flame	7760	6 Months	500 ml P,G	HNO3 to pH<2
	furnace	7761	6 Months	500 ml P,G	HNO3 to pH<2
	ICP	6010	6 Months	500 ml P,G	HNO3 to pH<2

Metals in Water-Continued...					
Parameter [3]	Technique	Method	Holding Time	Container	Preservation
Sodium	flame	273.1	6 Months	500 ml P,G	HNO3 to pH < 2
	ICP	200.7	6 Months	500 ml P,G	HNO3 to pH < 2
	flame	7770	6 Months	500 ml P,G	HNO3 to pH < 2
	ICP	6010	6 Months	500 ml P,G	HNO3 to pH < 2
Thallium	flame	279.1	6 Months	500 ml P,G	HNO3 to pH < 2
	furnace	279.2	6 Months	500 ml P,G	HNO3 to pH < 2
	ICP	200.7	6 Months	500 ml P,G	HNO3 to pH < 2
	flame	7840	6 Months	500 ml P,G	HNO3 to pH < 2
	furnace	7841	6 Months	500 ml P,G	HNO3 to pH < 2
	ICP	6010	6 Months	500 ml P,G	HNO3 to pH < 2
Tin	flame	282.1	6 Months	500 ml P,G	HNO3 to pH < 2
	furnace	282.2	6 Months	500 ml P,G	HNO3 to pH < 2
	flame	7870	6 Months	500 ml P,G	HNO3 to pH < 2
Titanium	flame	283.1	6 Months	500 ml P,G	HNO3 to pH < 2
	furnace	283.2	6 Months	500 ml P,G	HNO3 to pH < 2
Vanadium	flame	286.1	6 Months	500 ml P,G	HNO3 to pH < 2
	furnace	286.2	6 Months	500 ml P,G	HNO3 to pH < 2
	ICP	200.7	6 Months	500 ml P,G	HNO3 to pH < 2
	flame	7910	6 Months	500 ml P,G	HNO3 to pH < 2
	furnace	7911	6 Months	500 ml P,G	HNO3 to pH < 2
	ICP	6010	6 Months	500 ml P,G	HNO3 to pH < 2
Zinc	flame	289.1	6 Months	500 ml P,G	HNO3 to pH < 2
	furnace	289.2	6 Months	500 ml P,G	HNO3 to pH < 2
	ICP	200.7	6 Months	500 ml P,G	HNO3 to pH < 2
	flame	7950	6 Months	500 ml P,G	HNO3 to pH < 2
	furnace	7951	6 Months	500 ml P,G	HNO3 to pH < 2
	ICP	6010	6 Months	500 ml P,G	HNO3 to pH < 2

Wet Chemistries in Water					
Parameter [1]	Technique	Method	Holding Time	Container	Preservation
Acidity	titrimetric	305.1	14 Days	100 ml P,G	Cool 4 C.
Alkalinity	titrimetric	310.1	14 Days	100 ml P,G	Cool 4 C.
Biochemical Oxygen Demand (BOD)	5 days, 20 C.	405.1	48 Hours	1000 ml P,G	Cool 4 C.
Bromide	titrimetric	320.1	28 Days	100 ml P,G	none required
Chemical Oxygen Demand (COD)	titrimetric, mid-level	410.1	28 Days	50 ml P,G	Cool 4 C, H2SO4 to pH <2
	titrimetric, low-level	410.2	28 Days	50 ml P,G	Cool 4 C, H2SO4 to pH <2
	titrimetric, high-level	410.3	28 Days	50 ml P,G	Cool 4 C, H2SO4 to pH <2
	automated-colorimetric	410.4	28 Days	50 ml P,G	Cool 4 C, H2SO4 to pH <2
Chloride	colorimetric	325.2	28 Days	50 ml P,G	none required
	colorimetric	9250	28 Days	50 ml P,G	none required
	titrimetric	9252	28 Days	50 ml P,G	none required
	colorimetric	9257	28 Days	50 ml P,G	none required
Cyanide	amenable to chlorine	335.1	14 Days [2]	500 ml P,G	Cool 4 C, NaOH to pH>12 Ascorbic Acid [1]
	spectrophotometric	335.2	14 Days [2]	500 ml P,G	Cool 4 C, NaOH to pH>12 Ascorbic Acid [1]
	Total, UV	335.3	14 Days [2]	500 ml P,G	Cool 4 C, NaOH to pH>12 Ascorbic Acid [1]
	colorimetric	9012	14 Days [2]	500 ml P,G	Cool 4 C, NaOH to pH>12 Ascorbic Acid [1]
Fluoride	distillation	340.1	28 Days	500 ml P,G	none required
	ion selective electrode	340.2	28 Days	500 ml P,G	none required
	colorimetric	340.3	28 Days	500 ml P,G	none required
Hardness, Total	colorimetric	130.1	6 Months	100 ml P,G	HNO3 to pH<2
	titrimetric	130.2	6 Months	100 ml P,G	HNO3 to pH<2
Iodide	titrimetric	345.1	24 Days	100 ml P,G	Cool 4 C.
Methylene Blue Active Substances	colorimetric	425.1	48 Hours	500 ml P,G	Cool 4 C.
Nitrogen Ammonia	colorimetric,phenate	350.1	28 Days	500 ml P,G	Cool 4 C, H2SO4 to pH <2
	distillation	350.2	28 Days	500 ml P,G	Cool 4 C, H2SO4 to pH <2
	ion selective electrode	350.3	28 Days	500 ml P,G	Cool 4 C, H2SO4 to pH <2

Wet Chemistries in Water-Continued...					
Parameter [1]	Technique	Method	Holding Time	Container	Preservation
Nitrogen-TKN	colorimetric,phenate	351.1	28 Days	500 ml P,G	Cool 4 C, H2SO4 to pH <2
	block digestor	351.2	28 Days	500 ml P,G	Cool 4 C, H2SO4 to pH <2
	colorimetric	351.3	28 Days	500 ml P,G	Cool 4 C, H2SO4 to pH <2
	ion selective electrode	351.4	28 Days	500 ml P,G	Cool 4 C, H2SO4 to pH <2
Nitrate	colorimetric, brucine	352.1	48 Hours	100 ml P,G	Cool 4 C.
	colorimetric, brucine	9200	48 Hours	100 ml P,G	Cool 4 C.
Nitrate-Nitrite	colorimetric, hydrazine	353.1	28 Days	100 ml P,G	Cool 4 C, H2SO4 to pH <2
	cadmium reduction, auto	353.2	28 Days	100 ml P,G	Cool 4 C, H2SO4 to pH <2
	cadmium reduction, manual	353.3	28 Days	100 ml P,G	Cool 4 C, H2SO4 to pH <2
Nitrite	spectrophotometric	354.1	48 Hours	100 ml P,G	Cool 4 C.
Oil & Grease, Total	gravimetric	413.1	28 Days	1000 ml G only	Cool 4 C, HCL or H2SO4 to pH <2
	IR	413.2	28 Days	1000 ml G only	Cool 4 C, HCL or H2SO4 to pH <2
	gravimetric	9070	28 Days	1000 ml G only	Cool 4 C, HCL or H2SO4 to pH <2
	gravimetric-sludge	9071	28 Days	1000 ml G only	Cool 4 C, HCL or H2SO4 to pH <2
Petroleum Hydrocarbons	IR	418.1	14 Days	1000 ml G only	Cool 4 C, HCL to pH <2
pH	electrode	150.1	in-field	50 ml P,G	not applicable
	electrode	9040	in-field	50 ml P,G	not applicable
	test paper	9041	in-field	50 ml P,G	not applicable
Phenolics, T-Recoverable	spectrophotometric	420.1	28 Days	500 ml G only	Cool 4 C, H2SO4 to pH <2
	colorimetric	420.2	28 Days	500 ml G only	Cool 4 C, H2SO4 to pH <2
	4AAP, Manual, Distillation	9065	28 Days	500 ml G only	Cool 4 C, H2SO4 to pH <2
	4AAP, Auto, Distillation	9066	28 Days	500 ml G only	Cool 4 C, H2SO4 to pH <2
	MBTH, Distillation	9067	28 Days	500 ml G only	Cool 4 C, H2SO4 to pH <2
Phosphorus, Ortho	colorimetric, auto	365.1	48 Hours	50 ml P,G	Filter immediately, Cool 4 C
	colorimetric, single	365.2	48 Hours	50 ml P,G	Filter immediately, Cool 4 C
	colorimetric-dual	365.3	48 Hours	50 ml P,G	Filter immediately, Cool 4 C
	total, auto, block digester	365.4	48 Hours	50 ml P,G	Filter immediately, Cool 4 C

Wet Chemistries in Water-Continued...					
Parameter [3]	Technique	Method	Holding Time	Container	Preservation
Phosphorus, Total	colorimetric, auto	365.1	28 Days	50 ml P,G	Cool 4 C, H2SO4 to pH <2
	colorimetric, single	365.2	48 Hours	50 ml P,G	Cool 4 C, H2SO4 to pH <2
	colorimetric-dual	365.3	48 Hours	50 ml P,G	Cool 4 C, H2SO4 to pH <2
	total, auto, block digester	365.4	48 Hours	50 ml P,G	Cool 4 C, H2SO4 to pH <2
Residue (Solids)	filterable (TDS)	160.1	7 Days	100 ml P,G	Cool 4 C.
	non-filterable (TSS)	160.2	7 Days	100 ml P,G	Cool 4 C.
	total (TS)	160.3	7 Days	100 ml P,G	Cool 4 C.
	volatile	160.4	7 Days	100 ml P,G	Cool 4 C.
	settleable	160.5	48 Hours	100 ml P,G	Cool 4 C.
Specific Conductance	meter	120.1	28 Days	100 ml P,G	Cool 4 C.
	meter	9050	28 Days	100 ml P,G	Cool 4 C.
Sulfate	ion chromatography	300.0	28 Days	50 ml P,G	Cool 4 C.
	colorimetric	375.1	28 Days	50 ml P,G	Cool 4 C.
	gravimetric	375.3	28 Days	50 ml P,G	Cool 4 C.
	turbidimetric	375.4	28 Days	50 ml P,G	Cool 4 C.
	colorimetric	9035	28 Days	50 ml P,G	Cool 4 C.
	colorimetric	9036	28 Days	50 ml P,G	Cool 4 C.
	turbidimetric	9038	28 Days	50 ml P,G	Cool 4 C.
Sulfide	titrimetric	376.1	7 Days	500 ml P,G	Cool 4 C, ZnAc/NaOH to pH>9
	colorimetric	376.2	7 Days	500 ml P,G	Cool 4 C, ZnAc/NaOH to pH>9
	colorimetric	9030	7 Days	500 ml P,G	Cool 4 C, ZnAc/NaOH to pH>9
Total Organic Carbon (TOC)	combustion or oxidation	415.1	28 Days	50 ml P,G	Cool 4 C, HCL or H2SO4 to pH <2
	combustion or oxidation	9060	28 Days	50 ml P,G	Cool 4 C, HCL or H2SO4 to pH <2
Total Organic Halides (TOX)	titrimetric	9020	28 Days	1000 ml G only [4] No Headspace	Cool 4 C, H2SO4 to pH <2 [5] Sodium Sulfite
Turbidity	nephelometric	180.1	48 Hours	100 ml P,G	Cool 4 C

Parameters by Gas Chromatography in Water					
Parameter [1]	Technique	Method	Holding Time	Container	Preservation
Halogenated Volatile Organics	gas chromatography	601	14 Days	3x40 ml vials	Cool 4 C.,Thiosulfate [6]
	gas chromatography	8010	14 Days	3x40 ml vials	Cool 4 C.,Thiosulfate [6]
Non-Halogenated Volatile Organics	gas chromatography	8015	14 Days	3x40 ml vials	Cool 4 C., HCL to pH < 2, Thiosulfate [6]
Purgeable Aromatics	gas chromatography	602	7/14 Days [7]	3x40 ml vials	Cool 4 C., HCL to pH < 2, Thiosulfate [6]
	gas chromatography	8020	7/14 Days [7]	3x40 ml vials	Cool 4 C., HCL to pH < 2, Thiosulfate [6]
Acrolein & Acrylonitrile	gas chromatography	603	14 Days	3x40 ml vials	Cool 4 C., HCL to pH 5, Thiosulfate [6]
	gas chromatography	8030	14 Days	3x40 ml vials	Cool 4 C., HCL to pH 5, Thiosulfate [6]
Phenols	gas chromatography	604	ext.-7 Days anal.-40 Days	1 L, Amber G	Cool 4 C.,Thiosulfate [6]
	gas chromatography	8040	ext.-7 Days anal.-40 Days	1 L, Amber G	Cool 4 C.,Thiosulfate [6]
Phthalate Esters	gas chromatography	606	ext.-7 Days anal.-40 Days	1 L, Amber G	Cool 4 C.,Thiosulfate [6]
	gas chromatography	8060	ext.-7 Days anal.-40 Days	1 L, Amber G	Cool 4 C.,Thiosulfate [6]
Nitrosamines	gas chromatography	607	ext.-7 Days anal.-40 Days	1 L, Amber G	Cool 4 C.,Thiosulfate [6]
Organochlorine Pesticides and PCB's	gas chromatography	608	ext.-7 Days anal.-40 Days	1 L, Amber G	Cool 4 C.,Thiosulfate [6]
	gas chromatography	8080	ext.-7 Days anal.-40 Days	1 L, Amber G	Cool 4 C.,Thiosulfate [6]
Polynuclear Aromatic Hydrocarbons (PNA's)	gas chromatography/LC	610	ext.-7 Days anal.-40 Days	1 L, Amber G	Cool 4 C.,Thiosulfate [6]
	gas chromatography	8100	ext.-7 Days anal.-40 Days	1 L, Amber G	Cool 4 C.,Thiosulfate [6]
	HPLC	8310	ext.-7 Days anal.-40 Days	1 L, Amber G	Cool 4 C.,Thiosulfate [6]

Parameters by Gas Chromatography in Water-Continued...					
Parameter [3]	Technique	Method	Holding Time	Container	Preservation
Haloethers	gas chromatography	611	ext.-7 Days anal.-40 Days	1 L, Amber G	Cool 4 C.,Thiosulfate [4]
Chlorinated Hydrocarbons	gas chromatography	612	ext.-7 Days anal.-40 Days	1 L, Amber G	Cool 4 C.,Thiosulfate [4]
	gas chromatography	8120	ext.-7 Days anal.-40 Days	1 L, Amber G	Cool 4 C.,Thiosulfate [4]
Organophosphorus Pesticides	gas chromatography	8140	ext.-7 Days anal.-40 Days	1 L, Amber G	Cool 4 C.,Thiosulfate [4]
Chlorinated Herbicides	gas chromatography	8150	ext.-7 Days anal.-40 Days	1 L, Amber G	Cool 4 C.,Thiosulfate [4]

Parameters by GC/MS in Water					
Parameter [1]	Technique	Method	Holding Time	Container	Preservation
Purgeables	GC/MS-624 list	624	7/14 Days [7]	3x40 ml vials	Cool 4 C., HCL to pH < 2, Thiosulfate [6]
	Priority Pollutant list	624	7/14 Days [7]	3x40 ml vials	Cool 4 C., HCL to pH < 2, Thiosulfate [6]
	Hazardous Substance list	624	7/14 Days [7]	3x40 ml vials	Cool 4 C., HCL to pH < 2, Thiosulfate [6]
	Target Compound list (TCL)	624	7/14 Days [7]	3x40 ml vials	Cool 4 C., HCL to pH < 2, Thiosulfate [6]
	Appendix IX list	624	7/14 Days [7]	3x40 ml vials	Cool 4 C., HCL to pH < 2, Thiosulfate [6]
	Priority Pollutant list	8240	7/14 Days [7]	3x40 ml vials	Cool 4 C., HCL to pH < 2, Thiosulfate [6]
	Hazardous Substance list	8240	7/14 Days [7]	3x40 ml vials	Cool 4 C., HCL to pH < 2, Thiosulfate [6]
	Target Compound list (TCL)	8240	7/14 Days [7]	3x40 ml vials	Cool 4 C., HCL to pH < 2, Thiosulfate [6]
	Appendix IX list	8240	7/14 Days [7]	3x40 ml vials	Cool 4 C., HCL to pH < 2, Thiosulfate [6]

Parameters by GC/MS in Water					
Parameter [3]	Technique	Method	Holding Time	Container	Preservation
Base-Neutral & Acid Extractables	625 list	625	ext.-7 Days anal.-40 Days	1 L, Amber G	Cool 4 C.,Thiosulfate [4]
	Priority Pollutant list	625	ext.-7 Days anal.-40 Days	1 L, Amber G	Cool 4 C.,Thiosulfate [4]
	Hazardous Substance list	625	ext.-7 Days anal.-40 Days	1 L, Amber G	Cool 4 C.,Thiosulfate [4]
	Target Compound list (TCL)	625	ext.-7 Days anal.-40 Days	1 L, Amber G	Cool 4 C.,Thiosulfate [4]
	Appendix IX list	625	ext.-7 Days anal.-40 Days	1 L, Amber G	Cool 4 C.,Thiosulfate [4]
	Priority Pollutant list	8250	ext.-7 Days anal.-40 Days	1 L, Amber G	Cool 4 C.,Thiosulfate [4]
	Hazardous Substance list	8250	ext.-7 Days anal.-40 Days	1 L, Amber G	Cool 4 C.,Thiosulfate [4]
	Target Compound list (TCL)	8250	ext.-7 Days anal.-40 Days	1 L, Amber G	Cool 4 C.,Thiosulfate [4]
	Appendix IX list	8250	ext.-7 Days anal.-40 Days	1 L, Amber G	Cool 4 C.,Thiosulfate [4]
	Priority Pollutant list	8270	ext.-7 Days anal.-40 Days	1 L, Amber G	Cool 4 C.,Thiosulfate [4]
	Hazardous Substance list	8270	ext.-7 Days anal.-40 Days	1 L, Amber G	Cool 4 C.,Thiosulfate [4]
	Target Compound list (TCL)	8270	ext.-7 Days anal.-40 Days	1 L, Amber G	Cool 4 C.,Thiosulfate [4]
	Appendix IX list	8270	ext.-7 Days anal.-40 Days	1 L, Amber G	Cool 4 C.,Thiosulfate [4]

Metals in Soil					
Parameter	Technique	Method	Holding Time	Container	Preservation
Aluminum	flame	7020	6 Months	100 g P,G	Cool 4 C.
	ICP	6010	6 Months	100 g P,G	Cool 4 C.
Antimony	flame	7040	6 Months	100 g P,G	Cool 4 C.
	furnace	7041	6 Months	100 g P,G	Cool 4 C.
	ICP	6010	6 Months	100 g P,G	Cool 4 C.
Arsenic	ICP	6010	6 Months	100 g P,G	Cool 4 C.
	furnace	7060	6 Months	100 g P,G	Cool 4 C.
	AA, hydride	7061	6 Months	100 g P,G	Cool 4 C.
Barium	flame	7080	6 Months	100 g P,G	Cool 4 C.
	furnace	7081	6 Months	100 g P,G	Cool 4 C.
	ICP	6010	6 Months	100 g P,G	Cool 4 C.
Beryllium	flame	7090	6 Months	100 g P,G	Cool 4 C.
	furnace	7091	6 Months	100 g P,G	Cool 4 C.
	ICP	6010	6 Months	100 g P,G	Cool 4 C.
Boron	ICP	6010	6 Months	100 g P,G	Cool 4 C.
Cadmium	flame	7130	6 Months	100 g P,G	Cool 4 C.
	furnace	7131	6 Months	100 g P,G	Cool 4 C.
	ICP	6010	6 Months	100 g P,G	Cool 4 C.
Calcium	flame	7140	6 Months	100 g P,G	Cool 4 C.
	ICP	6010	6 Months	100 g P,G	Cool 4 C.
Chromium	flame	7190	6 Months	100 g P,G	Cool 4 C.
	furnace	7191	6 Months	100 g P,G	Cool 4 C.
	ICP	6010	6 Months	100 g P,G	Cool 4 C.

Metals in Soil-Continued...					
Parameter [1]	Technique	Method	Holding Time	Container	Preservation
Cobalt	flame	7200	6 Months	100 g P,G	Cool 4 C.
	furnace	7201	6 Months	100 g P,G	Cool 4 C.
	ICP	6010	6 Months	100 g P,G	Cool 4 C.
Copper	flame	7210	6 Months	100 g P,G	Cool 4 C.
	furnace	7211	6 Months	100 g P,G	Cool 4 C.
	ICP	6010	6 Months	100 g P,G	Cool 4 C.
Iron	flame	7380	6 Months	100 g P,G	Cool 4 C.
	furnace	7381	6 Months	100 g P,G	Cool 4 C.
	ICP	6010	6 Months	100 g P,G	Cool 4 C.
Lead	flame	7420	6 Months	100 g P,G	Cool 4 C.
	furnace	7421	6 Months	100 g P,G	Cool 4 C.
	ICP	6010	6 Months	100 g P,G	Cool 4 C.
Magnesium	flame	7450	6 Months	100 g P,G	Cool 4 C.
	ICP	6010	6 Months	100 g P,G	Cool 4 C.
Manganese	flame	7460	6 Months	100 g P,G	Cool 4 C.
	furnace	7461	6 Months	100 g P,G	Cool 4 C.
	ICP	6010	6 Months	100 g P,G	Cool 4 C.
Mercury	cold vapor-manual	7470	28 Days	100 g P,G	Cool 4 C.
	cold vapor-manual	7471	28 Days	100 g P,G	Cool 4 C.
Molybdenum	flame	7480	6 Months	100 g P,G	Cool 4 C.
	furnace	7481	6 Months	100 g P,G	Cool 4 C.
	ICP	6010	6 Months	100 g P,G	Cool 4 C.
Nickel	flame	7520	6 Months	100 g P,G	Cool 4 C.
	furnace	7521	6 Months	100 g P,G	Cool 4 C.
	ICP	6010	6 Months	100 g P,G	Cool 4 C.
Potassium	flame	7610	6 Months	100 g P,G	Cool 4 C.
	ICP	6010	6 Months	100 g P,G	Cool 4 C.
Selenium	ICP	6010	6 Months	100 g P,G	Cool 4 C.
	furnace	7740	6 Months	100 g P,G	Cool 4 C.
	AA, hydride	7741	6 Months	100 g P,G	Cool 4 C.

Metals in Soil-Continued...					
Parameter [1]	Technique	Method	Holding Time	Container	Preservation
Silver	flame	7760	6 Months	100 g P,G	Cool 4 C.
	furnace	7761	6 Months	100 g P,G	Cool 4 C.
	ICP	6010	6 Months	100 g P,G	Cool 4 C.
Sodium	flame	7770	6 Months	100 g P,G	Cool 4 C.
	ICP	6010	6 Months	100 g P,G	Cool 4 C.
Thallium	flame	7840	6 Months	100 g P,G	Cool 4 C.
	furnace	7841	6 Months	100 g P,G	Cool 4 C.
	ICP	6010	6 Months	100 g P,G	Cool 4 C.
Tin	flame	7870	6 Months	100 g P,G	Cool 4 C.
Vanadium	flame	7910	6 Months	100 g P,G	Cool 4 C.
	furnace	7911	6 Months	100 g P,G	Cool 4 C.
	ICP	6010	6 Months	100 g P,G	Cool 4 C.
Zinc	flame	7950	6 Months	100 g P,G	Cool 4 C.
	furnace	7951	6 Months	100 g P,G	Cool 4 C.
	ICP	6010	6 Months	100 g P,G	Cool 4 C.

Wet Chemistries in Soil					
Parameter [1]	Technique	Method	Holding Time	Container	Preservation
Cyanide	spectrophotometric	9010	14 Days	100 g P,G	Cool 4 C.
	colorimetric	9012	14 Days	100 g P,G	Cool 4 C.
Sulfate	colorimetric	9035	28 Days	100 g P,G	Cool 4 C.
	colorimetric	9036	28 Days	100 g P,G	Cool 4 C.
	turbidimetric	9038	28 Days	100 g P,G	Cool 4 C.
Sulfide	colorimetric	9030		100 g P,G	Cool 4 C.

Parameters by Gas Chromatography in Soil					
Parameter [3]	Technique	Method	Holding Time	Container	Preservation
Halogenated Volatile Organics	gas chromatography	8010	14 Days	3x40 ml vials[5]	Cool 4 C.
Non-Halogenated Volatile Organics	gas chromatography	8015	14 Days	3x40 ml vials[5]	Cool 4 C.
Purgeable Aromatics	gas chromatography	8020	14 Days	3x40 ml vials[5]	Cool 4 C.
Acrolein & Acrylonitrile	gas chromatography	8030	14 Days	3x40 ml vials[5]	Cool 4 C.
Phenols	gas chromatography	8040	ext.-14 Days anal.-40 Days	100 g ,G	Cool 4 C.
Phthalate Esters	gas chromatography	8060	ext.-14 Days anal.-40 Days	100 g ,G	Cool 4 C.
Nitrosamines	gas chromatography	8070	ext.-14 Days anal.-40 Days	100 g ,G	Cool 4 C.
Organochlorine Pesticides and PCB's	gas chromatography	8080	ext.-14 Days anal.-40 Days	100 g ,G	Cool 4 C.
Polynuclear Aromatic Hydrocarbons (PNA's)	gas chromatography	8100	ext.-14 Days anal.-40 Days	100 g ,G	Cool 4 C.
	HPLC	8310	ext.-14 Days anal.-40 Days	100 g ,G	Cool 4 C.
Chlorinated Hydrocarbons	gas chromatography	8120	ext.-14 Days anal.-40 Days	100 g ,G	Cool 4 C.
Organophosphorus Pesticides	gas chromatography	8140	ext.-14 Days anal.-40 Days	100 g ,G	Cool 4 C.
Chlorinated Herbicides	gas chromatography	8150	ext.-14 Days anal.-40 Days	100 g ,G	Cool 4 C.

Parameters by GC/MS in Soil					
Parameter [1]	Technique	Method	Holding Time	Container	Preservation
Volatile organics	packed column	8240	14 Days	3x40 ml vials[6]	Cool 4 C.
	capillary column	8260	14 Days	3x40 ml vials[6]	Cool 4 C.
Base-Neutral & Acid Extractables	semi-vol packed	8250	ext.-14 Days anal.-40 Days	100 g ,G	Cool 4 C.
	semi-vol capillary	8270	ext.-14 Days anal.-40 Days	100 g ,G	Cool 4 C.

Footnotes

[1] If residual chlorine is present in the sample, 0.6 g of ascorbic acid is utilized. Ascorbic acid is only used if residual chlorine is present.

[2] Maximum holding time is 24 hours when sulfide is present. Optionally, all samples may be tested with lead acetate paper before pH adjustments in order to determine if sulfide is present. If sulfide is present, it can be removed by the addition of cadmium nitrate powder until a negative spot test is obtained. The sample is filtered and then NaOH is added to pH 12.

[3] The following information is based upon EPA requirements as outlined in Table II, Part 136, Title 40 of the Code of Federal Regulations, July 1991. This reference should be consulted if further clarification is desired. Various state agencies have differing requirements for both holding times and preservation from those listed above. In such cases, the local requirements supercede the EPA information.

[4] All samples should be collected in bottles with teflon septa and be protected from light. If this is not possible, use 250 ml bottles fitted with teflon lined caps. Samples should contain no headspace.

[5] If samples contain residual chlorine, it must be removed in the field by adding sulfite to the sample bottle (5 mg sodium sulfite crystals per liter of sample).

[6] If samples contain residual chlorine, 0.008% sodium thiosulfate must be added at the time of sampling and should only be used if residual chlorine is present.

[7] If samples do not receive pH adjustment, the holding time is 7 days. With pH adjustment, the holding time is 14 days.

[8] Alternatively, wide mouth glass jars designed for volatile samples may be utilized with teflon lined caps.

Attachment V

SAMPLE LOG-IN CHECKLIST FOR IEA-NORTH CAROLINA

Client _____

1.	Samples were received intact (ie. No Broken bottles, leaks, etc.)	Yes	No	N/A
2.	Was a discrepancy created due to temperature or pH deviation, bottle breakage or protocol expiration?	Yes	No	N/A
3.	Were custody seals intact?	Yes	No	N/A
4.	Was a Chain of Custody received, signed, and temperature noted?	Yes	No	N/A
5.	Does the number of bottles received agree with the COC?	Yes	No	N/A
6.	Were any transmittal forms or letters sent with the samples?	Yes	No	N/A
7.	If a letter was sent, does it match the COC?	Yes	No	N/A
8.	Does the Quote match the COC?	Yes	No	N/A
9.	Does the Quote contain a "Memo"?	Yes	No	N/A
10.	Was the memo printed and placed in the Project folder?	Yes	No	N/A
11.	Were shipping documents stapled to the Project folder?	Yes	No	N/A
12.	Do the parameters require a pH check or chlorine test? If so, who performed them? _____	Yes	No	N/A
13.	Were samples received in a chilled condition? (4 $\pm$ 2° C)	Yes	No	N/A
14.	Was the IEA Project number entered on the COC and were appropriate remark boxes checked on the COC?	Yes	No	N/A
15.	Were bottle labels verified to correspond with the client's ID's? Who verified the bottle labels as correct? _____	Yes	No	N/A
16.	Were INF, database, review and dry weight codes placed in the workorder?	Yes	No	N/A
17.	Have project due dates been checked?	Yes	No	N/A
18.	Was a copy of the project folder information given to the project manager?	Yes	No	N/A

The following signature indicates that the individual has performed the above checks during the login process.

Log-in Completed by: _____

Date of Log-In _____

Time of Log-In _____

Attachment VI

Sample Request/Delivery Form

Date:_____ Department Name:_____

#	Requestor	Project #	Sample #	Location	QC Level				Out	In	Matrix*
					I	II	III	CLP			
1											
2											
3											
4											
5											
6											
7											
8											
9											
10											
11											
12											
13											
14											
15											
16											
17											
18											
19											
20											

Matrix*: L = Leachate S = Soil W = Water O = Other

1.0 APPROVALS

The signature of the following individuals indicates that this SOP is complete and meets the requirements specified in corporate Document # QAS00200.NET.

Laboratory Director _____

Quality Assurance Manager _____

2.0 SCOPE AND APPLICATION

2.1 The objective of this document is to describe the procedure to be followed when subcontracting work to another laboratory.

2.2 Areas to be addressed include paperwork, packaging, and shipment of samples.

3.0 SAFETY

3.1 Safety glasses, labcoats, and vinyl gloves should always be worn when handling samples.

4.0 APPARATUS AND MATERIALS

4.1 All subcontracting work is to be written down on the Subcontracting Log (Attachment 1) at the time of shipment. Transpak coolers will be used to ship all samples to other laboratories.

5.0 PROCEDURE

5.1 The subcontracting process begins in Sample Management with the receipt of the Requisition For Network Order (Attachment 2). These forms will provide Sample Management with all of the information needed to correctly retrieve and ship the samples.

The following information must be provided:

1) IEA Project ID
2) Client Name
3) Sample Numbers to be subcontracted
4) Parameter(s) requested
5) Matrix of samples (Soil, water, extract, etc.)

6) Approval signatures by the QC Director and Lab Director
7) Subcontract laboratory

Take the project ID off of the forms and look up the project using the SAM system to determine the sample's current location and the project's due date. Once the samples have been retrieved from their current location, the next step is to fill out an IEA Chain of Custody form (Attachment 3). See the enclosed attachment for the correct method of completing this form.

5.2 Before the samples are delivered to the Transpak Area for Packaging, make a copy of the Requisition For Network Order and Request To Subcontract Analysis and staple them to a copy of the IEA Chain of Custody. Give the copies to the Department Manager and be sure to place another copy of the Chain of Custody in the Project Folder. The original copies and all samples should then be taken to the Transpak Area for packing and shipment.

5.3 When packaging samples for shipment there are a two rules that must be followed. The first : all volatile samples must be sent in their own Transpak with a trip blank. A trip blank should be included for each method involved. For example, a trip blank for 602 in water would consist of 3x40ml vials of HCL preserved volatile-free water. Trip blanks for 601 in water would consist of 3x40 ml vials of unpreserved volatile-free water. If a combination of unpreserved and preserved methods are ordered, for example 601/602, then only 2 vials per method can be used (2 preserved and 2 unpreserved vials of volatile-free water). When preparing the blanks have a GC/MS operator fill out the instrument ID and data filename on the Blank Request Form (Attachment 4). This information should be written on the Chain of Custody and on the Subcontract Log.

5.4 The Transpak Area will then follow the above rules for packaging subcontracted samples and then prepare the shipment for Federal Express delivery. Any special delivery requirements, like Saturday delivery, should be noted on the Requisition For Network Order.

6.0 SUPPLEMENTAL DOCUMENTS

7.0 REFERENCES

8.0 SUBSTANTIVE REVISIONS

Attachment 1

Week ending ____________

IEA

SUB-CONTRACT LOG

DATE SHIPPED	PROJECT NO.	CUSTOMER NAME	SHIPPED TO:

Attachment 2

Requisition For Network Order

.trix	IEA Sample ID	Parameter/Method	Detect. Limits	Reptg. Level	Report due date	State Cert.	Unit Price

Workorder No.:_____

B. MEMO written? Yes No Please attach if applicable

C. Are samples currently in house? Yes No If not, arrival date in house:_____

D. Rush Order? Yes No **E.** Client contacted? Yes No

F. Date protocol holding time expires:_____

G. Samples leached or extracted? Yes No If yes, please attach appropriate paperwork from lab

H. Sample arrival date to network/subcontract lab:_____

I. Date results needed from network/subcontract lab:_____

J. Did you check for internal execution with:

CT NJ IL FL MA NC VT Other_____ Yes No

(1) If no, "Why?" _____

(2) If "other," is the contractor approved by IEA? Yes No

_____ _____

Requested By Date/Location Lab Manager Authorization Date/Location

Attachment 3

IEA
An Aquarion Company
3000 WESTON PKWY.
CARY, N.C. 27513
PH # 919-677-0090
FAX # 919-677-0427

CHAIN OF C' TODY RECORD

NO: 00654

REGULATORY CLASSIFICATION - PLEASE SPECIFY

☐ NPDES ☐ DRINKING WATER ☐ RCRA ☐ OTHER _____

COMPANY

Page _____ of _____

PROJECT # | PROJECT NAME

SAMPLERS: *(SIGNATURE)*

SAMPLE I.D.	DATE	TIME	# OF CONTAINERS	MATRIX: SOIL	MATRIX: WATER	STATION LOCATION	REQUESTED PARAMETERS

5C COMP ☐ GRAB ☐

SUBCONTRACT

REPORT DELIVERABLE REQUEST

- ☐ BLANK DATA
- ☐ DUPLICATE DATA / % RPD
- ☐ SURROGATE / % RECOVERY
- ☐ MS/MSD SUMMARY WITH % RECOVERY, % RPD
- ☐ RETURN SIGNED COC
- ☐ ANALYSTS' FULL NAME
- ☐ DATE ANALYZED
- ☐ DATE EXTRACTED
- ☐ CLP PACKAGE
- ☐ SPECIAL ANALYTICAL REQUEST

RELINQUISHED BY *(SIGNATURE)*	DATE	TIME	RECEIVED BY	DATE	TIME
RELINQUISHED BY *(SIGNATURE)*	DATE	TIME	RECEIVED FOR LAB BY	DATE	TIME

IEA QUOTE NO.	IEA RUSH NO.
PROJECT MANAGER (PLEASE PRINT)	**P.O. NO.**

REMARKS ON SAMPLE RECEIPT	IEA REMARKS	FIELD REMARKS
☐ BOTTLE INTACT ☐ CUSTODY SEALS ☐ PRESERVED ☐ SEALS INTACT ☐ CHILLED ☐ SEE REMARKS		

Attachment 4

BLANK REQUEST FORM

NUMBER OF VIALS PRESERVED TIME NEEDED

REQUESTED BY PREPARED BY

INSTRUMENT ID DATA FILENAME

BLANK REQUEST FORM

NUMER OF VIALS PRESERVED TIME NEEDED

REQUESTED BY PREPARED BY

INSTRUMENT ID DATA FILENAME

5. IEA SOP FOR COMMERCIAL SOLVENT APPROVAL

1.0 APPROVALS

The signature of the following individual indicates that this SOP is complete and meets the requirements specified in corporate Document # QAS00200.NET.

Corporate QA Director James A. Pleiss

UNCONTROLLED COPY
PROPRIETARY DOCUMENT

2.0 SCOPE AND APPLICATION

2.1 As part of the company's Quality Assurance program, all commonly used solvents and chemical reagents that are used in sufficient volume are centrally checked and approved prior to use at any Network laboratory. All items monitored under this program are listed in section 5.1 of this document. All laboratories are required to use approved lots of these materials for each of the applications listed in this table. The solvents/chemical reagents covered under this program are concentrated (where applicable) and tested for the presence of interfering substances relative to their intended use. If the particular lot of solvents or chemical reagent passes the defined acceptance criteria, the vendor is notified and the lot is reserved for use by the entire corporation. The approved lot numbers are provided to all laboratories and only approved solvents and chemicals can be employed. As a clarification, this solvent/chemical reagent checking program does not obviate the need for each laboratory to perform batch method blanks during daily operations. This program functions as a preventative measure to ensure consistent quality is being provided by our vendors. This SOP describes details of the solvent/chemical reagent monitoring process.

2.2 This SOP addresses the following issues:

What solvents and chemicals are included in the program?
Who checks these materials?
How are the materials checked?
How are all labs notified of approved lots?
How are these materials ordered through the vendor?
Who monitors solvent/chemical reagent quantities to ensure continued availability?

3.0 SAFETY

3.1 Various solvents and chemicals used in the laboratory can be hazardous if handled improperly. All solvents and chemical reagents should be handled in a safe manner as specified in the routine SOP's for processing of samples. Human exposure to solvent vapors should be minimized wherever possible. Consult the Material Safety Data Sheet (MSDS) for the solvent or reagent prior to use.

4.0 APPARATUS AND MATERIALS

4.1 No unusual equipment is required other than that needed for solvent extraction of routine samples with the exception that a logbook is maintained to document the testing and approval or rejection of the solvent.

5.0 PROCEDURE

5.1 The solvents and chemical reagents that are monitored as part of the corporate program are listed below along with their intended analytical uses:

Solvent/Chemical	Approved Analytical Use	Suitable for Use with Methods	QC Lab
Acetone-P	SV. and Pest. in Soils by GC+GC/MS	CLP,8080,8270	NC
Methylene Chloride-P	SV. and Pest. in Water+Soils by GC+GC/MS	CLP,608,8080,625,8270	NC
Hexane-P	Pesticides in Water+Soils by GC	CLP,608,8080	NC
Methanol-P+T	Volatiles in Water+Soils by GC+GC/MS	601,602,8010,8020, CLP,624,8240	NC
Freon 1-R	O+G-Grav/IR,TPH-IR	413.1,413.2,418.1 9070,9071	NC
Freon 2-P	TPH-GC/IR,O+G-Grav/IR	413.2,418.1	NC
Sodium Sulfate-A (Granular 60-80 Mesh) (Anhydrous)	Drying Reagent for Solvent Extracts	Used for CLP Pest Soils	NC
Sodium Sulfate-B (Powdered, Anhydrous)	Drying Reagent for Solvent Extracts	General Purpose	NC

In the above table, P = Pesticide grade, P+T = Purge and Trap, A = Anhydrous, R = Reagent grade.

5.2 The laboratory responsible for performing the quality control testing procedure on the solvents is listed above. Regardless of location performing the QC tests, all supporting documentation is provided to the corporate QA Director and is kept on file.

Once a lot of a particular solvent is approved, this information is passed on to all the network laboratories via fax from the QA Director to the various QA Managers at each facility utilizing the form presented in Figure 2. The local QA Managers are responsible for maintaining communication with operational personnel relative to approved solvent lots.

Lots of approved solvents are maintained in the vendors warehouse facilities and shipped directly to the laboratory ordering the solvent. It is the responsibility of the laboratory QA Manager to ensure that only approved solvents are employed at their respective locations.

5.3 **Approval Criteria for each Solvent/Chemical Reagent**

The following represent the criteria used to determine if a variety of solvents or chemical reagents are of suitable purity for use in numerous laboratory applications. All data, regardless of whether the material was accepted or rejected are to be provided to corporate QA for maintenance of permanent records. In most cases, a blank is prepared utilizing approved solvent and is processed along with duplicate aliquots of the material being tested. Acceptance criteria is based on the duplicate which indicates the minimum amount of contaminants present. In order to minimize the opportunity for contamination potential, surrogates are not added to any of the solvents or blanks being processed.

5.3.1 **Acetone-P**

Acetone is utilized in both the GC/MS and GC areas, each with specific acceptance criteria. In order to be approved, the acetone must meet both sets of criteria.

GC/MS Acceptance

Surrogate compounds are not added to any of the aliquots being tested.

A solvent blank is prepared utilizing previously approved solvent and is processed along with the following duplicate analyses of the solvent being tested.

Two aliquots of 250 mls each are concentrated to an approximate volume of 1 milliliter. Fifty (50) mls of methylene chloride (from a previously approved lot) are added and the mixture is further evaporated to a final volume of exactly 1.0 mls.

The resulting extracts are then analyzed on a GC/MS utilizing a DB-5 capillary column or equivalent. The entire GC/MS system must be demonstrated to be free of interferences prior to running the solvent QC test. In order to be acceptable there must be no peaks

responding between 30 seconds prior to the elution of 2-Fluorophenol and 30 seconds after the elution of Benzo-(ghi)-perylene with an area greater than 10% of the response the nearest internal standard at a concentration of 20 ng/ul. Any compounds that elute earlier than 30 seconds prior to 2-fluorophenol will be evaluated on a case-by-case basis due to the nature of Aldol condensation by-products associated with acetone/methylene chloride extractions.

The solvent aliquot containing the least amount of contaminants is utilized to determine if the acceptance criteria have been achieved.

GC Acceptance

Surrogate compounds are not added to any of the aliquots being tested.

A solvent blank is prepared utilizing previously approved solvent and is processed along with the following duplicate analyses of the solvent being tested.

A sample of the acetone is prepared in duplicate according to the following procedure.

Two aliquots of 250 mls each are concentrated to an approximate volume of 1 milliliter. Fifty (50) mls of hexane (from a previously approved lot) are added and the mixture is further evaporated to a final volume of exactly 10.0 mls.

The resulting extracts are then analyzed on a GC with an electron capture detector utilizing a DB-1701, DB-608, or equivalent megabore capillary column. The entire GC system must be demonstrated to be free of interferences prior to running the solvent QC test. In order to be acceptable there must be no peaks responding between the elution of the first major component peak of Aroclor-1221 (which elutes before tetrachloro-m-xylene (TCX) and decachlorobiphenyl (DCB) with an area of greater than 25% of the response of TCX @ 5 ng/mL. A standard containing Aroclor-1221 @ 0.20 ug/mL (200 ng/mL), TCX @ 5 ng/mL, and DCB @ 5 ng/mL is analyzed with all solvent approval tests.

The solvent aliquot containing the least amount of contaminants is utilized to determine if the acceptance criteria have been achieved.

5.3.2 Methylene Chloride-P

Methylene chloride is utilized in both the GC/MS and GC areas, each with specific acceptance criteria. In order to be approved, the methylene chloride must meet both sets of criteria.

5.3.2.1 GC/MS Acceptance

5.3.2.1.1 Surrogate compounds are not added to any of the aliquots being tested.

5.3.2.1.2 A solvent blank is prepared utilizing previously approved solvent and is processed along with the following duplicate analyses of the solvent being tested.

5.3.2.1.3 A sample of the methylene chloride is prepared in duplicate according to the following procedure.

5.3.2.1.4 Two aliquots of 250 mls each are concentrated to a final volume of exactly 1.0 mls.

5.3.2.1.5 The resulting extracts are then analyzed on a GC/MS utilizing a DB-5 capillary column or equivalent. The entire GC/MS system must be demonstrated to be free of interferences prior to running the solvent QC test.In order to be acceptable there must be no peaks responding between the elution of N-nitrosodimethylamine and Benzo (ghi) perylene with an area greater than 10% of the response of the nearest internal standard at a concentration of 20 ng/ul.

5.3.2.1.6 The solvent aliquot containing the least amount of contaminants is utilized to determine if the acceptance criteria have been achieved.

5.3.2.2 GC Acceptance

5.3.2.2.1 Surrogate compounds are not added to any of the aliquots being tested.

5.3.2.2.2 A solvent blank is prepared utilizing previously approved solvent and is processed along with the following duplicate analyses of the solvent being tested.

5.3.2.2.3 A sample of the methylene chloride is prepared in duplicate according to the following procedure and the mean result must pass the acceptance criteria.

5.3.2.2.4 Two aliquots of 250 mls each are concentrated to an approximate volume of 1 milliliter. Fifty (50) mls of hexane (from a previously approved lot) are added and the mixture is further evaporated to a final volume of exactly 10.0 mls.

5.3.2.2.5 The resulting extracts are then analyzed on a GC with an electron capture detector utilizing a DB-1701, DB-608, or equivalent megabore capillary column. The entire GC system must be demonstrated to be free of interferences prior to running the solvent QC test. In order to be acceptable there must be no peaks responding between the elution of the first major component peak of Aroclor-1221 (which elutes before tetrachloro-m-xylene (TCX) and decachlorobiphenyl (DCB) with an area of greater than 25% of the response of TCX @ 5 ng/mL. A standard containing Aroclor-1221 @ 0.20 ug/mL (200 ng/mL), TCX @ 5 ng/mL, and DCB @ 5 ng/mL is analyzed with all solvent approval tests.

5.3.2.2.6 The solvent aliquot containing the least amount of contaminants is utilized to determine if the acceptance criteria have been achieved.

5.3.3 Hexane-P

Hexane is utilized in the GC area for solvent exchanges. In order to be approved, the hexane must meet the following criteria.

GC Acceptance

Surrogate compounds are not added to any of the aliquots being tested.

A solvent blank is prepared utilizing previously approved solvent and is processed along with the following duplicate analyses of the solvent being tested.

A sample of the hexane is prepared in duplicate according to the following procedure and the mean result must pass the acceptance criteria.

Two aliquots of 250 mls each are concentrated to a final volume of exactly 10.0 mls.

The resulting extracts are then analyzed on a GC with an electron capture detector utilizing a DB-1701, DB-608, or equivalent megabore capillary column. The entire GC system must be demonstrated to be free of interferences prior to running the solvent QC test. In order to be acceptable there must be no peaks responding between the elution of the first major component peak of Aroclor-1221 (which elutes before tetrachloro-m-xylene (TCX) and decachlorobiphenyl (DCB) with an area of greater than 25% of the response of TCX @ 5 ng/mL. A standard containing Aroclor-1221 @ 0.20 ug/mL (200 ng/mL), TCX @ 5 ng/mL, and DCB @ 5 ng/mL is analyzed with all solvent approval tests.

The solvent aliquot containing the least amount of contaminants is utilized to determine if the acceptance criteria have been achieved.

5.3.4 Methanol (Purge and Trap)

Methanol is utilized in both the GC/MS and GC areas for volatile compound analyses therefore it must meet both requirements to become approved.

GC Acceptance

A solvent blank is prepared utilizing previously approved solvent and is processed along with the following analyses of the solvent being tested.

A 100 ul aliquot of Purge and Trap grade methanol, for GC analysis, is added to 5 mls of reagent water in a purge and trap vessel known to be free of contamination. Internal standards and surrogate standards are also added to the purge vessel. The entire GC system must be demonstrated to be free of interferences prior to running the solvent QC test.

The QC sample is then analyzed on the GC utilizing a Rtx-Volatiles column or equivalent. In order to be acceptable for 8010 and 601 analyses there must be no peaks responding between the elution of Dichlorodifluoromethane and 1,2-Dichlorobenzene that quantitate to more than 1/2 the Practical Quantitation Limit (PQL), which is 1.0 ug/L.

In order for the solvent to be approved for 8020 and 602 analyses, there must be no peaks present between the elution of MTBE and 1,2-Dichlorobenzene that quantitate to more than 1/2 the PQL, which is 1.0 ug/L.

If a single determination does not yield the solvent as acceptable, a second analysis must be conducted to verify the solvent is unacceptable. If this analysis is still unsuitable, the solvent is rejected.

GC/MS Acceptance

A solvent blank is prepared utilizing previously approved solvent and is processed along with the following analyses of the solvent being tested.

A 200 ul aliquot of Purge and Trap grade methanol is added to 5 mls of reagent water in a purge and trap vessel known to be free of contamination. Internal standards are also added to the purge vessel. The entire GC/MS system must be demonstrated to be free of interferences prior to running the solvent QC test.

The QC sample is then analyzed on the GC/MS utilizing a DB-624 capillary column or equivalent. In order to be acceptable there must be no peaks responding between the elution of Chloromethane and 1,2-Dichlorobenzene with an area greater than 10% of the response of the nearest internal standard at a concentration of 50 ug/L.

If a single determination does not yield the solvent as acceptable, a second analysis must be conducted to verify the solvent is unacceptable. If this analysis is still unsuitable, the solvent is rejected.

5.3.5 **Ethyl Ether (Anhydrous)** Procedure "Reserved"-not currently part of Corp. Program.

Surrogate compounds are not added to any of the aliquots being tested.

A solvent blank is prepared utilizing previously approved solvent and is processed along with the following duplicate analyses of the solvent being tested.

A sample of ethyl ether is prepared in duplicate according to the following procedure.

Two aliquots of 250 mls each are concentrated to a final volume of approximately 0.5 mls and 0.1 mls of methanol (from an approved lot) are added.

Note: (Diazomethane is a carcinogen and can explode under certain conditions) See the actual analytical method for further safety precautions.

The samples are derivatized by adding 2.0 ml of diazomethane solution. Then 5.0 mls of hexane (from an approved lot) is added to dissolve any residue.

The resulting extracts are then analyzed on a GC with an electron capture detector utilizing a DB-1701, DB-608, or equivalent megabore capillary column. The entire GC system must be demonstrated to be free of interferences prior to running the solvent QC test. In order to be acceptable there must be no peaks responding between the elution of the first major component peak of Aroclor-1221 (which elutes before tetrachloro-m-xylene (TCX) and decachlorobiphenyl (DCB) with an area of greater than 25% of the response of TCX @ 5 ng/mL. A standard containing Aroclor-1221 @ 0.20 ug/mL (200 ng/mL), TCX @ 5 ng/mL, and DCB @ 5 ng/mL is analyzed with all solvent approval tests.

The solvent aliquot containing the least amount of contaminants is utilized to determine if the acceptance criteria have been achieved.

5.3.6 **Freon 1**

This solvent is utilized for both IR, and Gravimetric applications therefore it must pass both acceptance criteria to become approved.

Gravimetric Approval

A sample of the freon is prepared in duplicate according to the following procedure and the mean result must pass the acceptance criteria.

Two aliquots of 200 mls each are evaporated according to the procedure described in EPA method 413.1 except that the final concentration is performed in 40 ml volatile vials which have been engraved for identification purposes. The use of these vials allows for more accurate weighings to be accomplished. Only the pure solvent is evaporated, blank

water or other reagents are not utilized since they could contribute residue causing the solvent to be improperly rejected.

The average of the two determinations must not exceed five (5) milligrams of residue/200 mls of original solvent. This procedure takes into account the possibility of 500 mls of sample being extracted rather than the 1000 mls recommended.

Infrared (IR) Approval

A one hundred (100) millimeter IR cell is filled with the solvent to be tested and the IR analysis is conducted according to the procedure in EPA method 418.1. The absorbance of the solvent is measured at the absorbance maximum at about wavelength 2930 cm^{-1}.

The absorbance of the solvent must be less than the absorbance of a known level (0.04 ug/ml) of working standard. The standard consists of the EPA reference oil listed in section 6.5.1 of EPA method 418.1. A 0.4 ug/l standard is diluted 1:10 with freon from an approved lot.

If the single determination does not yield the solvent as acceptable, the cell should be thoroughly cleaned and rinsed and a second aliquot of solvent taken for analysis. If this analysis is still unsuitable, the solvent is rejected.

5.3.7 **Freon 2**

This solvent is utilized for both GC, IR, and Gravimetric applications therefore it must pass all three acceptance criteria to become approved.

Gravimetric Approval

The solvent must pass the QC test described in section 5.3.6 to be approved for use in gravimetric procedures.

Infrared (IR) Approval

A one hundred (100) millimeter IR cell is filled with the solvent to be tested and the IR analysis is conducted according to the procedure in EPA method 418.1. The absorbance of the solvent is measured at the absorbance maximum at about wavelength 2930 cm^{-1}.

The absorbance of the solvent must be less than the absorbance of a known level (0.04 ug/ml) of working standard. The standard consists of the EPA reference oil listed in section 6.5.1 of EPA method 418.1. A 0.4 ug/l standard is diluted 1:10 with freon from an approved lot.

If the single determination does not yield the solvent as acceptable, the cell should be thoroughly cleaned and rinsed and a second aliquot of solvent taken for analysis. If this analysis is still unsuitable, the solvent is rejected.

Gas Chromatography (GC) Approval

A sample of freon is prepared in duplicate according to the following procedure and the mean result must pass the acceptance criteria.

A solvent blank is prepared utilizing previously approved solvent and is processed along with the following duplicate analyses of the solvent being tested.

Two aliquots of 250 mls each are concentrated to a final volume of 1.0 mls.

The resulting extracts are then analyzed on a GC with a flame ionization detector utilizing a RTx-5 or equivalent chromatography column. The entire GC system must be demonstrated to be free of interferences prior to running the solvent QC test.In order to be acceptable there must be no peaks responding between the elution of Toluene and Dodecane with an area greater than 10% of the response of a Naphthalene standard at a concentration of 10 ug/ml.

The solvent aliquot containing the least amount of contaminants is utilized to determine if the acceptance criteria have been achieved.

5.3.8 Sodium Sulfate

The following procedure is utilized to evaluate both granular and powdered types of sodium sulfate. All sodium sulfate must pass both GC and GC/MS acceptance criteria to be approved.

Extraction of Sodium Sulfate

There is no pretreatment of the sodium sulfate prior to evaluation and surrogate compounds are not added to any of the aliquots being tested.

A solvent blank (methylene chloride/acetone) is prepared utilizing previously approved solvent and is processed along with the sodium sulfate being tested.

A sample of the sodium sulfate is prepared in duplicate according to the following procedure.

60 grams of sodium sulfate are placed into a beaker with 100 mls of a 50/50 mixture of methylene chloride and acetone (previously approved solvents). The sodium sulfate is extracted for 90 seconds using a sonicator unit utilizing standard CLP settings. The extraction is performed a total of 3 times and each time fresh solvent is added to the beaker. The approximately 300 mls of extraction solvent is collected in a kuderna-danish concentrator vessel and the solvent volume is reduced to 1.0 ml. on a steam bath. The resulting extract must be evaluated for both GC and GC/MS acceptability.

For GC/MS acceptability the extraction solvent is concentrated to 1.0 ml and the extract is analyzed according to the procedure presented in section 5.3.2.1.5 of this document.

For GC acceptability, the extraction solvent is exchanged into 50 mls of hexane (approved lot) and then concentrated to 1.0 ml. This extract is then subjected to the analytical procedure specified in section 5.3.2.2.5 of this document.

5.4 How to Order Approved Materials?

There is one individual designated by our vendor as the program coordinator for administering the program between our organizations. This individual will communicate directly with IEA's Corporate QA Director in order to administer the program. Once a solvent or chemical reagent is approved it will receive a product designation (for example, IEA D151-4A). Each laboratory is to contact their local sales representative and request the indicated product designation. The sales representative will then place that order through their local branch and deduct that solvent from the standing inventory.

The program coordinator for the vendor will keep track of the amount of all solvents available to the network and when the level drops below a pre-agreed upon level, corporate QA is contacted to make arrangements for testing and approval of additional lots.

The goal is to minimize the frequency of testing. It is preferable to test and approve 6 lots of a given solventor chemical reagent semi-annually rather than 1 lot per month.

6.0	SUPPLEMENTAL DOCUMENTS	None
7.0	REFERENCES	None
8.0	SUBSTANTIVE REVISIONS	Sodium Sulfate added in 1994 revision.

Figure 1

Solvent Approval Procedure

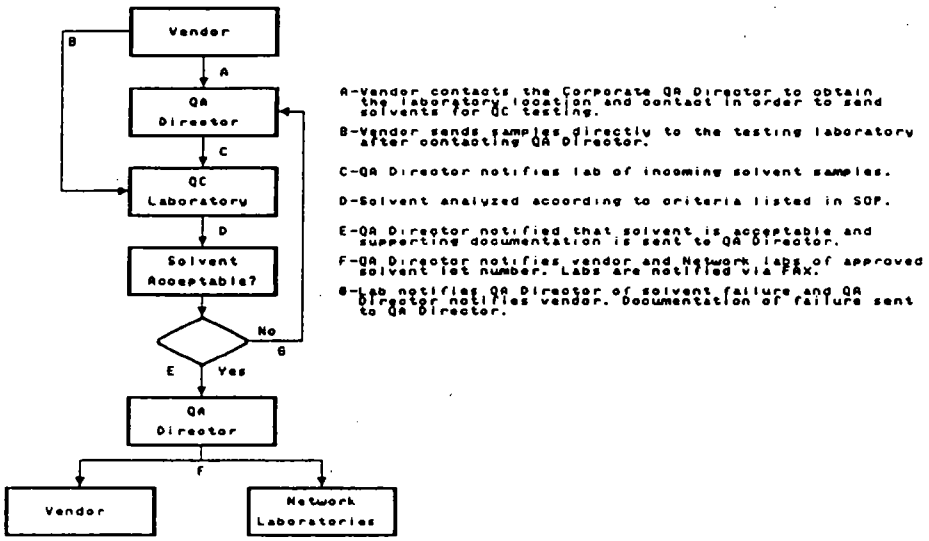


Figure 2

Solvent Approval Notification Form

Solvent	Approved Analytical Use	Suitable for Use with Methods	Vendor	Approved Lot# and Order Number
Acetone-P	SV. and Pest. in Soils by GC+GC/MS	CLP,8080,8270	Baxter (B+J) Baxter (B+J) Baxter (B+J) Baxter (B+J) Baxter (B+J)	9/9/92-BD461 11/24/92-BE125 4/22/93-BF002 8/3/93-BF775 10/26/93-BG131
Methylene Chloride-P	SV. and Pest. in Water+Soils by GC+GC/MS	CLP,608,8080,625, 8270	Baxter (B+J) " " " " " " " " "	7/27/92-BD197(2) 9/2/92-BD381 9/2/92-BD388 12/17/92-BE250 12/17/92-BE223 2/22/93-925806 7/21/93-BF656 7/21/93-BF728 12/9/93-BG472 12/13/93-BG485
Hexane-P	Pesticides in Water+Soils by GC	CLP,608,8080	Baxter (B+J) "	2/22/93-BE574 7/21/93-BF752 1/7/94-BG656
Methanol-P+T	Volatiles in Water+Soils by GC+GC/MS	601,602,8010,8020, CLP,624,8240	Baxter (B+J)	9/14/92-BD415
Freon 1-P	O+G-Grav/IR	413.1,413.2,418.1	DuPont	1/20/94-105-3M
Freon 2-P	TPH-GC/IR,O+G-Grav/IR	413.1,413.2,418.1 9070,9071	Baxter " "	9/14/92-BD454 2/18/93-BE599 9/9/93-BF094
Sodium Sulfate-A (Granular)	Drying Reagent for Solvent Extracts Anhydrous	Required for CLP Pest. Soils	Baxter	?
Sodium Sulfate-B (Powdered)	Drying Reagent for Solvent Extracts Anhydrous	General Purpose	Baxter	?

In the above table, P = Pesticide grade, P+T = Purge and Trap, A = Anhydrous, R = Reagent grade.

Figure 3

Request for QC Analysis

Date: _____

Solvent/Chemical: _____

Lot Number: _____

Please test this material to meet specifications in the "Corporate SOP for Solvent Approval (QAS00401.NET). Please Note! This is a new SOP which replaces the previous procedure.

The above material must be evaluated for:

- ❑ GC
- ❑ GC/MS
- ❑ Wet Chemistry

__ GC Approved Signature _____ Date: _____

__ GC/MS Approved Signature _____ Date: _____

__ GC Rejected Signature _____ Date: _____

__ GC/MS Rejected Signature _____ Date: _____

Attach all supporting data including chromatograms and submit to QA Director.

Final Approval (Corporate QA Director)

Signature _____ Date: _____

6. IEA SOP FOR SW-846 8260A VOCs ANALYSIS

1.0 APPROVALS

The signatures of the following individuals indicate that this SOP is complete and meets the requirements specified in corporate document # QAS00200.NET. In addition, it signifies that the content meets the specifications of the referenced "Test Code".

Laboratory Director

Quality Assurance Manager

2.0 SCOPE AND APPLICATION

2.1 The objective of this document is to outline the techniques for determining the presence and concentration of various volatile organic target and non-target compounds in multi-media, multi-concentration samples. The target compounds for this method are listed in Table 1.0.

2.2 The document control number for this SOP is MSS01201.NC.

2.3 This SOP is applicable to test codes: MSV_36 and MSV_37.

3.0 SUMMARY OF METHOD

3.1 This method employs the technique of purge and trap, coupled with a gas chromatograph/mass spectrometer. An aliquot of sample is purged in a gas tight chamber with UHP grade helium to extract the volatile compounds. The vapor is swept onto a sorbent trap where the volatiles are adsorbed. Next, the sorbent trap is heated and backflushed, thereby desorbing the volatiles onto the megabore column within the gas chromatograph. The fused silica capillary column is then temperature programmed to separate the volatiles prior to detection by the mass spectrometer.

4.0 INTERFERENCES

4.1 Method interferences may be caused by contaminants in solvents, reagents, out-gassing from equipment plumbing, and laboratory solvent vapors. This can lead to discrete artifacts and/or elevated baseline. These materials are demonstrated to be free from interferences by the analysis and reporting of laboratory reagent blanks.

4.2 Interferences may also be caused by the diffusion of volatiles through the septum seal during storage and handling. Bi-monthly, a refrigerator blank prepared from reagent water is stored with the samples and analyzed to verify a contaminant-free environment. The results for the refrigerator blanks are submitted to the QA Manager to be reviewed and filed for future reference.

4.3 Burdick and Jackson Purge and Trap grade methanol (or equivalent) shall be used for standards and sample dilutions to prevent interferences associated with non-high purity solvents.

4.4 Only reagent water from the Dracor water system in the volatiles laboratory shall be used for sample dilutions, reagent blanks, and aqueous standards to ensure that contaminant-free water is used.

4.5 Interferences from samples with high concentrations of targets are eliminated by ensuring the system is cleared of high levels with the analysis of a blank (see Section 11.2.1.9).

5.0 SAFETY

5.1 Analysts are provided with lab coats, safety glasses, and chemical resistant gloves for handling samples and preparing standards. Analysts shall treat all samples as a potential health hazard and take all appropriate safety precautions.

5.2 Solvents, samples and all standards shall be used in the fume hoods to minimize exposure to solvent vapors.

5.3 Material Safety Data Sheets (MSDS) for all chemicals used in the laboratory are present in the laboratory for immediate access.

6.0 SAMPLE COLLECTION, PRESERVATION AND STORAGE

6.1 All samples for volatile analysis must be protected from light and refrigerated at 4 $\pm$ 2°C from the time of collection until analysis.

6.2 All samples for volatile analysis shall be analyzed within 14 days from sampling, or less.

6.3 Water samples may be collected in glass containers having a minimum volume of 40 mL with a Teflon-lined septum and an open top screw-top cap. Samples should be collected so that no air bubbles pass through the sample while the sample container is being filled. Seal the vial so that no air bubbles are trapped inside. Water samples must be preserved at a pH $<$ 2 at the time of collection.

6.4 Soil samples may be collected in glass containers with Teflon liner or closed end tubes. Headspace should be avoided.

7.0 APPARATUS AND MATERIALS

7.1 Concentrator

7.1.1 Tekmar Purge and Trap Model 2000

7.1.2 Tekmar Purge and Trap Model 3000

7.2 Autosampler

7.2.1 Purge and Trap - Tekmar Model 2016

7.2.2 Sample heater

7.3 Volatile Trap - VOCARB 3000 (Carbopack B/Carboxen 1000 & 1001) and EPA624 equivalent

7.4 Detectors

7.4.1 Sample Analysis

7.4.1.1 Mass Spectrometer HP5970

7.4.1.2 Mass Spectrometer HP5971

7.5 Gas Chromatograph

7.5.1 Sample analysis - HP5890 Series II

7.6 Instrument data system

7.6.1 Sample analysis - A DOS based PC HP Chemstation with Enviroquant software and an NBS75K library

7.7 Syringes - 10 μL, 25 μL, 50 μL, 100 μL, 250 μL, 500 μL, 1000 μL, 5 mL, 10 mL, and 25 mL gastight

7.8 Balance - Fisher toploading with 0.01 g sensitivity

7.9 Glassware - 10 mL, 50 mL, and 100 mL class A volumetric flasks

7.10 Column - J&W Scientific 75 meter 0.530 mm ID DB624 or equivalent

7.11 pH paper - Hydracid paper with ranges from 1 to 6 and 1 to 12

7.12 Purified Solid Matrix - Washed, dried, and baked sea sand

7.13 Extraction Vial - 20 mL glass vial with Teflon-lined septum and open top screw-top cap

7.14 Extract Storage Vial - 2 mL amber glass vial with Teflon-lined septum and open top screw-top cap

7.15 Minicartridge - Data tape for storage system. (3M's DC-2120 or equivalent)

7.16 Pipette - 1 to 5 mL autopipette with disposable tips

7.17 Chlorine test strips

8.0 REAGENTS AND STANDARD PREPARATION

8.1 Reagent Water - provided by the Dracor system in the volatiles laboratory.

8.2 Methanol - Burdick and Jackson Purge and Trap Grade, or equivalent.

8.3 Sample Analysis Standards - all standard solutions must be brought to ambient temperature before use. Preparation of all standards must be documented in the Standards Logbook (Attachment I).

8.3.1 Stock Standards - certified standards purchased from commercial sources containing ampulated mixes of target compounds, matrix spike compounds, surrogates, and internal standards are used. Stock standard receipt is documented in the Stock Standards Receipt Logbook (Attachment II).

8.3.2 Working Standards - all standard solutions are dilutions of stock standards and are stored in mini-vials with mininert valves. All standards must be brought to ambient temperature before use.

8.3.2.1 Calibration Standard (Cocktail) - A solution is prepared directly into a mininert vial in methanol containing 200 ppm of all target compounds found in Table 1.

8.3.2.2 Internal Standards (IS) - A solution is prepared in methanol containing 25 ppm of chlorobenzene-d5, and 1,4-difluorobenzene, 1,4-dichlorobenzene-d4 and pentafluorobenzene.

8.3.2.3 System Monitoring Compounds (SMC/Surrogates) - A solution is prepared in methanol containing 25 ppm of 1,2-dichloroethane-d4, toluene-d8, and 4-bromofluorobenzene.

8.3.2.4 Internal Standards/System Monitoring Compounds (IS/SMC mix) - A solution is prepared in methanol containing 25 ppm of the compounds listed in 8.3.2.2 and 8.3.2.3.

8.3.2.5 Matrix Spike Compounds (MS) - A solution is prepared in methanol containing 25 ppm of 1,1-dichloroethene, trichloroethene, chlorobenzene, toluene, and benzene.

8.3.2.6 Instrument Performance Check Solution - Either of the solutions in 8.3.2.3 or 8.3.2.4 may be used for the BFB tune standard.

8.3.2.7 Labortory Control Sample Solution (LCS) - A solution is prepared in methanol containing 25 ppm of all compounds in Section 13.7. The LCS solution must be prepared from a different standard lot or standard mix from a different vendor than used in Section 8.3.2.1.

8.4 Expiration Dates for Standards

8.4.1 Standards at a concentration $<$ 1000 ppm are replaced weekly or sooner if suspect.

8.4.2 Standards at a concentration $\geq$ 1000 ppm are replaced monthly of sooner if suspect.

8.4.3 Ampulated standards have an expiration date specified by the manufacturer.

9.0 CALIBRATION

9.1 Sample Analysis Calibration

9.2 The mass spectrometer must first pass the BFB tuning criteria given in Section 13.6.

9.3 Five levels of aqueous initial calibration standards containing target compounds and surrogates are prepared at curve concentrations of 10, 20, 50, 100, and 200 ppb. These standards are prepared from working standards listed in Section 8.4.

9.4 The working calibration of this method is defined by the initial calibration curve. All samples with a target compound exceeding the concentration of the highest curve point must be diluted to within the upper half of the calibration range.

9.5 The calibration curve is prepared by adding the following amounts of working calibration standard to a 100 mL volumetric flask containing $\approx$99 mL of reagent water.

Concentration Level (base) (ppb)	Calibration Standard (uL)	SMC Standard (uL)
10	5	2
20	10	4
50	25	10
100	50	20
200	100	40

After the spiking standard solution into flask, bring to volume, cap and invert three (3) times. Immediately decant 5 mL of the aqueous standard, omitting the portion contained in the neck of the flask.

9.5.1 IS spiking solution is added (10 μL) to all calibration standards, once decanted into the 5 mL gas tight syringe, for a final concentration of 50 μg/L.

9.5.2 SMC standard solution is added to all calibration standards once decanted into the 5 mL syringe.

9.6 An initial calibration must be analyzed on each GC/MS system when the ion source is changed, when the column is changed and when the continuing calibration criteria can not be met even after all necessary corrective actions have been performed. The initial calibration must meet criteria as stated in Section 13.5.1.

9.7 Separate initial and continuing calibration must be analyzed for water and low soil samples and meet criteria as stated in Section 13.5. Extracts of medium level soil samples are to be analyzed on a water calibration.

9.8 If less than 12 hours has expired since the tuning compound was injected, after meeting the initial calibration criteria, samples may be analyzed. No continuing calibration is required. Quantitation is based on the third level of the initial calibration. A method blank must be analyzed. If time does not remain in the 12 hour time period, then a new injection of the instrument performance check solution must be made.

10.0 QUALITY CONTROL

10.1 A Method detection limit (MDL) determination is required by this method, and is performed on an annual basis in accordance with the SOP for Condcuting MDL Studies, IEA Doc# QAS02000.NET.

10.2 Each analyst must make an initial, one-time demonstration of the ability to generate acceptable percision and accuracy with this method.

10.3 Instrument Performance Tests

10.3.1 Prior to initiating any data collection activities, it is necessary to establish that a given GC/MS system meets the instrument performance criteria. This is accomplished through the analysis of 50 ng of 4-bromofluorobenzene (BFB), from Section 8.3.2.6, at the start of every 12 hour sequence.

10.3.2 The key ions produced during the analysis of BFB and their respective ion abundance criteria are given in Section 13.6. This criteria must be met before any calibration standards, blanks, or samples may be analyzed.

10.3.3 If the criteria is not met, the BFB must be reanalyzed. Repeated failure requires the instrument to be manually hardware tuned. After manual tune, the BFB must be reinjected and the abundance criteria must be met before proceeding.

10.3.4 After the instrument performance criteria is met, the initial calibration curve must be verified through the analysis of a continuing calibration standard near the midpoint of the calibration range. The continuing calibration criteria in Section 13.5.2 must be met before any method blank or sample analysis may proceed.

10.3.5 A method blank consisting of 5 mL of reagent water spiked with 10 μL IS/SMC at 25 ppm for a final concentration of 50 ppb is analyzed after any daily standard. An acceptable method blank must meet the criteria in Section 13.1.

10.3.6 All volatile analyses associated with a method blank that do not meet the above requirements must be considered for reanalysis. Should reanalysis not occur, the sample data associated with that method blank will be flagged with a "B" for the failing compound(s).

10.4 Matrix Spike and Matrix Spike Duplicates (MS/MSD)

10.4.1 A MS/MSD must be analyzed per matrix, per instrument, per day, per sample batch. (Day is defined as the 24 hour period following the first tune of the day.)

10.4.2 The acceptance limits for matrix spike compound recovery and relative percent difference (RPD) are given in Section 13.4.

10.4.3 The MS/MSD should be analyzed at the same dilution as the sample.

10.4.4 The IS/SMC recoveries should match the trend established in the sample.

10.5 System Monitoring Compounds (Surrogates)

10.5.1 SMC's are added to each sample, blank, standard, and MS/MSD, prior to purging or extracting at 50 μg/L for waters, 50 μg/Kg for low level soils, and 6250 μg/Kg for medium/high level soils.

10.5.2 SMC recoveries must be within the QC limits given in Section 13.2. If the recovery for any one SMC is not within limits, the following are required:

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

10.5.2.2 Check for matrix interference. If the matrix causes ion interference, use a secondary ion to quantitate the analyte in question.

10.5.2.3 Reanalyze the sample if none of the above steps solves the problem.

10.5.2.4 If re-analysis also fails, check for a confirming trend. If no such trend is found, then repeat the re-analysis. Report all of the data for review by data reviewers and/or department manager for reporting with narrative comments regarding the surrogate recovery.

10.5.3 For medium level soil extracts, do not reanalyze or reextract if a SMC recovery is outside the limits when a dilution greater than 500x is made of the sample. If the dilution is 500 or less, then proceed as above with reanalysis and/or reextraction of the sample. Medium level soil samples must meet soil SMC recovery limits.

10.6 Internal Standards

10.6.1 IS's are added to each sample, blank, standard, and MS/MSD, at 50 μg/L at the time of purging.

10.6.2 The retention times (RT) and area of each IS must be evaluated for all analyses. The IS areas must be monitored and evaluated for each sample, blank, MS, and MSD. IS recoveries must pass the criteria given in Section 13.3.

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

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

10.6.3.2 Reanalyze the sample if none of the above steps solve the problem.

10.6.3.3 If the reanalysis also fails, check for a confirming trend. If no such trend is found, repeat the reanalysis. Report all of the data for review by data reviewers and/or department manager for reporting with narrative comments regarding the surrogate recovery.

10.7 Laboratory Control Sample (LCS)

10.7.1 LCS standards must not be made from the same stock used to prepare the initial calibration standards. A different manufacturer's stock mixes or a different lot from the same manufacturer may be used.

10.7.2 The percent recovery limits for the LCS are provided to QA for approval. A minimum of 20 sample results of LCS are compiled. The concentration of each analyte is used to calculate a standard deviation and percent recoveries. The limit for each compound is set to three times the standard deviation.

10.7.3 LCS limits are updated annually and are found in Section 13.7.

10.7.4 An LCS must be analyzed daily, per sample batch, per instrument. The criteria are listed in Section 13.7.

10.7.5 The large number of compounds quantitated by this method presents a substantial probability that one or more may fail the acceptance criteria. When one or more of the compounds fail, repeat the LCS only for those compounds that failed to meet criteria. A second failure, however, confirms a general problem with the measurement system. If this occurs, sample analysis must stop until the source of the problem is located and corrected. Samples already analyzed should be considered for re-analysis and a PSR issued notifying the project manager of the reanalysis requirements.

10.8 Analytical Documentation Procedures

10.8.1 An instrument directory is created for each tune batch to organize the associated data. Batch designations are of the format YYMMDDXX.b. Where, YY = year, MM = month, DD = day, XX =instrument ID. Numbers are used for first shift, letters for second shift. Example: The batch identification number for a tune batch run on instrument MSD9 during the first shift on 09/15/94 would be 9409159.b. The batch identification number for a tune batch run on MSD9 during the second shift on 09/15/94 would be 940915I.b. Note that "I" is the ninth letter of the alphabet.

10.8.2 Instrument Runlogs

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

10.8.2.2 Each instrument has its own set of bound runlogs which are sequentially numbered and paginated. Runlogs are archived with QA once they have been completed. Each analytical sequence shall be started on a new page of the log and continued on the next page, if necessary. The header information designating the standard codes used shall be completed for each sequence. See Attachment III.

10.8.3 Project Status Report (PSR)

10.8.3.1 A PSR is issued when any deviation from this SOP occurs. The PSR is prepared by the person first identifying the problem and is submitted to the department's manager. The manager shall copy and distribute the PSR to the department individuals involved.

10.8.3.2 The lower portion of the PSR is for client notification and the recommended corrective action. It is completed by the project manager when a corrective action decision has been made. The PSR is then redistributed to all the department individuals involved. See Attachment IV. A copy of the completed PSR shall be placed in the project folder.

10.9 Internal Chain of Custody (COC's)

10.9.1 When samples requiring internal COC's are removed from storage for preparation or analysis they must be signed out utilizing the internal chain-of-custody record. The samples shall then be signed back in on the COC upon their return to storage. See Attachment V.

10.10 Practical quantitation limits (PQL's) for this method are based on a 5 mL/ 5 g to 5 mL purge volume and are listed in Table 2.0.

11.0 SAMPLE PREPARATION AND INSTRUMENTAL PROCEDURES

11.1 Instrumental Conditions

11.1.1 Autosampler

11.1.1.1 Sample Analysis Purge and Trap Device

Purge Gas: Helium
Purge Time: 11 $\pm$ 0.2 minutes
Purge Flow: $\approx$40 mL/min
Trap Purge Temp.: 35°C
Trap Desorb Preheat Temp.: 245°C
Trap Desorb Temp.: 250°C
Desorb Flow: $\approx$10 mL/min
Desorb Time: 6.0 min
Bake Temp.: 260°C
Bake Time: 8.0 min

11.1.2 Gas Chromatograph

11.1.2.1 Sample analysis

Carrier Gas:Helium
Flow Rate: ≈10 mL/min
Initial Temp.: 35°C
Initial Hold: 5 minutes
Ramp Rate 1: 6°/min
Final Temp.: 155°C
Final Hold: ≈1.0 min

11.1.3 Mass Spectrometer

Electron Energy: 70 eV
Mass Range: 35 - 300 amu
Scan Time: 0.6-2 sec/scan

11.2 Sample Analysis Procedures

11.2.1 Analyzing Water Samples

11.2.1.1 Remove the sample from the refrigerator. Sign the internal chain-of-custody form, if required. If insufficient sample amount or a multiphase sample is received, a PSR should be issued before sample analysis.

11.2.1.2 Allow the samples to warm to ambient temperature.

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

11.2.1.4 In a gas tight 5 mL syringe, load a 5 mL aliquot of sample by first removing the plunger. Open the sample and carefully pour it into the barrel of the syringe until just short of overflowing. Replace the syringe plunger and adjust to 5 mL. This procedure destroys the integrity of the sample for future analysis, therefore, if there is only one vial, the analyst shall fill a second gas tight syringe in the same manner, attaching a closed syringe valve. This second syringe is maintained only until such time as the analyst has determined that the first sample has been properly analyzed. If an analysis is required from the second 5 mL syringe, it must be performed within 24 hours.

11.2.1.5 Spike the sample with 10 μL of the IS/SMC 25 ppm spiking solution, then inject the syringe contents into the sparging vessel. If an autosampler is utilized, set up the autosampler start and stop positions for the sequence being purged.

11.2.1.6 The pH of the remaining sample in the VOA vial is then measured using 1-6 range pH paper and recorded in the instrument log. Test the pH by placing one or two drops of the sample on the pH paper (do not add pH paper to the vial). Record the pH of each water sample in the instrument run log. No pH adjustments shall be performed by the laboratory. Should th pH exceed 2, issue a PSR to notify the project manager.

11.2.1.7 The chlorine test strip is used to determine if residual chlorine remains in the sample. Use the remaining sample in the vial to test for residual chlorine in the same manner as pH was tested. Notify the project manager with a PSR if a sample tests positive for residual chlorine. The client will be notified of a possible need for resampling.

11.2.1.8 Start the purge cycle on the Purge and Trap device.

11.2.1.9 Rinse the chamber with a minimum of two 5 mL flushes of reagent water between samples to avoid carryover.

11.2.1.10 When a sample with saturated ions from a non-target or a target exceeding the calibration range has been analyzed, the analysis must be followed by an instrument blank in the same purge chamber. A blank in this chamber containing less than the PQL for the target that exceeded the calibration range and/or a non-target less than 10% of the area of the nearest IS must be analyzed before that chamber is used for sample analysis again.

11.2.1.11 When a sample with saturated ions from a non-target or a target exceeding the calibration range has been analyzed on an autosampler, the following analyses must be monitored. The first sample to contain less than the PQL for the target that exceeded the calibration range and/or a non-target less than 10% of the area of the nearest IS may be submitted. The following analysis may also be considered for submittal. However, the contaminated chamber must still have an instrument blank containing less than the PQL for the target that exceeded the calibration range and/or a non-target less than 10% of the area of the nearest IS to prove decontamination of that chamber before sample analysis in that chamber, even on another autosequence, can resume.

11.2.1.12 If a sample containing target compounds at concentrations greater than the initial calibration upper limit (but not at saturating levels) is analyzed, the sample must be reanalyzed at an appropriate dilution. The purge and trap system shall be demonstrated to be free from carryover through the subsequent analyses of blanks and/or samples which do not contain the target compound at a concentration greater than the PQL.

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

11.2.1.14 A water MS/MSD is prepared by spiking the sample aliquot with 10 μL IS/SMC, along with 10 μL of matrix spike solution. The spiked sample is then analyzed as previously described.

11.2.1.15 A method blank must be analyzed every 12 hours after the calibration criteria have been achieved. The method blank consists of 5 mL reagent water spiked with 10 μL of IS/SMC, and carried through the analytical procedure. Method blank criteria is defined in Section 13.1.

11.2.2 Analyzing Low Level Soil Samples

11.2.2.1 Remove the sample from the refrigerator. Sign the internal chain-of-custody form, if required. If insufficient sample amount or a multiphase sample is received, a PSR should be issued before sample analysis.

11.2.2.2 Allow the samples to warm to ambient temperature.

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

11.2.2.4 The sample consists of the entire contents of the sample container. Do not discard any supernatant liquids. Mix the contents of the sample container with a clean, narrow metal spatula. Weigh out 5.0 grams of sample (or the amount determined by screening) into a culture test tube on a balance calibrated once each 12-hour work shift. Attach the test tube to the purge and trap device immediately and tighten the fitting to ensure a gas tight seal.

11.2.2.5 In a gas tight 5 mL syringe, load a 5 mL aliquot of reagent water and spike with 10 μL of IS/SMC solution, then inject it into the test tube with the soil sample. If an autosampler is utilized, set up the autosampler start and stop positions for the sequence being purged, utilizing the sample heater for a heated purge of 40 °C.

11.2.2.6 If a sample containing target compounds at concentrations greater than the initial calibration upper limit is analyzed, then the sample must be reanalyzed at an appropriate dilution. No less than 0.5 g of sample may be analyzed utilizing the low level soil method. If a larger dilution is required, then the medium level soil method (11.2.3) must be employed.

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

11.2.2.8 A low level soil MS/MSD is prepared by spiking the sample aliquot with 10 μL IS/SMC, along with 10 μL of matrix spike solution. The spiked sample is then analyzed as previously described.

11.2.2.9 A low level soil method blank must be analyzed every 12 hours after the calibration criteria has been achieved. The method blank consists of 5.0 grams of purified sand added to 5.0 mL of reagent water. Method blank criteria is defined in Section 13.1.

11.2.3 Analyzing Medium/High Level Soil Samples

11.2.3.1 Remove the sample from the refrigerator. Sign the internal chain-of-custody form, if required. If insufficient sample amount or a multiphase sample is received, a PSR should be issued before sample analysis.

11.2.3.2 Allow samples to warm to ambient temperature.

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

11.2.3.4 The sample consists of the entire contents of the sample container. Do not discard any supernatant liquids. Mix the contents of the sample container with a narrow clean, metal spatula. A medium level soil extract is prepared by weighing out 4.0 grams of sample into a 20 mL extraction vial. Quickly add 9.0 mL of purge and trap grade methanol and 1.0 mL of SMC mix to the vial. Cap and shake the extraction vial for two minutes. Allow the extraction to proceed for at least three minutes, centrifuge as necessary.

11.2.3.5 Using a disposable pipette, transfer $\approx$2 mL of sample extract to a 2 mL vial for storage. The remainder of the extract may be discarded. These extracts may be refrigerated prior to analysis.

11.2.3.6 Fill a gas tight 5 mL syringe until overflowing with reagent water and adjust the plunger to 4.9 mL, spike with 10 μL of IS spiking solution and 100 μL of sample extract or the amount determined by screening. The total volume of methanol must be 100 μL excluding IS. If an autosampler is utilized, set up the autosampler start and stop positions for the sequence being purged.

11.2.3.7 Analyze the medium level soil extract under a water initial and continuing calibration.

11.2.3.8 If an extract containing target compounds at concentrations greater than the initial calibration upper limit is analyzed, then the extract must be reanalyzed at an appropriate dilution. Dilutions requiring volumes of less than 10 μL of sample extract shall be diluted serially. Add the determined volume of sample extract and the volume of reagent methanol so that the total volume of methanol purged is 100 μL.

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

11.2.3.10 A medium level soil MS/MSD is prepared by weighing out 4.0 grams of sample. Quickly add 8.0 mL methanol, 1.0 mL SMC, along with 1.0 mL of matrix spike solution. Continue the extraction process as in Sections 11.2.3.4 - 11.2.3.6. The spiked sample extract is then analyzed as previously described.

11.2.3.11 A method blank must be analyzed every 12 hours after the calibration criteria have been achieved. The method blank consists of 4.0 grams of sand for method blanks, spiked with 1.0 mL SMC and 9.0 mL of reagent methanol. Method blank criteria are defined in Section 13.1.

11.3 Qualitative Analysis

11.3.1 Target Compounds

11.3.1.1 The relative retention time of a target compound must be within +/- 0.06 RRT units of the RRT of the calibration standard for a positive identification. For reference, the standard must be analyzed within the same 12 hour time period as the sample. If the sample is analyzed within the same 12 hour time period as the initial calibration, use the 50 μg/L standard as the reference. If coelution of interfering components prohibits accurate assignment of the sample compounds RRT from the total ion chromatogram, the RRT shall be assigned by using the extracted ion current profiles for ions unique to the component of interest.

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

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

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

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

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

11.3.2 Tentatively Identified Compounds

11.3.2.1 A library search should be performed for non-target compounds in the sample for purposes of tentative identification. For this purpose, the most recent release available of the NBS mass spectral library shall be used.

11.3.2.2 Up to 10 volatile organic compounds not listed in Table 1, shall be tentatively identified via a probability based matching (PBM) library search. Only compounds with responses greater than 10% of the closest IS, exhibiting no interference, that met the TIC guidelines are identified. A TIC must between elute 30 seconds before the first target compound and 3 minutes after the last. Methanol, carbon dioxide, and semi-volitile targets are not identified as TICs.

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

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

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

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

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

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

11.4 Quantitative Analysis

11.4.1 Target Compounds

11.4.1.1 Target compounds are quantitated by the internal standard technique (calculations are in Sections 12.5.1-12.5.3).

11.4.1.2 All manual integrations must be initialed and dated by the analyst.

11.4.1.3 The first order linear regression equation from the initial calibration is used to calculate the concentration in the sample. When compound concentrations are below the PQL and above the MDL, but the compound meets identification criteria, report the concentration with a "J" qualifier. The "J" qualifier also applies to TICs.

11.5 Instrument Maintenance

11.5.1 All instrumentation is covered by a service contract with an external instrumentation service vendor or by IEA personnel trained in preventive maintenance. Preventive maintenance is performed at scheduled intervals on all equipment. All instrument preventive maintenance is performed according to the manufacturers recommended procedures, by trained personnel. All preventive maintenance shall be thoroughly documented in the maintenance log (Attachment VI).

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

12.0 CALCULATIONS

12.1 Relative Response Factor (RRF)

$$RRF = (Ax)(Cis)/(Ais)(Cx)$$

where,

Ax = area of the compound quantitation ion

Ais = area of IS quantitation ion

Cis = IS concentration

Cx = compound concentration

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

The RRF used for quantitation of Xylenes (total) is based upon the ortho-Xylene isomer peak. The total area from the three isomers is used as the area for Xylenes (total). All three isomers must be present in the initial and continuing calibration standards.

12.2 Percent Relative Standard Deviation (%RSD)

$$\%RSD = \frac{\text{(standard deviation)}}{\text{average RF}} \times 100\%$$

average RF = average of five initial Response Factors for a compound

12.3 Percent Difference (%D)

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

where,

average RRFi = average RRF from the initial calibration

RRFc = RRF from the continuing calibration standard

12.4 Percent Moisture

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

12.5 Target Compound Concentrations

12.5.1 Water samples

Quantitate from first order linear regression curve for each target compound.

12.5.1.1 Determination of the slope (m):

$$m = \frac{\sum xy}{\sum x^2}$$

where,

x = concentration of the compound in the standard

y = response factor from the curve

12.5.1.2 Determination of the concentration of the compound in the sample (x):

$$x = y/m$$

where,

y = response factor of the compound in the sample

m = slope (Section 12.5.1.1)

12.5.2 Low soil samples

Calculate the concentration for each target compound as in water calculations factoring in the dry weight of the sample.

Final concentration of the compound in the sample:

Final concentration = (x)(DW)

where,

x = concentration in the sample

DW = Dry weight

12.5.3 Medium soil samples

Calculate in the same manner as low soils.

12.6 SMC Percent Recovery

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

12.7 Matrix Spike Recovery

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

where,

SSR = spiked sample result

SR = sample result

SA = spike added

12.8 Relative Percent Difference

$$\text{RPD} = \left[\frac{\text{MSR - MSDR}}{\text{(MSR+MSDR)/2}} \right] \times 100\%$$

where,

MSR = matrix spike recovery

MSDR = matrix spike duplicate recovery

12.9 Adjusted Practical Quantitation Limit for Samples

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

where,

D = (100 - %moisture)/100

Df = Dilution factor

12.10 Percent dry weight

$$\% \text{ dry weight} = \frac{\text{(g) dry sample}}{\text{(g) wet sample}} \times 100\%$$

13.0 ACCEPTANCE OF DATA

13.1 Method Blank

13.1.1 The method blank must contain less than or equal to the PQL of the target compounds, except methylene chloride, acetone, and 2-butanone, which must be less than or equal to five times the PQL.

13.1.2 If a method blank exceeds the limits for contamination above, the laboratory shall investigate the source of the contamination. Appropriate corrective actions will be taken and documented before further sample analysis proceeds.

13.2 Surrogate Recovery limits must be met for all blanks, samples, and MS/MSD's. The criteria are as follows for both waters and soils:

Compound	%Recovery Water/Soil
1,2-Dichloroethane-d4	70-139
Toluene-d8	90-123
Bromofluorobenzene	75-129

13.3 Internal Standard limits must be met for all blanks, samples, LCSs and MS/MSDs. The criteria are as follows:

13.3.1 The IS area for the samples must be between -50 - +200% of the area in the continuing calibration.

13.3.2 The RT shift must be between ±30 seconds from the RT in the last continuing calibration.

13.4 Matrix Spike/Matrix Spike Duplicate criteria are as follows:

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

13.5 Initial and Continuing calibration criteria.

13.5.1 Initial Calibration

13.5.1.1 System Performance Check Compounds (SPCCs) are checked for minimuim average response factor.

SPCC	Minimum average RRF
Chloromethane	0.100
1,1-Dichloroethane	0.100
Bromoform	0.250
1,1,2,2-Tetrachloroethane	0.300
Chlorobenzene	0.300

In addition, the minimum RRF for any compound in the base curve has a limit of 0.01, except for the following compounds for which this is an advisory limit only: acrylonitrile, acetone, 2-butanone, cis-1,4-dichloro-2-butene and trans-1,4-dichloro-2-butene.

13.5.1.2 Calibration Check Compounds (CCCs) are checked for maximum percent relative standard deviation (%RSD).

CCC	Maximum %RSD
1,1-Dichloroethene	<30%
Chloroform	<30%
1,2-Dichloropropane	<30%
Toluene	<30%
Ethylbenzene	<30%
Vinyl chloride	<30%

The %RSD may not be equal to 30%. The maximum %RSD for any other compounds is <50%.

13.5.2 Continuing Calibration

13.5.2.1 System Performance Check Compounds (SPCCs) are checked for minimuim response factor.

SPCC	Minimum average RRF
Chloromethane	0.100
1,1-Dichloroethane	0.100
Bromoform	0.250
1,1,2,2-Tetrachloroethane	0.300
Chlorobenzene	0.300

In addition, the minimum RRF for any compound in the base curve has a limit of 0.01, except for the following compounds: acrylonitrile, acetone, 2-butanone, cis-1,4-dichloro-2-butene and trans-1,4-dichloro-2-butene.

13.5.2.2 Calibration Check Compounds (CCCs) are checked for maximum percent difference (%D) from the curve.

CCC	Maximum %D
1,1-Dichloroethene	<20%
Chloroform	<20%
1,2-Dichloropropane	<20%
Toluene	<20%
Ethylbenzene	<20%
Vinyl chloride	<20%

The %D may not be equal to 20%. The %D for any other compound has an advisory limit of <50%.

13.6 Bromofluorobenzene tuning criteria.

Mass	Relative to mass	Lower limit	Upper limit
50	95	15%	40%
75	95	30%	60%
95	95	100%	100%
96	95	5%	9%
173	174	0%	2%
174	95	>50%	
175	174	5%	9%
176	174	>95%	<101%
177	176	5%	9%

13.7 The LCS acceptance criteria is 10-200% recovery for all compounds until precision and accuracy analyses are performed to determine the limit for each compound.

14.0 REPORTING OF RESULTS

14.1 Results will be reported in the following format:

14.2 A batch is defined as all samples per instrument, per method, per matrix, per day, up to 20 reportable samples.

14.3 Level I: Results will be reported by form I's (results page) for samples and blanks only.

14.4 There are four types of level II QC as described by the following.

14.4.1 Designated MS/MSD for projects which require LCS's to be reported.

14.4.1.1 Report only project specific MS/MSD.

14.4.1.2 Report all LCS's associated with any daily batch which contains any or all of the project samples.

14.4.2 Designated MS/MSD for standard level II report.

14.4.2.1 Report only project specific MS/MSD.

14.4.2.2 If the MS/MSD passes then only report the MS/MSD as the daily batch QC.

14.4.2.3 If the MS/MSD contains any failing recoveries, report the failing MS/MSD and the passing associated LCS as the daily batch QC (when the MS/MSD fails and the LCS is reported, provide a written explanation in a brief case narrative).

14.4.3 Batch MS/MSD for project which requires LCS's to be reported.

14.4.3.1 Report the MS/MSD for the first daily batch that includes the samples from the project being reported.

14.4.3.2 Report all LCS's associated with any daily batch which contains any or all of the project samples.

14.4.4 Batch MS/MSD for standard level II project.

14.4.4.1 Report the MS/MSD for the first daily batch that includes the samples from the project being reported.

14.4.4.2 If the MS/MSD passes then only report the MS/MSD as the daily batch QC.

14.4.4.3 If the MS/MSD contains any failing recoveries, report the failing MS/MSD and the passing associated LCS as the daily batch QC (when the MS/MSD fails and the LCS is reported, provide a written explanation in a brief case narrative).

14.4.5 The laboratory will analyze an LCS with all MS/MSD's whether they pass or fail.

14.4.6 Level III QC will also follow the same requirements as a level II designated MS/MSD except that the LCS is not reported unless specifically requested.

14.4.6.1 Report only project specific MS/MSD.

14.4.6.2 If the MS/MSD passes then only report the MS/MSD as the QC.

14.4.6.3 If the MS/MSD contains any failing recoveries, it will be reported and an LCS must be analyzed and pass to show the instrument is in control (when an LCS is analyzed and is not reported, however, it must be explained in the case narrative that the MS/MSD contained failing recoveries and that an LCS was analyzed and passed therefore showing that the system remained in control).

15.0 SUPPLEMENTAL DOCUMENTS

15.1 SOP for Conducting MDL Studies, IEA Doc# QAS02000.NET.

16.0 REFERENCES

16.1 "Test Methods for Evaluating Solid Waste", SW-846 Third Edition, September 1986, USEPA, Method 8260.

16.2 Code of Federal Regulations, CFR 40, Part 136, Appendix A, July 1, 1991 Revision, Method 624-Purgeables.

16.3 USEPA CLP Statement of Work for Organics Analysis, OLM03.1.

17.0 SUBSTANTIVE REVISIONS

17.1 Section 2.2 revised to reflect revision of the SOP. Section 8.3.2.2 revised: Bromochloromethane omitted. 1,4-dichlorobenzene-d4 and pentafluorobenzene added. Addednum to Section 11.1.1.1: Bake temperature and bake time specified.

TABLE 1

TARGET COMPOUNDS FOR 8260A VOLATILE ANALYSIS

ANALYTE	QUANTITATION ION	SECONDARY ION
Acetone	43	58
Acrylonitrile	53	52, 51
Allyl Chloride	76	41, 39, 78
Benzene	78	
Bromobenzene	156	77, 158
Bromochloromethane	128	49, 130
Bromodichloromethane	83	85, 127
Bromoform	173	175, 254
Bromomethane	94	96
2-Butanone	43	72
n-Butylbenzene	91	92, 134
tert-Butylbenzene	119	91, 134
sec-Butylbenzene	105	134
Carbon Disulfide	76	78
Carbon Tetrachloride	117	119
Chlorobenzene	112	77, 114
Chlorodibromomethane	129	208, 206
Chloroethane	64	66
2-Chloroethylvinyl Ether	63	65, 106
Chloroform	83	85
Chloromethane	50	52
2-Chlorotoluene	91	126
4-Chlorotoluene	91	126
1,2-Dibromo-3-Chloropropane	75	155, 157
1,2-Dibromoethane	107	109, 188
Dibromomethane	93	95, 174
1,2-Dichlorobenzene	146	111, 148
1,3-Dichlorobenzene	146	111, 148
1,4-Dichlorobenzene	146	111, 148
Dichlorodifluoromethane	85	87
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
1,3-Dichloropropane	76	78
2,2-Dichloropropane	77	97
1,1-Dichloropropene	75	110, 77
cis-1,3-Dichloropropene	75	77, 39
trans-1,3-Dichloropropene	75	77, 39
cis-1,4-Dichloro-2-Butene	75	53, 77, 124
trans-1,4-Dichloro-2-Butene	53	88, 75

Attachment I

STANDARDS PREPARATION LOGBOOK

STOCK STANDARD PREPARATION
Lot Code #: _____
Solvent Used: _____

Prepared By: _____
Date Prepared: _____
Expiration Date: _____

Stock Name	Vendor	Stock Lot # / Lot Code #	Logbook / Page #	Stock Conc (ug/mL)
1.				
2.				
3.				
4.				
5.				
6.				
7.				
8.				
9.				
10.				
11.				
12.				

WORKING STANDARD PREPARATION

Standard	Amount Used (uL)	Final Volume (mLs)	Final Conc (ug/mL)	Prepared By	Date Prepared	Date Expires
1.						
2.						
3.						
4.						
5.						
6.						
7.						
8.						
9.						
10.						
11.						
12.						

Comments: _____

_____

Secondary Review By: _____ Date: _____

Attachment II

Stock Standards Receiving Log
GC/GCMS Laboratory

Date Received	Analyst	Standard Name	Stock/Standard Code Lot #	Vendor	Expiration Date	Comments

Secondary Review By:_____ Date:_____

Attachment III

Method: 8240 CLP 624 Other:____

GC/MS Volatile Runlog

Instrument-MSD*

Matrix: Water/Med Soil

EM: _____

Water Curve:_____ Soil Curve:_____

Batch: _____

Filename	IEA Sample ID.	Case #	Date	Time	ALS#	Init/Final Vol.	DF	Dilution Preparation/Comment	IS	Surr	Analyst	Rerun

Tunes:__/___ Surr Fail:___ IS Fail:___ Tot Inj:___ Suc Inj:___ Billables:___

Secondary Review By:_____ Date:_____

Attachment IV

PROJECT STATUS REPORT

Date_____

Project ID_____ Client_____

Lab Due Date_____ Project Due Date_____

Initiated By_____ Department_____

Parameter_____ Sample_____

☐ Late Notification

Status_____

Reason_____

Expected Completion_____

☐ Matrix Discrepancy ☐ Method Discrepancy

Problem_____

Resolution_____

☐ Protocol Exceeded

Date Sampled_____ Date Protocol Expired_____

Date Analyzed_____ Reason_____

CLIENT NOTIFICATION

_____

_____

_____

_____

_____

_____

Attachment V

IEA Corporation Internal Chain of Custody

Login By: Date Received: Time Received:

Workorder # Case # Department Location

Storage Return			Removed To/Reason						Frac	Matrix	Sample ID
Date	Time	By	Date	Time	Frac	Location	Code	By			

Applicable Codes:

AN=Analyze	LI=Login	DG=Digestion	AT=Autosampler
TR=Transfer	SC=Screen	EX=Extraction	
DI=Dispose	ST=Storage	DW=Dry Weight	

Verified by: Date:

Attachment VI

MAINTENANCE LOG BOOK

DATE_____

INSTRUMENT TYPE:_____ INSTRUMENT ID:_____	SERVICE TYPE ☐ DAILY ☐ ROUTINE ☐ REPAIR

SERVICE SUMMARY
DAILY (Preventive Maintenance performed on a daily basis) Service Performed by:_____
ROUTINE (Scheduled or needed service, minor or routine) Service Performed by:_____
REPAIR (Instrument inoperable or does not meet manufacturers specifications for acceptable operation) Service Performed by:_____

Secondary Review By:_____ Date:_____

Industrial & Environmental Analysts, Inc. (IEA)
SOIL VOLATILE MATRIX SPIKE/MATRIX SPIKE DUPLICATE RECOVERY

IEA Project No.: _____
IEA Reference No: _____

COMPOUND	SPIKE ADDED (ug/Kg)	SAMPLE CONCENTRATION (ug/Kg)	MS CONCENTRATION (ug/Kg)	MS % REC #	QC LIMITS REC
1,1-Dichloroethene	50	0	_____	0	59-172
Trichloroethene	50	0	_____	0	62-137
Benzene	50	0	_____	0	66-142
Toluene	50	0	_____	0	59-139
Chlorobenzene	50	0	_____	0	60-133

	SPIKE ADDED (ug/Kg)	MSD CONCENTRATION (ug/Kg)	MSD % REC #	% RPD #	QC LIMITS RPD	QC LIMITS REC
1,1-Dichloroethene	50	_____	0	ERR	22	59-172
Trichloroethene	50	_____	0	ERR	24	62-137
Benzene	50	_____	0	ERR	21	66-142
Toluene	50	_____	0	ERR	21	59-139
Chlorobenzene	50	_____	0	ERR	21	60-133

Comments:

Industrial & Environmental Analysts, Inc. (IEA)
GC/MS PURGEABLES SW-846 METHOD 8260
Laboratory Control Spike (LCS)

IEA Project Number: _____ Date Analyzed: _____
IEA Sample Number: _____ Analysis By: _____
Client Name: _____ Matrix: Soil
Client Project I.D.: _____
Sample Identification: VLCS##

Number	Compound	Amount Spiked (ug/Kg)	Amount Recovered (ug/Kg)	SLCS % REC	Control Limits Lower (%REC)	Upper (%REC)
1	Acetone	20	0.0	0	10	- 200
2	Acrylonitrile	20	0.0	0	10	- 200
3	Allyl Chloride	20	0.0	0	10	- 200
4	Benzene	20	0.0	0	10	- 200
5	Bromobenzene	20	0.0	0	10	- 200
6	Bromochloromethane	20	0.0	0	10	- 200
7	Bromodichloromethane	20	0.0	0	10	- 200
8	Bromoform	20	0.0	0	10	- 200
9	Bromomethane	20	0.0	0	10	- 200
10	2-Butanone	20	0.0	0	10	- 200
11	n-Butylbenzene	20	0.0	0	10	- 200
12	sec-Butylbenzene	20	0.0	0	10	- 200
13	tert-Butylbenzene	20	0.0	0	10	- 200
14	Carbon Disulfide	20	0.0	0	10	- 200
15	Carbon Tetrachloride	20	0.0	0	10	- 200
16	Chlorobenzene	20	0.0	0	10	- 200
17	Chlorodibromomethane	20	0.0	0	10	- 200
18	Chloroethane	20	0.0	0	10	- 200
19	2-Chloroethyl Vinyl Ether	20	0.0	0	10	- 200
20	Chloroform	20	0.0	0	10	- 200
21	Chloromethane	20	0.0	0	10	- 200
22	2-Chlorotoluene	20	0.0	0	10	- 200
23	4-Chlorotoluene	20	0.0	0	10	- 200
24	1,2-Dibromo-3-Chloropropane	20	0.0	0	10	- 200
25	1,2-Dibromoethane	20	0.0	0	10	- 200
26	Dibromomethane	20	0.0	0	10	- 200
27	1,2-Dichlorobenzene	20	0.0	0	10	- 200
28	1,3-Dichlorobenzene	20	0.0	0	10	- 200
29	1,4-Dichlorobenzene	20	0.0	0	10	- 200
30	1,3-Dichloropropane	20	0.0	0	10	- 200
31	2,2-Dichloropropane	20	0.0	0	10	- 200
32	1,1-Dichloropropene	20	0.0	0	10	- 200
33	cis-1,4-Dichloro-2-Butene	20	0.0	0	10	- 200
34	trans-1,4-Dichloro-2-Butene	20	0.0	0	10	- 200
35	Dichlorodifluoromethane	20	0.0	0	10	- 200
36	1,1-Dichloroethane	20	0.0	0	10	- 200
37	1,2-Dichloroethane	20	0.0	0	10	- 200
38	Hexachlorobutadiene	20	0.0	0	10	- 200
39	Isopropylbenzene	20	0.0	0	10	- 200

Industrial & Environmental Analysts, Inc. (IEA)
GC/MS PURGEABLES SW-846 METHOD 8260
Laboratory Control Spike (LCS)

IEA Project Number: _____ Date Analyzed: _____
IEA Sample Number: _____ Analysis By: _____
Client Name: _____ Matrix: Soil
Client Project I.D.: _____
Sample Identification: VLCS##

Number	Compound	Amount Spiked (ug/Kg)	Amount Recovered (ug/Kg)	SLCS % REC	Control Limits Lower (%REC)	Upper (%REC)
40	1,1-Dichloroethene	20	0.0	0	10	- 200
41	cis-1,2-Dichloroethene	20	0.0	0	10	- 200
42	trans-1,2-Dichloroethene	20	0.0	0	10	- 200
43	1,2-Dichloropropane	20	0.0	0	10	- 200
44	cis-1,3-Dichloropropene	20	0.0	0	10	- 200
45	trans-1,3-Dichloropropene	20	0.0	0	10	- 200
46	Ethylbenzene	20	0.0	0	10	- 200
47	Ethyl Methacrylate	20	0.0	0	10	- 200
48	2-Hexanone	20	0.0	0	10	- 200
49	Iodomethane	20	0.0	0	10	- 200
50	p-Isopropyltoluene	20	0.0	0	10	- 200
51	Methacrylonitrile	20	0.0	0	10	- 200
52	Methylene Chloride	20	0.0	0	10	- 200
53	Methyl Methacrylate	20	0.0	0	10	- 200
54	4-Methyl-2-Pentanone	20	0.0	0	10	- 200
55	Methyl-tert-Butyl Ether	20	0.0	0	10	- 200
56	Naphthalene	20	0.0	0	10	- 200
57	Pentachloroethane	20	0.0	0	10	- 200
58	n-Propylbenzene	20	0.0	0	10	- 200
59	Styrene	20	0.0	0	10	- 200
60	1,1,1,2-Tetrachloroethane	20	0.0	0	10	- 200
61	1,1,2,2-Tetrachloroethane	20	0.0	0	10	- 200
62	Tetrachloroethene	20	0.0	0	10	- 200
63	Toluene	20	0.0	0	10	- 200
64	1,2,4-Trichlorobenzene	20	0.0	0	10	- 200
65	1,1,1-Trichloroethane	20	0.0	0	10	- 200
66	1,1,2-Trichloroethane	20	0.0	0	10	- 200
67	Trichloroethene	20	0.0	0	10	- 200
68	Trichlorofluoromethane	20	0.0	0	10	- 200
69	1,2,3-Trichlorobenzene	20	0.0	0	10	- 200
70	1,2,4-Trimethylbenzene	20	0.0	0	10	- 200
71	1,3,5-Trimethylbenzene	20	0.0	0	10	- 200
72	1,2,3-Trichloropropane	20	0.0	0	10	- 200
73	Vinyl Acetate	20	0.0	0	10	- 200
74	Vinyl Chloride	20	0.0	0	10	- 200
75	Xylenes (Total)	60	0.0	0	10	- 200

Comments:

Corresponding Samples:

Industrial & Environmental Analysts, Inc. (IEA)
GC/MS PURGEABLES SW-846 METHOD 8260
Laboratory Control Spike (LCS)

IEA Project Number: _____ Date Analyzed: _____
IEA Sample Number: _____ Analysis By: _____
Client Name: _____ Matrix: Soil
Client Project I.D.: _____
Sample Identification: VLCS##

Number	Compound	Amount Spiked (ug/Kg)	Amount Recovered (ug/Kg)	SLCS % REC	Control Limits Lower (%REC)	Control Limits Upper (%REC)

Filename:

FORM 8260SLCS (2) Rev. 101695

7. IEA SOP FOR SW-846 8260A VOCs LOW CONCENTRATION ANALYSIS

1.0 APPROVALS

The signatures of the following individuals indicate that this SOP is complete and meets the requirements specified in corporate document # QAS00200.NET. In addition, it signifies that the content meets the specifications of the referenced "Test Code".

Laboratory Director

Quality Assurance Manager

2.0 SCOPE AND APPLICATION

2.1 The objective of this document is to outline the techniques for determining the presence and concentration of various volatile organic target and non-target compounds in aqueous, multi-concentration samples. The target compounds for this method are listed in Table 1.0.

2.2 The document control number for this SOP is MSS01100.NC.

2.3 The following test code is used to define analyses in accordance with all of the conditions set forth by this SOP: MSV_35.

3.0 SUMMARY OF METHOD

3.1 This method employs the technique of purge and trap, coupled with a gas chromatograph/mass spectrometer. An aliquot of sample is purged in a gas tight chamber with UHP grade helium to extract the volatile compounds. The vapor is swept onto a sorbent trap where the volatiles are adsorbed. Next, the sorbent trap is heated and backflushed, thereby desorbing the volatiles onto the megabore column within the gas chromatograph. The fused silica capillary column is then temperature programmed to separate the volatiles prior to detection by the mass spectrometer.

4.0 INTERFERENCES

4.1 Method interferences may be caused by contaminants in solvents, reagents, out-gassing from equipment plumbing, and laboratory solvent vapors. This can lead to discrete artifacts and/or elevated baseline. These materials are demonstrated to be free from interferences by the analysis and reporting of laboratory reagent blanks.

4.2 Interferences may also be caused by the diffusion of volatiles through the septum seal during storage and handling. Bi-monthly, a refrigerator blank prepared from reagent water is stored with the samples and analyzed to verify a contaminant-free environment. The results of the refrigerator blanks are submitted to the QA Manager to be reviewed and filed for future reference.

4.3 Burdick and Jackson Purge and Trap grade methanol (or equivalent) shall be used for standards and sample dilutions to prevent interferences associated with non-high purity solvents.

4.4 Only reagent water from the Dracor water system in the volatiles laboratory shall be used for sample dilutions, reagent blanks, and aqueous standards to ensure that contaminant-free water is used.

4.5 An instrument blank must be run in the chamber of any sample which has target compounds exceeding the calibration range.

5.0 SAFETY

5.1 Analysts are provided with lab coats, safety glasses, and chemical resistant gloves for handling samples and preparing standards. Analysts shall treat all samples as a potential health hazard and take all appropriate safety precautions.

5.2 Solvents, samples and all standards shall be used in the fume hoods to minimize exposure to solvent vapors.

5.3 Material Safety Data Sheets (MSDS) for all chemicals used in the laboratory are present in the laboratory for immediate access.

6.0 SAMPLE COLLECTION, PRESERVATION AND STORAGE

6.1 All samples for volatile analysis must be protected from light and refrigerated at 4 $\pm$ 2°C from the time of collection until analysis.

6.2 All samples for volatile analysis shall be analyzed within 14 days from sampling, or less.

6.3 Water samples may be collected in glass containers having a minimum volume of at least 40 mL with a Teflon-lined septum and an open top screw-top cap. Sample should be collected so that no air bubbles pass through the sample while the sample container is being filled. Seal the vial so that no air bubbles are trapped inside. Water samples must be preserved at a pH $<$ 2 at the time of collection.

7.0 APPARATUS AND MATERIALS

7.1 Concentrator

7.1.1 Tekmar Purge and Trap model 2000

7.1.2 Tekmar Purge and Trap model 3000

7.2 Autosampler

7.2.1 Purge and Trap - Tekmar Model 2016

7.2.2 Screening - Perkin-Elmer Model HS-101

7.3 Volatile Trap - VOCARB 3000 (Carbopack B/Carboxen 1000 & 1001) and EPA624 equivalent.

7.4 Detectors

7.4.1 Sample Analysis

7.4.1.1 Mass Spectrometer HP5970

7.4.1.2 Mass Spectrometer HP5971

7.4.2 Sample Screening

7.4.2.1 Photo-ionization/Electroconductivity (PID/HALL in halogen mode)

7.4.2.2 Flame-ionization/Electroconductivity (FID/HALL in halogen mode)

7.5 Gas Chromatograph

7.5.1 Sample analysis - HP5890 series II

7.5.2 Sample screening - Perkin-Elmer 8500

7.6 Instrument data system

7.6.1 Sample analysis - A DOS based PC HP Chemstation with Enviroquant software and an NBS75K library.

7.6.2 Sample Screening - Spectra-Physics SP-4290 Integrator

7.7 Syringes - 10 μL, 25 μL, 50 μL, 100 μL, 250 μL, 500 μL, 1000 μL, 5 mL, 10 mL, and 25 mL gastight.

7.8 Balance - Fisher toploading with 0.01 g sensitivity.

7.9 Glassware - 10 mL, 50 mL, and 100 mL class A volumetric flasks.

7.10 Column - J&W Scientific 75 meter 0.53 mm ID DB624 or equivalent.

7.11 pH paper - Hydracid paper with ranges from 1 to 6 and 1 to 12.

7.15 Minicartridge - Data tape for storage system. (3M's DC-2120 or equivalent)

7.16 Screening Vial - 20 mL crimp top vial using teflon lined septa.

7.17 Pipette - 1 to 5 mL autopipette with disposable tips.

7.18 Chlorine test strips

7.19 Autosampler vials

8.0 REAGENTS AND STANDARD PREPARATION

8.1 Reagent Water - provided by the Dracor system in the volatiles laboratory.

8.2 Methanol - Burdick and Jackson Purge and Trap Grade, or equivalent.

8.3 Sample Analysis Standards - all standard solutions must be brought to ambient temperature before use. Preparation of all standards must be documented in the Volatiles Standards Preparation Logbook (Attachment VI).

8.3.1 Stock Standards - certified standards purchased from commercial sources containing ampulated mixes of all target compounds, matrix spike compounds, surrogates, and internal standards are used. Stock standards receipt is documented in the Stock Standards Receipt Logbook (Attachment VII).

8.3.2 Working Standards - all standard solutions must be brought to ambient temperature before use.

8.3.2.1 Calibration Standard (Cocktail) - A solution is prepared volumetrically in methanol containing 5 ppm of all target compounds identified in Table 1.

8.3.2.2 Internal Standards (IS) - A solution is prepared in methanol containing 12.5ppm of chlorobenzene-d5, 1,4-difluorobenzene, 1,4-dichlorobenzene-d4, and pentafluorobenzene.

8.3.2.3 System Monitoring Compounds (SMC or Surrogates) - A solution is prepared in methanol containing 12.5 ppm of 1,2-dichloroethane-d4, toluene-d8, and 4-bromofluorobenzene.

8.3.2.4 Internal Standards/System Monitoring Compounds (IS/SMC mix) -A solution is prepared in methanol containing 12.5 ppm of the compounds listed in 8.3.2.2 and 8.3.2.3.

8.3.2.5 Matrix Spike Compounds (MS) - A solution is prepared in methanol containing 12.5ppm of 1,1-dichloroethene, trichloroethene, chlorobenzene, toluene, and benzene.

8.3.2.6 Instrument Performance Check Solution (Tune Standard) - Either of the solutions in 8.3.2.3 or 8.3.2.4 may be used.

8.3.2.7 Ketone Standard (Ketone cocktail) - A solution is prepared in methanol containing 20 ppm of acetone, 2-butanone, 4-methyl-2-pentanone, and 2-hexanone.

8.3.2.8 Laboratory Control Sample Solution (LCS mix) - A solution is prepared in methanol containing 12.5 ppm of the LCS compounds in Table 1. The LCS is prepared from a different lot number or vendor from the calibration standards.

8.4 Screening Standards - all standard solutions must be brought to ambient temperature before use.

8.4.1 Stock Standards

8.4.1.1 Calibration Standard - certified standards purchased from commercial sources containing ampulated mixes of all target compounds in Table 1.

8.4.1.2 Internal Standard - a 2000 ppm solution of 2-chlorotoluene is prepared from neat.

8.4.2 Working Standards

8.4.1 Calibration standard - A solution is prepared at 500 ppm containing all screening target compounds.

8.4.2 Internal Standard - A solution is prepared in methanol containing 200 ppm of 2-chlorotoluene.

8.5 Expiration Dates for Standards

8.5.1 Standards at a concentration $<$ 1000 ppm are replaced weekly or sooner, if suspect.

8.5.2 Standards at a concentration $\geq$ 1000 ppm are replaced monthly or sooner, if suspect.

8.5.3 Ampulated standards have an expiration date specified by the manufacturer.

9.0 CALIBRATION

9.1 Screening Calibration

9.1.1 Two aqueous daily calibration standards are prepared at the 100 and 500 ppb levels. The standards are prepared from the working standards in 8.4.2.

9.1.2 The methanol contained in a screening standard must not exceed 50 μL/5mL.

9.1.3 The screening standards are prepared by adding the following amounts of working standard directly into a screening vial.

Final Concentration (ppb)	Calibration Standard (μL)
100	1
500	5

After adding the standard, immediately add 5μL of IS, cap and seal the vial.

9.2 Sample Analysis Calibration

9.2.1 Six levels of aqueous initial calibration standards containing target compounds and surrogates are prepared at the base curve concentrations of 1, 2, 5, 10, 20 and 25 ppb. The Ketones concentrations are 5 times the base curve; 5, 10, 25, 50, 100 and 125 ppb. These standards are prepared from working standards in 8.3. If a compound saturates in the highest level calibration standard, that compound and any others affected will be disabled at the highest level and the five lower points used as the initial calibration.

9.2.2 The working calibration of this method is defined by the initial calibration curve. All samples with a target compound exceeding the concentration of the highest curve point must be diluted to within 10 to 25 ppb on column. The ketone dilution range is 50 to 125 ppb.

9.2.3 The calibration curve is prepared by adding the following amounts of working calibration and SMC standard to a 25 mL syringe containing 25 mL of reagent water. These standards are prepared at the time of calibration.

Concentration Level (base) (ppb)	Calibration and Ketone Standard (μL)	SMC Standard (μL)	Ketone Concentration (ppb)
1	5	2	5
2	10	4	10
5	25	10	25
10	50	20	50
20	100	40	100
25	125	50	125

9.2.4 IS spiking solution is added (10 μL) to all calibration standards for a final concentration of 5 ppb.

9.2.5 An initial calibration must be analyzed on each GC/MS system when the ion source is changed, when the column is changed and when the continuing calibration criteria can not be met even after all necessary corrective actions have been performed.

9.2.6 If less than 12 hours has expired since the tune standard was injected, after meeting the initial calibration criteria, samples may be analyzed. No continuing calibration is required. Quantitation is based on the third level of the initial calibration. A method blank must be analyzed every 12 hour tune. If time does not remain in the 12 hour time period, then a new injection of the tune standard solution must be made.

10.0 QUALITY CONTROL

10.1 A Method Detection Limit (MDL) determination is required by this method, and is performed on an annual basis or when a change in the method or column occurs or after major repair.

10.1.1 A one-time initial demonstration of precision and accuracy is required by each analyst.

10.2 Practical quantitation limits (PQL's) for this method are based on a 25 mL purge volume and listed in Table 2.0.

10.3 Instrument Performance Tests

10.3.1 Prior to initiating any data collection activities it is necessary to establish that a given GC/MS system meets the instrument performance criteria. This is accomplished through the analysis of 50 ng of 4-bromofluorobenzene (BFB) at the start of every 12 hour sequence.

10.3.2 The key ions produced during the analysis of BFB and their respective ion abundance criteria are given in Section 13.6. This criteria must be met before any calibration standards, blanks, or samples may be analyzed.

10.3.4 If the criteria is not met, the BFB must be reanalyzed. Repeated failure requires the instrument to be manually hardware tuned. After manually tuning the instrument, the BFB must be reinjected and the abundance criteria must be met before proceeding.

10.3.5 After the instrument performance criteria is met, the initial calibration curve must be verified through the analysis of a continuing calibration standard at the mid-range concentration. The continuing calibration criteria must be met before any method blank or sample analysis may proceed. Section 13.5 defines acceptance criteria.

10.3.6 A method blank, consisting of 25 mL of reagent water spiked with 10 μL IS/SMC at 12.5 ppm for a final concentration of 5 ppb, is analyzed after any daily standards. An acceptable method blank must meet the criteria in Section 13.1.1.

10.3.7 All volatile analyses associated with a method blank that do not meet the above requirements must be considered for re-analysis. Should re-analysis not occur, the sample data associated with this method blank will be flagged with a "B" for the failing compounds.

10.4 Matrix Spike and Matrix Spike Duplicates (MS/MSD)

10.4.1 A MS/MSD must be analyzed per instrument, per sample batch, per day. A day is defined as the 24 hour period following the first tune of that day.

10.4.2 The limits for matrix spike compound recovery and relative percent difference (RPD) are given in Section 13.4.

10.4.3 The MS/MSD should be analyzed at the same dilution as the sample.

10.4.4 The IS recoveries should match the trend established in the sample. Note that MS/MSD may not be used to confirm failing IS recovery.

10.4.5 The SMC recoveries should match the trend established in the sample. However, MS/MSD may be used to confirm a surrogate recovery trend.

10.5 System Monitoring Compounds (Surrogates)

10.5.1 SMC's are added to each sample, blank, standard, and MS/MSD at 5pp prior to purging.

10.5.2 SMC recoveries must be within the QC limits given in Section 13.2. If the recovery for any one SMC is not within limits, the following are required:

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

10.5.2.2 Check for matrix interference. If the matrix causes ion interference, use a secondary ion to quantitate the surrogate in question.

10.5.2.3 Reanalyze the sample if none of the above steps solves the problem.

10.5.2.4 If re-analysis also fails, check for a confirming trend. If no such trend is found then repeat the re-analysis once. Report all data for evaluation by final data reviewers and/or the department manager. The final report will include a narrative commenting on the surrogate recovery.

10.6 Internal Standards

10.6.1 IS's are added to each sample, blank, standard, and MS/MSD, at 5 ppb at the time of purging.

10.6.2 The retention times (RT) and area of each IS must be evaluated for all analyses. The IS areas must be monitored and evaluated for each sample, blank, MS, and MSD. IS recoveries must pass the criteria given in Section 13.3.

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

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

10.6.3.2 Reanalyze the sample if none of the above steps solve the problem.

10.6.3.3 If the re-analysis also fails, check for a confirming trend. If no such trend is found, repeat the re-analysis once. Report all data for evaluation by final data reviewers and/or the department manager. The final report will include a narrative commenting on the surrogate recovery.

10.7 Laboratory Control Spike

10.7.1 All of the target compounds are in the LCS compound list.

10.7.2 The percent recovery limits for LCSs' are 10 - 200 minimum, for each compound, until historical data is compiled for developing limits.

10.7.2.1 A a minimum of 20 LCSs results are compiled. The concentration of each analyte is used to calculate a standard deviation based on recovery from the amount spiked. The limit for each compound is set to plus or minus three times the standard deviation.

10.7.3 LCS limits are updated anually.

10.7.4 An LCS must be analyzed per instrument, per sample batch, per day. The acceptance criteria are listed in Section 13.7.

10.7.5 The large number of compounds quantitated by this method presents a substantial probability that one or more may fail the acceptance criteria. When one or more of the compounds fail, repeat the LCS only for those compounds that failed to meet criteria. A second failure, however, will confirm a gereral problem with the measurement system. If this occurs, sample analysis must stop until the source of the problem is located and corrected. Samples already analyzed should be considered for re-analysis. Issue a Project Status Report (PSR) notifying the client via the project manager of the reanalysis requirements.

10.8 Analytical Documentation Procedures

10.8.1 An instrument directory is created for each tune batch to organize the associated data. Batch designations are of the format YYMMDDXX.b. Where, YY = year, MM = month, DD = day, XX = instrument ID. Numbers are used for first shift, letters for second shift. Example: The batch identification number for a tune batch run on instrument MSD9 during the first shift on 9/15/94 would be 9409159.b. The batch identification number for a tune batch run on MSD9 during the second shift on 9/15/94 would be 940915I.b. Note that "I" is the ninth letter of the alphabet.

10.8.2 Instrument Runlogs

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

10.8.2.2 Each instrument has its own set of bound run logs which are sequentially numbered and paginated. Run logs are archived once they have been filled and verified. Each analytical sequence shall be started on a new page of the log and continued on the next page, if necessary. The header information designating the standard codes used shall be completed for each sequence. Refer to Attachment I.

10.8.3 Project Status Report (PSR)

10.8.3.1 A PSR is issued when any deviation from this SOP occurs. The PSR is prepared by the person first identifying the problem and is submitted to the department manager and the project manager.

10.8.3.2 The lower portion of the PSR is for client notification and the recommended corrective action. It is completed by the project manager when a resolution has been agreed upon. The PSR is then redistributed to the department individuals involved. Refer to Attachment II. A completed copy of the PSR is placed in the client's job file by the project manager.

10.9.4 Internal Chain of Custody (COC's)

10.9.4.1 When samples requiring internal COC's are removed from storage for preparation or analysis they must be signed out utilizing the chain of custody record. The samples shall then be signed back in on the COC upon their return to storage. Refer to Attachment III.

11.0 SAMPLE PREPARATION AND INSTRUMENTAL PROCEDURES

11.1 Instrumental Conditions

11.1.1 Autosampler

11.1.1.1 Sample Analysis Purge and Trap Device -

Purge Gas: Helium
Purge Time: 11 $\pm$ 0.2 minutes
Purge Flow: $\approx$40 mL/min
Trap Purge Temp.: 35 °C
Trap Desorb Preheat Temp.: 245 °C
Trap Desorb Temp.: 250 °C
Desorb Flow: $\approx$ 10 mL/min
Desorb Time: 6.0 min

11.1.1.2 Screening Headspace Device -

Sample Temp: 85 °C
Needle Temp: 120 °C
Transfer Temp: 150 °C
Thermostat Time: 5.0 min

11.1.2 Gas Chromatograph

11.1.2.1 Sample analysis -

Carrier Gas:Helium
Flow Rate: ≈10 mL/min
Initial Temp.: 35 °C
Initial Hold: 5 minutes
Ramp Rate 1: 6°/min
Final Temp.: 155°C
Final Hold: ≈1.0 min

11.1.2.2 Screening -

Carrier Gas:Helium
Flow Rate: ≈10 mL/min

Initial Temp.: 40 °C
Initial Hold: 4 minutes
Ramp Rate 1 to Temp: 6°/min to 155 °C
Hold 2: 0 min
Ramp Rate 2 to Temp: 20°/min to 200 °C
Final Hold: 2 min

11.1.3 Mass Spectrometer

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

11.2 Sample Screening Procedure

11.2.1 Screening Samples

11.2.1.1 Label a screening vial with the sample identification and the dilution factor.

11.2.1.2 Perform any sample dilution necessary. Then, using a repeating pipettor transfer the sample to the vial, changing pipette tips between samples.

11.2.1.3 Add 5 μL of the screening IS solution to the vial. Immediately cap and seal the vial.

11.2.1.4 Load the samples on the HS-101 autosampler after the calibration standards and blank. Start the sequence on the Perkin-Elmer 8500. Record the sample information in the Screening Logbook (Attachment IV).

11.2.1.5 Quantitate the screening data using the internal standard method. Then make dilution calls based on a target concentration range of 15$\pm$5ppb. Record the dilution calls in the Screening Logbook.

11.3 Sample Analysis Procedure

11.3.1 Analyzing Samples

11.3.1.1 Remove the sample from the refrigerator. Sign the internal chain of custody form, if required. If insufficient sample amount or a multiphase sample is received, a PSR should be issued before sample analysis.

11.3.1.2 Allow the samples to warm to ambient temperature.

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

11.3.1.4 In a gas tight 25 mL syringe, load a 25 mL aliquot of sample by first removing the plunger. Then open the sample and carefully pour it into the barrel of the syringe until just short of overflowing. Replace the syringe plunger and adjust to 25 mL.

11.3.1.4.1 A second gas tight syringe can be prepared at this time if limited sample volume is provided. However, the second syringe must be used wihin 24 hours.

11.3.1.5 Next, spike the sample with 10 μL of the IS/SMC 12.5 ppm spiking solution, then inject the syringe contents into the sparging vessel. If an autosampler is utilized, set up the autosampler start and stop positions for the sequence being purged.

11.3.1.6 The pH of the remaining sample in the VOA vial is then measured using 1-6 range pH paper and this is recorded in the instrument runlog. Test the pH by placing one or two drops of the sample on the pH paper (do not add pH paper to the vial). No pH adjustments shall be performed by the laboratory. However, a PSR should be issued if the ph of the sample is >2.

11.3.1.7 The chlorine test strip is used to determine if residual chlorine reamains in the sample. Use the remaining sample in the vial to test for residual chlorine in the same manner as pH was tested. Notify the project manager with a PSR if a sample tests positive for residual chlorine. The client will be notified of a possible need for resampling.

11.3.1.8 Start the purge cycle on the Purge and Trap device.

11.3.1.9 Rinse the chamber with a minimum of three flushes of reagent water between samples to avoid carryover.

11.3.1.10 When a sample with saturated ions from a non-target or a target exceeding the calibration range has been analyzed, the analysis must be followed by an instrument blank in the same purge chamber. A blank in this chamber containing less than the PQL for the target that exceeded the calibration range and/or a non-target less than 40% of the area of the nearest IS must be analyzed before that chamber is used for sample analysis again.

11.3.1.11 When a sample with saturated ions from a non-target or a target exceeding the calibration range has been analyzed on an autosampler, the following analyses must be monitored. The first sample in subsequent chambers to contain less than the PQL for the target that exceeded the calibration range and/or a non-target less than 40% of the area of the nearest IS may be submitted. All samples following this must be evaluated for carryover and may require reanalysis. The following analysis may also be considered for submittal. However, the contaminated chamber must still have an instrument blank containing less than the PQL for the target that exceeded the calibration range and/or a non-target less than 40% of the area of the nearest IS to prove decontamination of that chamber before sample analysis in that chamber, even on another autosequence, can resume.

11.3.1.12 If a sample containing target compounds at concentrations greater than the initial calibration upper limit (but not at saturating levels) is analyzed then the sample must be reanalyzed at an appropriate dilution. The purge and trap system shall be demonstrated to be free from carryover through the subsequent analyses of blanks and/or samples which do not contain the target compound at a concentration greater than the PQL.

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

11.3.1.14 A water MS is prepared by spiking a sample aliquot with 10 μL IS/SMC, along with 10 μL of matrix spike solution. The spiked sample is then analyzed as previously described. This step is repeated to prepare an MSD sample.

11.3.1.15 A method blank must be analyzed every 12 hours after the calibration criteria has been achieved. The method blank consists of 25 mL reagent water spiked with 10 μL of IS/SMC, and carried through the analytical procedure. Method blank criteria is defined in Section 13.1.1.

11.4 Qualitative Analysis

11.4.1 Target Compounds

11.4.1.1 The relative retention time of a target compound must be within +/- 0.06 RRT units of the RRT of the calibration standard for a positive identification. For reference, the standard must be analyzed within the same 12 hour time period as the sample. If the sample is analyzed within the same 12 hour time period as the initial calibration, then use the 5 ppb standard as the reference. If coelution of interfering components prohibits accurate assignment of the sample compounds RRT from the total ion chromatogram, the RRT shall be assigned by using the extracted ion current profiles for ions unique to the component of interest.

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

11.4.1.2.1 All characteristic ions present in the reference spectrum at a relative intensity greater than 30% must be present in the sample spectrum.

11.4.1.2.2 The relative intensities of the ions above 30% must agree with 30% between the reference and sample spectra.

11.4.1.2.3 Ions greater than 30% in the sample spectrum but not present in the reference spectrum must be considered and accounted for.

11.4.1.3 If a compound cannot be verified by the above criteria, but in the technical judgment of the analyst, the identification is correct, then the compound shall be reported. However the reported data should include a narrative statement of the identification by this criteria.

11.5.2 Tentatively Identified Compounds

11.5.2.1 A library search may be performed, if requested, for non-target compounds in the sample for purposes of tentative identification. For this purpose, the most recent release available of the NBS mass spectral library shall be used.

11.5.2.2 Up to 10 volatile organic compounds not listed in Table 1, shall be tentatively identified via a probability based matching (PBM) library search. Only compounds with responses greater than 40% of the closest IS, exhibiting no interference, that met the TIC guidelines are identified. A TIC must elute between 30 seconds before the first target compound and 3 minutes after the last. Methanol, carbon dioxide, and semi-volatile targets are not identified as TICs.

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

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

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

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

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

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

11.6 Quantitative Analysis

11.6.1 Target Compounds

11.6.1.1 All manual integrations performed on target compounds must be initialed and dated by the analyst or data reviewer performing the manual integration.

11.6.1.2 The linear regression equation from the initial calibration is used to calculate the concentration in the sample (see Sections 12.10, 12.11 and 12.12). When compound concentrations are below the PQL and above the MDL, but the compound meets identification criteria, report the concentration with a "J" qualifier, when requested. The "J" flag denotes an "estimated concentration".

11.7 Instrument Maintenance

11.7.1 All instrumentation is covered by a service contract with an external instrumentation service vendor or by IEA personnel trained in preventive maintenance. Preventive maintenance is performed at scheduled intervals on all equipment. All instrument preventive maintenance is performed according to the manufacturers recommended procedures, by trained personnel. All preventive maintenance shall be thoroughly documented in the instrument-specific Maintenance Logbook (Attachment V).

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

12.0 CALCULATIONS

12.1 Relative Response Factor (RRF)

$$RRF = (Ax)(Cis)/(Ais)(Cx)$$

where,

Ax = area of the compound quantitation ion

Ais = area of IS quantitation ion

Cis = IS concentration

Cx = compound concentration

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

The RRF used for quantitation of Xylenes (total) is based upon the ortho-Xylene isomer peak. The total area from the three isomers is used as the area for Xylenes (total) in samples. All three isomers must be present in the initial and continuing calibration standards.

12.2 Percent Relative Standard Deviation (%RSD)

$$\%RSD = \frac{SD}{mean} \times 100\%$$

where,

SD = standard deviation of relative response factors per compound.

mean = average relative response factors per compound.

12.3 Percent Difference (%D)

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

where,

average RRFi = average RRF from the initial calibration

RRFc = RRF from the continuing calibration standard

12.4 Percent Moisture

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

12.5 Response Factor

$$\text{Resposne Factor} = \frac{Ax}{Ais}$$

where,

Ax = Area of the quantitation ion of the target compound

Ais = Area of the quantitation ion of the associated IS

12.6 SMC Percent Recovery

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

12.7 Matrix Spike Recovery

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

where,

SSR = spiked sample result

SR = sample result

SA = spike added

12.8 Relative Percent Difference

$$RPD = \left[\frac{MSR - MSDR}{(MSR + MSDR)/2} \right] \times 100\%$$

where,

MSR = matrix spike %recovery

MSDR = matrix spike duplicate %recovery

12.9 Adjusted Practical Quantitation Limit for Samples

Adjusted PQL = (PQL)*Df/D

where,

D = (100 - %moisture)/100

Df = Dilution factor

12.10 Determination of the slope (m):

$$m = \frac{\sum xy}{\sum x^2}$$

where,

x = concentration of the compound in the standard

y = response factor from the curve

12.11 Determination of the concentration of the compound in the sample (x):

x = y/m

where,

y = response factor of the compound in the sample

m = slope (Section 12.10)

12.12 Final concentration of the compound in the sample:

Final concentration = (x)(Df)

where,

x = concentration in the sample (Section 12.11)

Df = Dilution factor

13.0 ACCEPTANCE OF DATA

13.1 Method Blank

13.1.1 The method blank must contain less than the PQL of the target compounds.

13.1.2 If a method blank exceeds the limits for contamination above, the laboratory shall investigate the source of the contamination. Appropriate corrective actions will be taken and documented before further sample analysis proceeds.

13.2 Surrogate Recovery limits must be met for all blanks, samples, and MS/MSD's. The criteria are as follows:

Compound	%Recovery Water
1,2-Dichloroethane-d4	62-121
Toluene-d8	90-108
Bromofluorobenzene	78-115

13.3 Internal Standard limits must be met for all blanks, samples, LCSs and MS/MSDs. The criteria are as follows:

13.3.1 The IS area for a continuing calibration must be between -50 and 100% of the IS area in the last continuing calibration.

13.3.2 The RT shift must be between $\pm$30 seconds from the RT in the last continuing calibration.

13.4 Matrix Spike/Matrix Spike Duplicate criteria are as follows:

Compound	%Recovery Water	RPD Water
1,1-Dichloroethene	60-140	20
Benzene	60-140	20
Trichloroethene	60-140	20
Toluene	60-140	20
Chlorobenzene	60-140	20

13.5 Initial and Continuing calibration criteria.

13.5.1 Initial Calibration

Construct the calibration curves of area ratio versus concentration using first order forced origin linear regression. These curves are verfied each tune by purging a performance standard.

13.5.1.1 System Performance Check Compounds (SPCCs) are checked for minimuim average response factor.

SPCC	Minimum average RRF
Chloromethane	0.100
1,1-Dichloroethane	0.100
Bromoform	0.250
1,1,2,2-Tetrachloroethane	0.300
Chlorobenzene	0.300

In addition, the minimum RRF for any compound, except for acrylonitrile, acetone, 2-butanone, cis-1,4-dichloro-2-butene and trans-1,4-dichloro-2-butene, in the base curve has a limit of 0.01.

13.5.1.2 Calibration Check Compounds (CCCs) are checked for maximum percent relative standard deviation (%RSD).

CCC	Maximum %RSD
1,1-Dichloroethene	<30%
Chloroform	<30%
1,2-Dichloropropane	<30%
Toluene	<30%
Ethylbenzene	<30%
Vinyl chloride	<30%

The %RSD may not be equal to 30%. In addition, the maximun %RSD for any other compound has an advisory limit of <50%.

13.5.2 Continuing Calibration

13.5.2.1 System Performance Check Compounds (SPCCs) are checked for minimuim response factor.

SPCC	Minimum average RRF
Chloromethane	0.100
1,1-Dichloroethane	0.100
Bromoform	0.250
1,1,2,2-Tetrachloroethane	0.300
Chlorobenzene	0.300

In addition, the minimum RRF for any compound in the base curve has a limit of 0.01, except for acrylonitrile, acetone, 2-butanone, cis-1,4-dichloro-2-butene and trans-1,4-dichloro-2-butene.

13.5.2.2 Calibration Check Compounds (CCCs) are checked for maximum percent difference (%D) from the curve.

CCC	Maximum %D
1,1-Dichloroethene	<20%
Chloroform	<20%
1,2-Dichloropropane	<20%
Toluene	<20%
Ethylbenzene	<20%
Vinyl chloride	<20%

The %D may not be equal to 20%. In addition, the maximum %D for any other compound has an advisory limit of <50%.

13.6 Bromofluorobenzene tuning criteria.

Mass	Relative to mass	Lower limit	Upper limit
50	95	15%	40%
75	95	30%	60%
95	95	100%	100%
96	95	5%	9%
173	174	0%	<2%
174	95	>50%	---
175	174	5%	9%
176	174	>95%	<101%
177	176	5%	9%

13.7 The LCS acceptance criteria is 10-200% recovery for all compounds until precision and accuracy analyses are performed to determine the limit for each compound.

14.0 REPORTING OF RESULTS

14.1 Results will be reported in the following format:

14.2 A batch is defined as all samples per instrument, per method, per matrix, per day, up to 20 reportable samples.

14.3 Level I: Results will be reported by form I's(results page) for samples and blanks only.

14.4 Level II QC as described by the following:

14.4.1 Designated MS/MSD for project which requires LCS's to be reported.

14.4.1.1 Report only project specific MS/MSD.

14.4.1.2 Report all LCS's associated with any daily batch which contains any or all of the project samples.

14.4.2 Designated MS/MSD for standard level II report.

14.4.2.1 Report only project specific MS/MSD.

14.4.2.2 If the MS/MSD passes, report only the MS/MSD as the daily batch QC.

14.4.2.3 If the MS/MSD contains any failing recoveries, report the MS/MSD and the acceptable associated LCS as the daily batch QC when the MS/MSD fails and the LCS is reported, provide a written explanation in a brief case narrative.

14.4.3 Batch MS/MSD for project which requires LCS's to be reported.

14.4.3.1 Report the MS/MSD for the first daily batch that includes the samples from the project being reported.

14.4.3.2 Report all LCS's associated with any daily batch which contains any or all of the project samples.

14.4.4 Batch MS/MSD for standard level II project.

14.4.4.1 Report the MS/MSD for the first daily batch that includes the samples from the project being reported.

14.4.4.2 If the MS/MSD passes then only report the MS/MSD as the daily batch QC.

14.4.4.3 If the MS/MSD contains any failing recoveries, then report the MS/MSD and the acceptable associated LCS as the daily batch QC (when the MS/MSD fails and the LCS is reported, provide a written explanation in a brief case narrative.

14.4.5 The laboratory will analyze an LCS with all MS/MSD's.

14.4.6 Level III QC will also follow the same requirements as a level II reportables except that the LCS is not reported unless specifically requested and:

14.4.6.1 Report only project specific MS/MSD.

14.4.6.2 If the MS/MSD passes then only report the MS/MSD as the QC.

14.4.6.3 If the MS/MSD contains any failing recoveries it will be reported and an LCS must be analyzed and pass to show the instrument is in control (when an LCS is analyzed and is not reported, however, it must be explained in the case narrative that the MS/MSD contained failing recoveries and that an LCS was analyzed and passed; therefore, showing that the system is in control)

15.0 SUPPLEMENTAL DOCUMENTS

15.1 SOP for Maintenance Logbook, IEA Doc# QAS01100.NC.

15.2 SOP for Conducting MDL Studies, IEA Doc# QAS02000.NET.

15.3 SOP for Chain-of-Custody, IEA Doc# QAS01301.NC.

16.0 REFERENCES

16.1 Test Methods for Evaluating Solid Watse, 3rd Edition, July 1992 Revision, Method 8260A.

16.2 Code of Federal Regulations, Part 136, Appendix A, Method 624-Purgeables, July 1, 1991 Revision.

16.3 USEPA CLP Statement of Work for Organics Analysis, OLM03.1

17.0 SUBSTANTIVE REVISIONS

17.1 Section 2.3. added. Section 13.3.1 revised to correct the acceptance window for the internal standard area recovery.

TABLE 1

TARGET COMPOUNDS FOR 8260A VOLATILE ANALYSIS

ANALYTE	QUANTITATION ION	SECONDARY ION
Acetone	43	58
Acrylonitrile	53	52, 51
Allyl Chloride	76	41, 39, 78
Benzene	78	
Bromobenzene	156	77, 158
Bromochloromethane	128	49, 130
Bromodichloromethane	83	85, 127
Bromoform	173	175, 254
Bromomethane	94	96
2-Butanone	43	72
n-Butylbenzene	91	92, 134
tert-Butylbenzene	119	91, 134
sec-Butylbenzene	105	134
Carbon Disulfide	76	78
Carbon Tetrachloride	117	119
Chlorobenzene	112	77, 114
Chlorodibromomethane	129	208, 206
Chloroethane	64	66
2-Chloroethylvinyl Ether	63	65, 106
Chloroform	83	85
Chloromethane	50	52
2-Chlorotoluene	91	126
4-Chlorotoluene	91	126
1,2-Dibromo-3-Chloropropane	75	155, 157
1,2-Dibromoethane	107	109, 188
Dibromomethane	93	95, 174
1,2-Dichlorobenzene	146	111, 148
1,3-Dichlorobenzene	146	111, 148
1,4-Dichlorobenzene	146	111, 148
Dichlorodifluoromethane	85	87
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
1,3-Dichloropropane	76	78

TABLE 1 (Continued)

TARGET COMPOUNDS FOR 8260A VOLATILE ANALYSIS

ANALYTE	QUANTITATION ION	SECONDARY ION
2,2-Dichloropropane	77	97
1,1-Dichloropropene	75	110, 77
cis-1,3-Dichloropropene	75	77, 39
trans-1,3-Dichloropropene	75	77, 39
cis-1,4-Dichloro-2-Butene	75	53, 77, 124
trans-1,4-Dichloro-2-Butene	53	88, 75
Ethyl Benzene	91	106
Ethyl Methacrylate	69	41, 99, 86
Hexachlorobutadiene	225	223, 27
2-Hexanone	43	58, 100
Iodomethane	142	127, 141
Isopropylbenzene	105	120
p-Isopropyltoluene	119	134, 91
Methacrylonitrile	41	67, 39, 52
Methylene Chloride	84	86, 49
Methyl Methacrylate	69	41, 100, 39
4-Methyl-2-Pentanone	43	58, 100, 85
Methyl-tert-Butyl Ether	73	57
Naphthalene	128	
Pentachloroethane	167	130, 169
n-Propylbenzene	91	120
Styrene	104	78
1,2,3-Trichlorobenzene	180	182, 145
1,2,4-Trichlorobenzene	180	182, 145
1,1,1,2-Tetrachloroethane	131	133, 119
1,1,2,2-Tetrachloroethane	83	131, 85
Tetrachloroethene	164	129, 166
Toluene	92	91
1,1,1-Trichloroethane	97	99, 61
1,1,2-Trichloroethane	83	97, 85
Trichloroethene	95	130, 132
Trichlorofluoromethane	101	103
1,2,3-Trichloropropane	75	77
1,2,4-Trimethylbenzene	105	120
1,3,5-Trimethylbenzene	105	120
Vinyl Acetate	43	86
Vinyl Chloride	62	64

TABLE 1 (Continued)

TARGET COMPOUNDS FOR 8260A VOLATILE ANALYSIS

ANALYTE	QUANTITATION ION	SECONDARY ION
o-Xylene	106	91
m,p-Xylene	106	91
INTERNAL STANDARDS		
1,4-Difluorobenzene	114	
Chlorobenzene-d5	117	
1,4-Dichlorobenzene-d4	152	115, 150
Pentafluorobenzene	168	
SURROGATES		
4-Bromofluorobenzene	95	174, 176
1,2-Dichloroethane-d4	65	102
Toluene-d8	98	100

TABLE 2

PRACTICAL QUANTITATION LIMITS FOR 8260A VOLATILE ANALYSIS

ANALYTE	PRACTICAL QUANTITATION LIMIT (PQL)(ug/L)
Acetone	5
Acrylonitrile	1
Allyl Chloride	1
Benzene	1
Bromobenzene	1
Bromochloromethane	1
Bromodichloromethane	1
Bromoform	1
Bromomethane	1
2-Butanone	5
n-Butylbenzene	1
tert-Butylbenzene	1
sec-Butylbenzene	1
Carbon Disulfide	1
Carbon Tetrachloride	1
Chlorobenzene	1
Chlorodibromomethane	1
Chloroethane	1
2-Chloroethylvinyl Ether	1
Chloroform	1
Chloromethane	1
2-Chlorotoluene	1
4-Chlorotoluene	1
1,2-Dibromo-3-Chloropropane	1
1,2-Dibromoethane	1
Dibromomethane	1
1,2-Dichlorobenzene	1
1,3-Dichlorobenzene	1
1,4-Dichlorobenzene	1
Dichlorodifluoromethane	1
1,1-Dichloroethane	1
1,2-Dichloroethane	1
1,1-Dichloroethene	1
cis-1,2-Dichloroethene	1
trans-1,2-Dichloroethene	1
1,2-Dichloropropane	1
1,3-Dichloropropane	1

TABLE 2 (Continued)

PRACTICAL QUANTITATION LIMITS FOR 8260A VOLATILE ANALYSIS

ANALYTE	PRACTICAL QUANTITATION LIMIT (PQL)(ug/L)
2,2-Dichloropropane	1
1,1-Dichloropropene	1
cis-1,3-Dichloropropene	1
trans-1,3-Dichloropropene	1
cis-1,4-Dichloro-2-Butene	1
trans-1,4-Dichloro-2-Butene	1
Ethyl Benzene	1
Ethyl Methacrylate	1
Hexachlorobutadiene	1
2-Hexanone	5
Iodomethane	1
Isopropylbenzene	1
p-Isopropyltoluene	1
Methacrylonitrile	1
Methylene Chloride	1
Methyl Methacrylate	1
4-Methyl-2-Pentanone	5
Methyl-tert-Butyl Ether	1
Naphthalene	1
Pentachloroethane	1
n-Propylbenzene	1
Styrene	1
1,2,3-Trichlorobenzene	1
1,2,4-Trichlorobenzene	1
1,1,1,2-Tetrachloroethane	1
1,1,2,2-Tetrachloroethane	1
Tetrachloroethane	1
Toluene	1
1,1,1-Trichloroethane	1
1,1,2-Trichloroethane	1
Trichloroethene	1
Trichlorofluoromethane	1
1,2,3-Trichloropropane	1
1,2,4-Trimethylbenzene	1
1,3,5-Trimethylbenzene	1
Vinyl Acetate	1
Vinyl Chloride	1

TABLE 2 (Continued)

PRACTICAL QUANTITATION LIMITS FOR 8260A VOLATILE ANALYSIS

ANALYTE	PRACTICAL QUANTITATION LIMIT (PQL)(ug/L)
o-Xylene	1
m,p-Xylene	2
Xylenes (Total)	5

Attachment I

Method: 8240 CLP 624 Other:____

GC/MS Volatile Runlog

Instrument-MSD*

Matrix: Water/Med Soil

EM: _____

Water Curve:_____ Soil Curve:_____

Batch: _____

Filename	IEA Sample ID.	Case #	Date	Time	ALS#	Init/Final Vol.	DF	Dilution Preparation/Comment	IS	Surr	Analyst	Rerun

Tunes:__/___ Surr Fail:___ IS Fail:___ Tot Inj:___ Suc Inj:___ Billables:___

Secondary Review By:_____ Date:_____

Attachment II

PROJECT STATUS REPORT

Date_____

Project ID_____ Client_____

Lab Due Date_____ Project Due Date_____

Initiated By_____ Department_____

Parameter_____ Sample_____

☐ Late Notification

Status_____

Reason_____

Expected Completion_____

☐ Matrix Discrepancy ☐ Method Discrepancy

Problem_____

Resolution_____

☐ Protocol Exceeded

Date Sampled_____ Date Protocol Expired_____

Date Analyzed_____ Reason_____

CLIENT NOTIFICATION

_____

_____

_____

_____

_____

_____

Attachment III

IEA Corporation Internal Chain of Custody

Login By: Date Received: Time Received:

Workorder # Case # Department Location

Storage Return			Removed To/Reason								
Date	Time	By	Date	Time	Frac	Location	Code	By	Frac	Matrix	Sample ID

Applicable Codes:

AN=Analyze	LI=Login	DG=Digestion	AT=Autosampler
TR=Transfer	SC=Screen	EX=Extraction	
DI=Dispose	ST=Storage	DW=Dry Weight	

Verified by: Date:

Attachment IV

Volatiles Screening Logbook
Instrument- PID/Hall #1

Date	ALS#	IEA #	Matrix	Analyst	Test Code	DF	Final DF

Secondary Review By:_______________ Date:__________

IEA, Inc. Form# GCL00601.NC

IEA Logbook# GC20*

Volatiles Screening Logbook
Instrument- FID/Hall #1

Date	ALS#	IEA #	Matrix	Analyst	Test Code	DF	Final DF

Secondary Review By:________________ Date:__________

Attachment V

MAINTENANCE LOG BOOK

DATE_____

INSTRUMENT TYPE:_____ INSTRUMENT ID:_____	SERVICE TYPE ☐ DAILY ☐ ROUTINE ☐ REPAIR

SERVICE SUMMARY

DAILY (Preventive Maintenance performed on a daily basis)

Service Performed by:_____

ROUTINE (Scheduled or needed service, minor or routine)

Service Performed by:_____

REPAIR (Instrument inoperable or does not meet manufacturers specifications for acceptable operation)

Service Performed by:_____

Secondary Review By:_____Date:_____

Attachment VI

STANDARDS PREPARATION LOGBOOK

STOCK STANDARD PREPARATION

Lot Code #: _____

Solvent Used: _____

Prepared By: _____

Date Prepared: _____

Expiration Date: _____

Stock Name	Vendor	Stock Lot # / Lot Code #	Logbook / Page #	Stock Conc (ug/mL)
1.				
2.				
3.				
4.				
5.				
6.				
7.				
8.				
9.				
10.				
11.				
12.				

WORKING STANDARD PREPARATION

Standard	Amount Used (uL)	Final Volume (mLs)	Final Conc (ug/mL)	Prepared By	Date Prepared	Date Expires
1.						
2.						
3.						
4.						
5.						
6.						
7.						
8.						
9.						
10.						
11.						
12.						

Comments: _____

_____

Secondary Review By: _____ Date: _____

Attachment VII

Stock Standards Receiving Log
GC/GCMS Laboratory

Date Received	Analyst	Standard Name	Stock/Standard Code Lot #	Vendor	Expiration Date	Comments

Secondary Review By:_____ Date:_____

IEA, Inc. Form# QAL01200.NC

IEA Logbook# QA*

8. IEA SOP FOR ZERO HEADSPACE VOC EXTRACTION

1.0 APPROVALS

The signature of the following individuals indicates that this SOP is complete and meets the requirements specified in corporate Document #QAS00200.NET. In addition, it also signifies that the content meets the specifications of the referenced "Test Code".

Laboratory Director _____

Quality Assurance Manager _____

2.0 SCOPE AND APPLICATION

2.1 The purpose of this Standard Operating Procedure is to outline the specific methods to prepare Volatile samples for ZHE analysis.

2.2 The following test codes is used to define analyses conducted in accordance with all of the conditions set forth by this SOP:

LCH_05

3.0 SUMMARY OF METHOD

A known amount of sample is leached using ZHE headspace extraction in order to analyze the sample as a volatile liquid and not as a raw solid sample.

4.0 INTERFERENCES

4.1 Interferences may occur through contamination of vessels, syringes or glassware. These are kept to a minimum by assaying prepped ZHE units prior to usage.

5.0 SAFETY

5.1 Chemical resistant polyvinyl gloves should be worn at all times when working with solvents.

5.2 Each analyst has been issued a personal lab coat and it should be worn at all times in the sample preparation laboratory area.

5.3 MSDS sheets are available in the lab area for immediate reference in the case of an emergency.

5.4 Vapor monitors are used monthly to determine exposure of solvents to employees.

6.0 SAMPLE CONTAINERS, COLLECTION AND PRESERVATION

6.1 Sample containers should be glass walled with teflon sealed lids to prevent exposure.

7.0 APPARATUS AND MATERIALS

7.1 Analytical balance - Toploading, accurate to +/- 0.01g

7.2 Hazardous Waste Filtration Unit - Millipore Catalog #YT30142HW or equivalent

7.3 Nitrogen hose

7.4 9.0 cm Glass Filter - Whatman GF/F Filter, Baxter Catalog #F2834-090 or equivalent

7.5 Rotator Unit - Capable of rotation in end-over-end fashion @ 30 +/-2 rpm

7.6 250 ml Glass Beaker

7.7 1000 ml Graduated Cylinder

7.8 Metal Spatula

7.9 Peristaltic Pump

7.10 Teflon Tubing - 1/8" diameter

7.11 40 ml vials - Capped with Teflon - lined Septum

7.12 Tedlar Bag = I1, SKC Catalog #7450 or equivalent

7.13 Syringe - 50 ml Glass Gas Tight, CPI Catalog #7450 or equivalent

7.14 15.0 cm Glass Fiber Filter - Whatman GF/F Filter, Baxter Catalog #7450 or equivalent

7.15 Drying Oven

8.0 REAGENTS AND STANDARD PREPARATION

8.1 Reagent water. Water free of any contamination

8.2 Hydrochloric Acid (1N), made from ACS reagent grade

8.3 Nitric Acid (1N), made from ACS reagent grade

8.4 Sodium Hydroxide (1N), made from ACS reagent grade

8.5 Glacial Acetic Acid, ACS reagent grade

8.6 Extraction fluid #1: Add 5.7 ml glacial HOac with reagent water to a volume of 1 liter

9.0 CALIBRATION

9.1 Analytical balance is calibrated daily to + or - 0.01g

9.2 pH meter is calibrated prior to use using pH or 4,7 and 10

10.0 QUALITY CONTROL

10.1 A method blank is done with each batch of samples

10.2 Extraction vessels are cleaned and tested for contamination prior to use

11.0 SAMPLE PREPARATION AND INSTRUMENTAL PROCEDURES

11.1 Characterization of Sample

11.2 If sample is entirely solid with no obvious liquid consistency:

11.2.1 Proceed to Section 11.5;

11.3 If sample is sludge: Determine the percent solids by using the following method:

11.3.1 Shake the sample container to assure homogenization of the sample;

11.3.2 Weigh a minimum of 100 gram sludge sample into a clean beaker;

11.3.3 Filter the sample (refer to Section 11.7: Preparation of Millipore Unit)

11.3.3.1 Consider the filtration process complete when all liquid has been removed from the sludge or filtration is stopped totally; Dispose of the liquid portion appropriately

11.3.4 Remove the solid plus filter paper from the filter unit and place in a drying oven for 3-4 hours;

11.3.5 Remove the filter with the solid sample from the oven:

11.3.5.1 Determine the weight of the dried solid; Use the weight and initial sludge weight to determine the % solids;

11.3.5.2 Discard the filter and the dried sample;

11.3.5.3 Use the % solids to determine the correct amount of extraction fluid needed to charge the ZHE unit

11.3.6 Proceed to Section 11.6 if sample is all liquid (<0.5% solids)

11.4 **Prefiltering a Sludge Sample:**

11.4.1 Weigh a 25 gram dry weight sludge sample into the ZHE unit;

11.4.2 Place the filter cap into the unit and bolt the lid. Attach the tedlar bag to the ZHE unit using the teflon tubing;

11.4.3 Attach the nitrogen line to the ZHE unit. Pressurize the unit to force the filterable liquid into the tedlar bag;

11.4.4 Detach the tedlar bag, label appropriately and place in storage at 4_oC

11.5 Charging the ZHE Unit:

11.5.1 If sample is all solid: Weigh a 25 gram sample into a beaker;

11.5.2 If particle size is >3/8": crush to a size that will allow passage through a 9.5 mm sieve:

11.5.2.1 Place filters in the ZHE unit and hold the lid tightly;

11.5.2.2 Attach the nitrogen line and pressurize the ZHE unit to 1-10 psi

11.5.3 Attach nitrogen line to the unit containing the remaining non-filterable sludge; pressurize the unit to 1-10 psi

11.5.4 Add extraction fluid to the ZHE vessel using a peristaltic pump. (Refer to Section 12 for equations necessary for determining the proper amount of extraction fluid;

11.5.4.1 When adding fluid: open the valve located on the base of the ZHE unit, to release the pressure and allow the ZHE to fill with fluid;

11.5.4.2 Once fluid stops flowing into the ZHE: close the bottom valve and the line from the pump;

11.5.4.3 Invert the unit 3 times to bring unwanted air bubbles to the top; release the top valve to expel air and eliminate the bubbles;

11.5.4.4 Re-attach the pump line and fill the remaining space left by expelling the air in the line

11.5.5 Pressurize the unit again to 1-10 psi;

11.5.6 Place the unit in a rotator and allow to revolve for 18 hours

11.6 Extracting Liquid Samples (<0.5% Solids):

11.6.1 Fill extraction unit with liquid;

11.6.2 Place filters into unit securely and bolt the lid;

11.6.3 Open the top valve; attach the nitrogen line and pressurize the unit by expelling air into the unit;

11.6.4 Place a 50 ml syringe on the top valve once liquid begins to flow from that valve; collect extract in the syringe;

11.6.5 Place extract into a 40 ml vial and leave zero headspace; collet 3 vials of the extract; proceed to Section 11.8.3

11.7 Preparation of the Millipore Unit for % Solid Determination:

11.7.1 Disassemble the unit into 3 parts (cover, canister and base):

11.7.1.1 Rinse each part thoroughly with de-ionized water;

11.7.1.2 Rinse with 10% nitric acid;

11.7.1.3 Repeat the rinsing method using de-ionized water

11.7.2 Rinse the filter paper with 10% nitric acid followed by a second rinse with de-ionized water.

11.7.2.1 Place the filter paper on the base of the filter unit and center it over the metal screen;

11.7.2.2 Place the canister evenly over the filter paper;

11.7.2.3 Using the 3 teflon bolts: secure the cover/canister onto the base of the unit

11.8 <u>Filtration of Solid Sludge Samples:</u>

11.8.1 If sample is all solid:

11.8.1.1 Attach the nitrogen line to the unit;

11.8.1.2 Connect a 50 ml syringe to the top of the unit and pressurize;

11.8.1.3 Collect the syringe into the syringe; transfer extract to a 40 ml vial and leave zero headspace;

11.8.1.4 Collect 3 vials of the extract and proceed to Section 11.5

11.8.2 If sample is a sludge:

11.8.2.1 Attach the nitrogen line to the unit;

11.8.2.2 Connect the tedlar bag to the top of the unit and pressurize:

11.8.2.2.1 If pre-filter and extract are homogenous: filter the full amount of extract into the bag; homogenize contents of the bag and fill three 40 ml vials, leaving zero headspace;

11.8.2.2.2 If pre-filtrate and extract are heterogeneous: remove the tedlar bag from the unit; collect extract using 50 ml syringe; place extract in a 40 ml vial and leave zero headspace. Collect three 40 ml vials, leaving zero headspace. Transfer contents of the tedlar bag into three 40 ml vials and leave zero headspace; label extracts vials: Extract. Label pre-filtrate vials: Pre-filtrate. Consider samples as separate for Sample Management login

11.8.3 Label vials with sample ID and complete the extraction sheet. Deliver bottles along with the Chain-of-Custody form to the Sample Management Department

12.0 CALCULATIONS

12.1.1 Characterization of the Samples:

Equation 1:

$$\% \text{ Solids} = \frac{\text{Dried Sample (g)} \times 100\%}{\text{Wet Sample (g)}}$$

12.1.2 Charging the ZHE units:

Equation 2:

$$\text{Extraction Fluid Added (mls)} = \text{Dried Solids (g)} \times 20 \frac{\text{mls}}{\text{g}}$$

13.0 ACCEPTANCE OF DATA

13.1 Method blanks must not contain target analytes exceeding the MDL for that specified analyte. Some exceptions may be made for common lab solvents such as methylene chloride. The supervisor of the department must be consulted whenever such conditions exist.

14.0 REPORTING OF RESULTS

Not applicable to this procedure.

15.0 SUPPLEMENTAL DOCUMENTS

15.1 SOP on assaying ZHE apparatus

16.0 REFERENCES

16.1 Federal Register, Vol. 55, No. 61, March 29, 1990.

17.0 SUBSTANTIVE REVISIONS

Original issue.

9. IEA SOP FOR METHOD 3550 SVOC SOLID MEDIA EXTRACTION

1.0 APPROVALS

The signatures of the following individuals indicate that this SOP is complete and meets the requirements specified in corporate document # QAS00200.NET. In addition, it signifies that the content meets the specifications of the referenced "Test Code".

Laboratory Director

Quality Assurance Manager

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PROPRIETARY DOCUMENT

2.0 SCOPE AND APPLICATION

2.1 The objective of this document is to outline the extraction methods necessary to prepare BNA soil samples for GC/MS analysis. The extraction method followed is 3550.

2.2 The following test codes are used to define analyses conducted in accordance with all of the conditions set forth by this SOP:

SVE_02 SVE_04

2.3 The document control number for this SOP shall be **SPS01301.NC**

3.0 SUMMARY OF METHOD

3.1 A known amount of sample is extracted using sonication. The extract is concentrated using a steambath and nitrogen evaporation to a known final volume and delivered to the GC/MS department for analysis.

4.0 INTERFERENCES

4.1 Interferences are kept to a minimum by baking the glassware to be used at 900F and prerinsing the glassware with methylene chloride.

5.0 SAFETY

5.1 Be sure to always wear chemical resistant gloves and eye protection at all times. In addition, each analyst has been issued a personal lab coat and it should be worn at all times while in the laboratory area.

5.2 MSDS sheets are available in the laboratory for immediate reference in case of an emergency.

5.3 Vapor monitors are used quarterly to determine exposure of employees to solvents.

6.0 SAMPLE CONTAINERS, COLLECTION AND PRESERVATION

6.1 Sample Containers - see appropriate analytical SOP.

6.2 Sample Collection - see appropriate analytical SOP.

6.3 Sample Preservation - see appropriate analytical SOP.

6.4 Sample Holding time is 14 days from sampling.

7.0 APPARATUS AND MATERIALS

7.1 250ml beaker

7.2 100ml graduated cylinder

7.3 75mm funnel

7.4 500ml K-D flask

7.5 10ml concentrator tube

7.6 Three ball snyder column

7.7 Aluminum drying pan

7.8 Whatman 41 filter paper

7.9 Keck clamps

7.10 Silicon Carbide boiling chips

7.11 Metal Spatula

7.12 Top loading balance, accurate to +/- 0.01g

7.13 5 3/4" Pastuer pipets

7.14 Drying Oven

7.15 Nitrogen Evaporator

7.16 Tekmar Sonic Disrupter

7.17 500ul gastight syringe

7.18 2.0 mL amber vial

8.0 REAGENTS AND STANDARD PREPARATION

8.1 Methylene Chloride - Baxter High Purity Solvent

8.2 Powdered Sodium Sulfate

8.3 Acetone - Baxter High Purity Solvent

8.4 BNA Surrogate Solution prepared by GC/MS staff

8.5 BNA QC Check Solution prepared by GC/MS staff

8.6 BNA Matrix Spiking Solution prepared by GC/MS staff

8.7 Surrogate Standard Preparation

8.7.1 Using a 500 uL gastight syringe, combine 500 uL of MeCl2 and 500 uL of BNA surrogate solution a 2.0 mL amber vial.

9.0 CALIBRATION

9.1 Top loader balance is calibrated daily to +/- 0.01g.

10.0 QUALITY CONTROL

10.1 All equipment to be used is rinsed with methylene chloride.

10.2 Batch QC

10.2.1 A method blank will be extracted daily with each group of up to 20 samples.

10.2.2 A blank spike, matrix spike, and matrix spike duplicate will be extracted daily with each group of up to 20 samples. The MS/MSD samples are chosen from the samples extracted that day.

11.0 SAMPLE PREPARATION AND INSTRUMENTAL PROCEDURES

11.1 Prerinse all glassware and sonicator tips with methylene chloride.

11.2 Dry weight determination

11.2.1 Weigh an aluminum pan and record the weight in the dry weight notebook.

11.2.2 Weigh approximately 3g of the soil sample into the aluminum pan and record the weight in the dry weight notebook.

11.2.3 Place pan with sample into a drying oven set at 100C for at least 2 hours.

11.2.4 Reweigh pan with sample and record in the dry weight notebook.

11.3 Sample Extraction

11.3.1 Place a labeled 250ml beaker onto the balance, tare the balance, weigh out approximately 30g of the sample into the beaker and record the weight on the extraction sheet.

11.3.2 Mix in 60g of powdered sodium sulfate with the sample and stir to homogenize. For the blank weigh out 60g of sodium sulfate only.

11.3.3 Add 0.5ml of BNA surrrogate to all samples. Add 1.0ml of QC Spike to blank spike. Add 0.5ml of matrix spike solution to matrix spike and matrix spike duplicate samples.

11.3.4 Add 100ml of 50/50 methylene chloride/acetone to the sample and sonicate for 90 seconds with sonicator on pulse mode with output setting at 10 (maximum) and a duty cycle set at 50%.

11.3.5 Pour extract through funnel containing filter paper and 5g sodium sulfate into the K-D setup.

11.3.6 Repeat steps 11.3.4 through 11.3.5 twice more.

11.3.7 Rinse funnel down with 5ml of methylene chloride.

11.4 Sample Concentration

11.4.1 Remove funnel and place snyder column on K-D setup containing 2 carbide boiling chips.

11.4.2 Place K-D setup on steambath until approximately 3ml remain in the concentrator tube. Remove at this point and let cool for 10 minutes.

11.4.3 Disassemble K-D setup and place concentrator tube on N-Evap until final volume is at 1ml.

11.4.4 Transfer extract into a 2ml amber vial and label with parameter, sample number, extraction date, and final volume. Deliver this with completed extraction sheet to GC/MS department for screening and analysis.

12.0 CALCULATIONS

12.1 Dry Weight Calculations

* %moisture =(dried sample weight - pan weight)/ wet sample weight x 100

* Sample dry weight = wet sample weight x % solid of sample

13.0 ACCEPTANCE OF DATA

13.1 Method blanks must not contain target analytes exceeding MDL for that specific analyte.

13.2 Surrogates and spikes must meet criteria set forth by analytical SOP's in GC/MS dept.

14.0 REPORTING OF RESULTS

N/A

15.0 SUPPLEMENTAL DOCUMENTS

N/A

16.0 REFERENCES

16.1 USEPA SW-846, 3rd Edition, Method 3550.

16.2 USEPA 40 CFR Part 136: Guidelines Establishing Test Procedures for the Analysis of Pollutants Under the Clean Water Act, October 1984, Method 625.

16.3 USEPA SW-846, 3rd. Edition, Method 8270.

17.0 SUBSTANTIVE REVISIONS

17.1 Section 2.3 amended: Document control number changed to SPS01301.NC to reflect SOP revision. Section 5.3 amended: Monitoring changed from monthly to quarterly. Section 7.17 amdended: 1000 uL gastight syringe changed to 500 uL. Addendum: Section 7.18: 2.0 mL amber vial. Section 8.1 amended: Methylene chloride grade

changed from Fisher Optima Grade to Baxter High Purity Solvent. Section 8.3 amended: Acetone grade changed from Fisher Optima Grade to Baxter High Purity Solvent. Addendum: Section 8.7 added to include surrogate standard preparation. Addendum: Section 8.7.1 added to detail surrogate standard preparation. Addendum to Section 10.2.2: Explains how MS/MSD samples are chosen. Amendment to 11.3.6: Repeated steps changed from 11.3.3 through 11.3.4 to 11.3.4 through 11.3.5. Amendment to 11.4.2: 1 mL changed to 3 mL. Amendment to Section 11.4.4: 2 mL vial changed to 2 mL amber vial. Addendum to Section 11.4.4: Delivery of extraction sheet to GC/MS department added. Section 11.4.5, detailing delivery of extracts to GC/MS department omitted. Section 12.1 amended: Parentheses added around dried sample weight - pan weight to clarify equation. Addendum to Section 13.2: Specifies SOP's in GC\MS department.

10. IEA SOP FOR METHOD 3520 SVOC LIQUID MEDIA EXTRACTION

1.0 APPROVALS

The signatures of the following individuals indicate that this SOP is complete and meets the requirements specified in corporate document # QAS00200.NET. In addition, it signifies that the content meets the specifications of the referenced "Test Code".

Laboratory Director

Quality Assurance Manager

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PROPRIETARY DOCUMENT

2.0 SCOPE AND APPLICATION

2.1 The objective of this document is to outline the extraction methods necessary to prepare BNA water samples for GC/MS analysis. The extraction method followed is 3520.

2.2 The following test codes are used to define analyses conducted in accordance with all of the conditions set forth by this SOP:

SVE_01 SVE_05 SVE_07 SVE_09 SVE_13

2.3 The document control number for this SOP shall be **SPS01401.NC**

3.0 SUMMARY OF METHOD

3.1 A known amount of sample is extracted using method 3520 (continuous liquid/liquid extraction) and 3510 (separatory funnel). The extract is concentrated using a steambath and nitrogen evaporation to a known final volume and delivered to the GC/MS department for analysis.

4.0 INTERFERENCES

4.1 Interferences are kept to a minimum by baking the glassware to be used at 900F and prerinsing the glassware with methylene chloride.

5.0 SAFETY

5.1 Be sure to always wear chemical resistant gloves and eye protection at all times. In addition, each analyst has been issued a personal lab coat and it should be worn at all times while in the laboratory area.

5.2 MSDS sheets are available in the laboratory for immediate reference in case of an emergency.

5.3 Vapor monitors are used quarterly to determine exposure of employees to solvents.

6.0 SAMPLE CONTAINERS, COLLECTION AND PRESERVATION

6.1 Sample Containers - see appropriate analytical SOP.

6.2 Sample Collection - see appropriate analytical SOP.

6.3 Sample Preservation - see appropriate analytical SOP.

6.4 Sample Holding time is 7 days from sampling.

7.0 APPARATUS AND MATERIALS

7.1 Continuous extractor

7.2 500 mL Round Bottom Flask

7.3 Drying column

7.4 500 mL K-D flask

7.5 10 mL concentrator tube

7.6 Three ball snyder column

7.7 Glasswool

7.8 Keck clamps

7.9 Silicon Carbide boiling chips

7.10 5 3/4" Pasteur pipets

7.11 Nitrogen Evaporator

7.12 1000 uL gastight syringe

7.13 500 uL gastight syringe

7.14 2.0 mL amber vials

7.15 2 L separatory funnels

8.0 REAGENTS AND STANDARD PREPARATION

8.1 Methylene Chloride - Baxter High Purity Solvent

8.2 Granular Sodium Sulfate

8.3 10 N NaOH - See SOP Doc# SPS02300.NC

8.4 1:1 Sulfuric Acid/Deionized Water : 500 mL of conc. Sulfuric acid added to 500 mL of deionized water.

8.5 Surrogate - prepared by GCMS dept.

8.6 MS/MSD Spiking Solution - prepared by GCMS dept.

8.7 TCLP Spiking Solution - prepared by GCMS dept.

8.8 QC Check Solution - prepared by GCMS dept.

9.0 CALIBRATION

Not Applicable

10.0 QUALITY CONTROL

10.1 All equipment to be used is rinsed with methylene chloride.

10.2 Batch QC

There will be one method blank,QC Check, matrix spike, and matrix spike duplicate for every batch of up to 20 samples daily.

11.0 SAMPLE PREPARATION AND INSTRUMENTAL PROCEDURES

11.1 Extraction for Test Codes SVE_01

11.1.1 Prerinse all glassware with Methylene Chloride.

11.1.2 Pour approximately 300 mL of methylene chloride into a 500 mL round bottom flask and add several silicon carbide boiling chips.

11.1.3 Attach continuous extractor to the round bottom and pour approximately 200 mL of methylene chloride into the extractor.

11.1.4 To the extractor, add 1 liter of sample for SVE_01. The QC samples associated with this test code should be treated in the same manner. Use 1 liter of methylene chloride extracted DI water for method blank and blank QC samples. Use methylene chloride extracted water to bring all sample volumes up to 1 liter.

11.1.5 Add appropriate surrogates and spikes (See Table 1).

11.1.6 Basify samples using 10 N NaOH. Check pH with wide range pH paper.

11.1.7 Place continuous extractor on condenser and set heating element to 30% power. Make sure boiling ships are causing boiling effect in round bottom and methylene chloride is flowing from extractor into round bottom.

11.1.8 Let sample extract for 18-24 hours. Turn power off and let heating element cool down.

11.1.9 Remove extractor from condenser.

11.1.10 Remove round bottom flask from extractor and replace with another round bottom containing approximately 200 mL of methylene chloride.

11.1.11 Acidify to pH <2 with 1:1 sulfuric acid/DI water. Set back on condenser and heating element. Set power to 30% and extract for 18-24 hours.

11.1.12 After sample has extracted for 18-24 hours on acid side and heating element has cooled down, remove from condenser and check final pH. If pH is >2, make note of it on extraction sheet.

11.1.13 After bringing the majority of the methylene chloride over to the round bottom from the extractor remove the round bottom flask from continuous and dispose of water portion and leftover methylene chloride in appropriate waste containers.

11.1.14 Go to 11.5.

11.2 Extraction for SVE_05 test code.

11.2.1 Pre-rinse all glassware with methylene chloride.

11.2.2 Pour approximately 300 mL of methylene chloride into a 500 mL round bottom flask and add several silicon carbide boiling chips.

11.2.3 Attach continuous extractor to the round bottom and pour approximately 200 mL of methylene chloride into extractor.

11.2.4 To the extractor, add 1 liter of sample for all samples and MS/MSD's. Add 1 liter of methylene chloride extracted DI water for blank and QC check. Use methylene chloride extracted water to bring all sample volumes to 1 liter.

11.2.5 Add appropriate surrogates and spikes. See Table 1.

11.2.6 Acidify to pH <2 with 1:1 sulfuric acid/DI water. Set extractor on condenser and set heating element to 30% power. Make sure boiling chips are causing boiling effect in round bottom and methylene chloride is flowing from extractor into round bottom.

11.2.7 Let sample extract for 18-24 hours. Turn power off and let heating element cool down.

11.2.8 Remove extractor from condenser and check final pH. If pH >2, make note of it on the extraction sheet.

11.2.9 After bringing the majority of the methylene chloride over to the round bottom from the extractor remove round bottom flask from continuous and dispose of water portion and leftover methylene chloride in appropriate waste containers.

11.2.10 Go to 11.5.

11.3 Extraction for SVE_07, SVE_09, and SVE_13 test codes.

11.3.1 Pre-rinse all glassware with methylene chloride.

11.3.2 To a 2 L separatory funnel add 500 mL of sample. Use 1 L of methylene chloride treated DI water for the blank and blank QC. For regulatory TCLP(SVE_07) the spikes are treated like the samples. See Table 1 for spiking amounts.

11.3.3 Using 10 N NaOH basify all samples, blank and blank QC to a pH > 11. Check pH with wide range pH paper.

11.3.4 Add 60 mL of methylene chloride to all separatory funnels. Shake for about 10 seconds and vent funnel.

11.3.5 Place funnels on auto-shaker and shake well for 2 minutes. Sample should almost reach stopcock while shaking. After they have finished let the solvent and the sample separate(~5 minutes). Drain the solvent layer through a drying column containing a glasswool plug and ~1 1/2" of granular sodium sulfate into a round bottom.

11.3.6 Repeat 11.3.4 and 11.3.5 two more times.

11.3.7 After shaking the funnels 3 times use 1:1 Sulfiric Acid/DI Water to change the pH to <2. Check pH with wide range pH paper.

11.3.8 Follow steps 11.3.4 through 11.3.6 using new round bottoms to collect the solvent.

11.4 Sample QC Spikes

TABLE 1
Spiking Chart

Test Code	BNA Surrogate	QC Check	MS/MSD Spike	BNA TCLP Spike
SVE_05	1L = 0.5ml	1L = 1.0ml	1L = 0.5ml	N/A
SVE_01	1L = 0.5ml	1L = 1.0ml	1L = 0.5ml	N/A
SVE_07	500ml=0.25ml	1L = 1.0ml	1L = 0.5ml	500ml = 0.25ml
SVE_09	500ml=0.25ml	1L = 1.0ml	1L = 0.5ml	N/A
SVE_13	500ml=0.25ml	1L = 1.0ml	1L = 0.5ml	N/A

*for samples with less volume than listed, lower amount spiked by the ratio.

11.5 Samples Concentration

11.5.1 Pour extract from round bottom through drying column containing a glasswool plug and 1 1/2" granular sodium sulfate into a K-D setup.

11.5.2 Remove drying column and place snyder column on K-D setup containing 2 carbide boiling chips.

11.5.3 Place K-D setup on steambath until approximately 4 mL remains in the concentrator tube. Remove at this point and let cool for 10 minutes.

11.5.4 Disassemble K-D setup and place concentrator tube on N-Evap until final volume is at 0.25 mL, 0.5 mL or 1 mL depending upon initial volumes of sample. For SVE_01 and SVE_05 if 1 liter is used then final volume is 1 mL or if 500 mL is used then the final volume is 0.5 mL. For SVE_07, SVE_13, or SVE_09 if 1 liter is used then final volume is 0.5 mL and if 500 mL is used then final volume is 0.25 mL.

11.5.5 Transfer extract into a 2ml amber vial and label with parameter, sample number, fraction, extraction date, and final volume. Deliver this with completed extraction sheet to screening area for screening analysis.

11.5.6 Extracts are delivered to GC/MS dept. for analysis.

12.0 CALCULATIONS

Not applicable

13.0 ACCEPTANCE OF DATA

13.1 Method blanks must not contain target analytes exceeding MDL for that specific analyte.

13.2 Blank QC and Matrix Spikes should meet acceptance criteria as defined in the analytical SOP.

14.0 REPORTING OF RESULTS

N/A

15.0 SUPPLEMENTAL DOCUMENTS

N/A

16.0 REFERENCES

16.1 USEPA SW-846, 3rd Edition, Method 3520.

16.2 USEPA SW-846, 3rd Edition, Method 8270.

16.3 "Guidelines Establishing Test Procedures for the Analysis of Pollutants Under the Clean Water Act", USEPA, 40 CFR Part 136, October 26, 1984. Method 625.

17.0 SUBSTANTIVE REVISIONS

17.1 Section 2.2 amended: SVE_03 extraction deleted. Setion 2.3 amended: SOP document control number changed to SPS01401.NC to reflect revision. Addendum to Section 3.1: 3510 (separatory funnel) included. Section 5.3 amended: Monitoring changed from monthly to quarterly, word order changed from exposure of solvents to employees to employees to solvents. Section 6.4 amended: Holding time changed from 14 to 7 days from sampling. Addendum: Section 7.13 500 uL gastight syringe added. Addendum: Section 7.14 2.0 mL amber vials added. Addendum: Section 7.14 2 L separatory funnels added. Amendment to Section 8.1: Methylene chloride grade changed from Fisher Optima Grade to Baxter High Purity Solvent. Addendum to Section 8.3: SOP Doc# SPS02300.NC referenced. Addendum to Section 8.4: Details preparation of 1:1 sulfuric acid/d.i. water. Addendum: Section 8.5 Surrogate - prepared by GCMS dept. added. Addendum: Section 8.6 MS/MSD spiking solution - prepared by GCMS dept. added. Addendum: Section 8.7 TCLP spiking solution - prepared by GCMS dept. added. Addendum: Section 8.8 QC check solution - prepared by GCMS dept. added. Amendment to Section 11.1: Test codes SVE_03, SVE_07, SVE_09 and SVE_13 omitted. Amendments to Section 11.1.4: Procedures relavent to test codes SVE_03, SVE_07, SVE_09, and SVE_13 omitted. Procedure relevent to TCLP blank and regulatory spikes omitted. Addendum to 11.1.3: Round bottom flask removed after the majority of methylene chloride is brought over. Section 11.1.4 amended: 11.4 changed to 11.5. Addendum to Section 11.2.9: Round bottom flask removed after the majority of the methylene chloride is brought over. Section 11.2.10 amended: Go to 11.4 changed to 11.5. Addendum: Section 11.3 (Sections 11.3.1 - 11.3.8) details extraction for SVE_07, SVE_09, and SVE_13 test codes. Amendments to Section 11.4 (formerly Section 11.3): Test code SVE_05 QC Check changed from 1 L = 0.5 mL to 1 L = 1.0 mL. Test code SVE_01 MS/MSD spike changed from 1 L = 1.0 mL to 1 L = 0.5 mL. SVE_03 omitted. Test codes SVE_07, SVE_09 and SVE_13 MS/MSD spike values changed from N/A to 1 L = 0.5 mL. Test code SVE_01 BNA Surrogate changed from 1 L = 1.0 mL to 1 L = 0.5 mL. Test codes SVE_07, SVE_09, and SVE_13 BNA Surrogates changed from 200 mL = 0.5 mL to 500 mL = 0.25 mL. Test code SVE_07 BNA TCLP Spike changed from 200 mL = 0.5 mL to 500 mL = 0.25 mL. Section 11.5 was formerly 11.4. Section 11.5.3 amended: 2 mL changed to 4 mL. Addenda to Section 11.5.4: 0.25 mL included as a possible volume. Examples of final volumes for test codes detailed (see Table 1). Section 11.5.5 amended: 2 mL vial is changed to 2 mL amber vial.

11. IEA SOP FOR SW-846 8270B SVOCs ANALYSIS

1.0 APPROVALS

The signatures of the following individuals indicate that this SOP is complete and meets the requirements specified in corporate document # QAS00200.NET. In addition, it signifies that the content meets the specifications of the referenced "Test Code".

Laboratory Director

Quality Assurance Manager

UNCONTROLLED COPY
PROPRIETARY DOCUMENT

2.0 SCOPE AND APPLICATION

2.1 The objective of this document is to outline the techniques for determining the presence and concentration of various semivolatile organic target and non-target compounds in multi-media, multi-concentration samples. The target compounds for this method are listed in Attachment A at the end of this SOP. The method used in this procedure is solvent extraction and gas chromatograph/mass spectrometer analysis.

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

2.3 The following test codes are used to define analyses conducted in accordance with all of the conditions set forth by this SOP:MSS03, MSS04, MSS07,MSS08, MSS11, MSS12, MSS18, MSS19, MSS13, MSS17, MSS14, MSS18, MSS15, MSS16.

3.0 SUMMARY OF METHOD

3.1 This method employs the technique of solvent extraction, coupled with a gas chromatograph/mass spectrometer analysis. An aliquot of sample extract is injected onto a gas chromatograph. The fused silica capillary column is then temperature programmed to separate the semivolatiles prior to detection by the mass spectrometer.

3.2 This method is based on the Test Methods for Evaluating Solid and Hazardous Waste, SW-846, Third edition, Revision 0, September 1986, Method 8270. It also references the USEPA Contract Laboratory Program Statement of Work for Organics Analysis, Document Number OLM01.0.

3.3 Method Deviations

The tuning solution contains DFTPP only in Methylene Chloride.
Reextraction criteria is based on SOW 1/91.

MS/MSD criteria is as specified by this SOP.
Compound list and Quantitation limits applicable are listed in Attachment A of this SOP.
Stock Standards are purchased from Restex Corporation.
A volume of 2ul is injected onto the column for every analysis.
The Final Temperature is held at 300 C.
The surrogate solution is made up with the acids at 150ug/ml and the bases at 100ug/ml.

4.0 INTERFERENCES

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

5.0 SAFETY

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

lab coats
safety glasses with side shields and
chemical resistant gloves

when handling samples or preparing standards.

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

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

6.0 SAMPLE PRESERVATION AND STORAGE

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

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

7.0 APPARATUS AND MATERIALS

7.1 GC/MS/DS System

7.1.1 Hewlett-Packard Model 5890/5971A GC/MS, Extrel Model ELQ-400

7.1.2 Splitless type injection port

7.1.3 GC Column - 30 m x 0.32 mm ID x 0.5 um film thickness DB-5 J&W Scientific

7.1.4 Unix with Target software, Extrel software versions 8.1, 8.2

7.1.5 8-track on-line magnetic tape storage unit and DAT.

7.2 Syringes - various size (10 ul to 1 ml) Hamilton gas-tight syringes

8.0 REAGENTS AND STANDARD PREPARATION

8.1 Stock Standards - certified standards purchased from Restek Corporation in a semivolatile organics kit containing ampulated mixes of target compounds, matrix spike compounds, surrogates. Internal standards made from neat compounds.

8.1.1 New ampules are opened every 12 months, or sooner, if the standard has degraded or evaporated

8.2 Working Standards

8.2.1. Calibration Standard Solutions

Calibration standards at five concentration levels 10, 25, 40, 60, and 80 ng/ul (or 20, 50, 80, 120 and 160 ng/2ul) are prepared from the stock solutions. Each standard contains all the target compounds and surrogates.

8.2.2 Internal Standard (IS) Spiking Solution

A 2000 ng/ul IS spiking solution containing the internal standards 1,4-dichlorobenzene-d4, naphthalene-d8, acenaphthene-d10, phenanthrene-d10, chrysene-d12, and perylene-d12, is prepared from stock solutions every 12 months, or sooner, if the solution has degraded or evaporated. Add 10 ul of this solution to 1.0 ml or 5 ul to 0.5 ml to an aliquot of extract, standard, or blank for a concentration of 40 ng/2ul.

8.2.3 Surrogate (SU) Spiking Solution

The acid and base-neutral SU spiking solutions containing the eight

semivolatile surrogate compounds in methanol are prepared by the Semivolatiles Group and are utilized in the preparation of the calibration standards. The concentration of acids are at 150 ug/ml and bases are at 100 ug/ml.

9.0 CALIBRATION

9.1 Calibration Standards

Five initial calibration standards containing all the semivolatile target compounds and SU's are analyzed by injecting 2 ul of the calibration standards prepared in section 8.2.1, resulting in final concentrations of 20, 50, 80, 120, and 160 ng/2ul, or ng on column.

9.2 The working calibration range of this method is defined by the initial calibration curve, 20 ng/2ul to 160 ng/2ul. All extracts with target compounds exceeding 160 ng/2ul must be diluted to within the upper half of the calibration range.

9.3 The continuing calibration standard is the 50 ng/2ul standard from the initial calibration or is a separate standard injected as above at 50 ng/2ul.

9.4 Acceptance criteria

9.4.1 Initial Calibration

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

The Calibration Check Compounds (CCC) listed in Table 1.0 must have a %RSD of 30% or less and the System Performance Check Compounds (SPCC) listed in Table 2.0 must have a response factor (RF) of 0.050 or greater for the initial calibration to be acceptable.

Table 1.0 Calibration Check Compounds (CCC)

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

Table 2.0 System Performance Check Compounds

N-Nitroso-di-n-propylamine
Hexachlorocyclopentadiene
2,4-Dinitrophenol
4-Nitrophenol

9.4.2 Continuing Calibration

The continuing calibration standard must be analyzed every 12 hours to verify that the initial calibration is still valid.
The CCC's previously listed in Table 1.0 must have a %D of 30% or less and the SPCC's previously listed in Table 2.0 must have a response factor (RF) of 0.050 or greater for the continuing calibration to be acceptable.
The continuing calibration will be used in quantitation of the samples analyzed within a 12 hr. period of the continuing calibration.

10.0 QUALITY CONTROL

10.1 No method detection limit determination is required by this method.

10.2 Practical quantitation limits for this method are defined as Practical Quantitation Limits (PQL's) and are listed in attached forms.

10.3 Daily Performance Tests

10.3.1 Prior to initiating any data collection activities or at the start of every 12 hour sequence, it is necessary to establish that a given GC/MS system meets the instrument performance criteria. This is accomplished through the analysis of 50 ng/2ul of decafluorotriphenylphosphine (DFTPP).

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

Table 3.0 DFTPP: Ion Abundance Criteria

Mass	Requirement		Ref. Ion
51	30-60%		198
68	<2%		69
70	<2%		69
127	40-60%		198
197	<1%		198
198	100%		
199	5-9%		198
275	10-30%		198
365	>1%		198
441	Present	<	443
442	>40%		198
443	17-23%		442

10.3.2 After the instrument performance criteria is met, the initial calibration curve must be verified through the analysis of a continuing calibration at 50 ng/2ul. The continuing calibration criteria must be met before any method blank or sample analyses may proceed.

10.3.3 A extraction method blank consisting of 1 L of reagent water (or 30.0g of sodium sulfate) spiked with surrogates is extracted with every batch of samples and must be analyzed with the sample extracts after calibration criteria has been met. An acceptable method blank must meet the following criteria:

* A blank must be analyzed on the same instrument as the samples associated with the blank.

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

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

10.4 Matrix Spike and Matrix Spike Duplicates

10.4.1 A MS and MSD must be extracted and analyzed for each group of samples of a similar matrix within each case, 20 samples, group of samples of a similar concentration level (soils only), or each 14 calendar day period; whichever is more frequent.

10.4.2 The limits for matrix spike compound recovery and relative percent difference (RPD) are given in Table 4.0. These limits are only advisory; therefore, no further action is required if the criteria limits are not achieved. However, frequent failures shall be investigated for possible laboratory generated error.

10.4.3 QC Qheck

A QC Check, containing all analytes of interest, is extracted and analyzed with each batch of samples. Internal limits are established but the recoveries must meet those limits listed in Table 6 of method 8270, where applicable. These limits are used for water samples. Soil recovery criteria are based on historical data and internally established control limits.

Table 4.0 Matrix Spike Compound Recovery and RPD

COMPOUND NAME	WATER RECOVERY	WATER RPD	SOIL RECOVERY	SOIL RPD
Phenol	12 - 110	42	26 - 90	35
2-Chlorophenol	27 - 123	40	25 - 102	50
1,4-Dichlorobenzene	36 - 97	28	28 - 104	27
N-Nitroso-di-n-propylamine	41 - 116	38	41 - 126	38
1,2,4-Trichlorobenzene	39 - 98	28	38 - 107	23
4-Chloro-3-methylphenol	23 - 97	42	26 - 103	33
Acenaphthalene	46 - 118	31	31 - 137	19
4-Nitrophenol	10 - 80	50	11 - 114	50
2,4-Dinitrotoluene	24 - 96	38	28 - 89	47
Pentachlorophenol	9 - 103	50	17 - 109	47
Pyrene	26 - 127	31	35 - 142	36

10.5 Do not dilute MS or MSD for matrix spike compounds above working calibration limit.

10.6 Surrogates

10.6.1 The surrogates Phenol-d5, 2,4,6-Tribromophenol, 2-Fluorophenol, Nitrobenzene-d5, Terphenyl-d14, and 2-Fluorobiphenyl are added to each sample, blank, standard, and MS/MSD, prior to extraction. The acid surrogates are added at 75 ug and the BN's at 50 ug for waters and soils (0.5 ml of 150 and 100 ug/ml solutions).

10.6.2 Surrogate recoveries must be within the QC limits given in Table 5.0. If the recovery for any two acid or base/neutral surrogates are not within QC limits, or if any surrogate is below 10%, the following actions are required:

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

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

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

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

If the reanalysis of the sample solves the problem, then only submit the second analysis. If the reanalysis does not solve the problem, then re-extract and reanalyze the sample. One B/N and one Acid surrogate are allowed to not meet criteria, however, each failing surrogate must be > 10% recovered. If a sample requires an acid only or a base only analysis, that sample is acceptable if one acid surrogate or one b/n surrogate fails criteria. Any failing surrogates must be >10% recovered for the data to be valid. If this criteria is not met, the sample will be re-extracted.

10.6.2a Surrogate recovery in Dilutions

Surrogates must be detected in all diluted extracts up to, and including, a 10x dilution. The surrogate recovery must meet criteria in all dilutions up to, and including a 5x dilution. It is not a requirement for surrogates to pass criteria in a 10x dilution, however, the surrogates must be detected for the data to be valid. Re-extraction will be performed for samples not meeting this protocol.

10.6.3 If the recovery of the surrogates in a method blank is not acceptable, then the method blank and all associated samples must be re-extracted and reanalyzed. Blanks can not have any surrogates outside of the limit.

Table 5.0 Surrogate Recovery Limits

SURROGATE COMPOUND	WATER RECOVERY (%)	SOIL RECOVERY (%)
Nitrobenzene-d5 (BN)	35 - 114	23 - 120
2-Fluorobiphenyl (BN)	43 - 116	30 - 115
p-Terphenyl-d14 (BN)	33 - 141	18 - 137
Phenol-d5 (AC)	10 - 94	24 - 113
2-Fluorophenol (AC)	21 - 100	25 - 121
1,2-Dichlorobenzene-d4 (BN)*	16-110	20-130
2-Chlorophenol-d4 (AC)*	33-110	20-130
2,4,6-Tribromophenol (AC)	10 - 123	19 - 122

*= Recovery limits are adopted from OLMO1.0
BN= base/neutral surrogate
AC= acid surrogate

10.7 Internal Standards

10.7.1 IS's are added to each sample, blank, standard, and MS/MSD, at 20 ng prior to injection. Hence, 40 ng on column.

10.7.2 The retention times (RT) and extracted ion current profile (EICP) of each IS must be monitored for all analyses. The IS areas in a sample must not vary by more than a factor of 2 (-50% to +100%) from the continuing calibration's IS area. The RT must be within 30 seconds of its RT in the continuing calibration. If IS criteria is not within limits, the following are required:

* After corrections are made, the lab must demonstrate that the mass spectrometer is functioning properly. This is accomplished by the analysis of a standard or sample that does meet, then samples can be re-analyzed.

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

standards

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

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

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

11.0 SAMPLE PREPARATION AND INSTRUMENTAL PROCEDURES

11.1 Instrumental Conditions

11.1.1 Gas Chromatograph

Carrier Gas:	Helium
Flow Rate:	30 cm/sec
Initial Temp.:	40 deg. C
Initial Hold:	4 min
Ramp Rate 1:	10 deg/min
Second Temp.:	270 deg. C
Second Hold:	12 min
Ramp Rate 2:	8 deg/min
Final Temp.:	310 deg. C - 300 deg. C
Final Hold:	10 min
Transfer Temp:	290 deg. C
Injector:	Grob-type, splitless
Sample Volume:	2 ul

11.1.3 Mass Spectrometer

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

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

11.2 Sample Analysis Procedures

11.2.1 Sample Extract Analysis

Sample extracts are removed from Sample storage - Frig R10 and are signed out on the extraction sheet. After analysis the sample extracts are transferred to R12 and are kept for 30 days at 4 deg. C.

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

In a 2ml screw top or autosampler vial, load a 1000 ul aliquot of sample extract and spike with 10 ul of IS spiking solution.

Inject 2 ul of the sample extract with IS onto the GC column and start the GC temperature program sequence.

If a sample is analyzed which contains target compounds at concentrations greater than the initial calibration upper limit, then the sample must be reanalyzed at an appropriate dilution. Do not dilute MS/MSDs for MS compounds.

11.3 Qualitative Analysis

11.3.1 Target Compounds

The relative retention time of a target compound must be within +/- 0.50 RRT units of the RRT of the calibration standard for a positive identification. In addition, a comparison must be made between the mass spectrum obtained in the sample analysis and the reference mass spectrum for that compound, which was obtained on that specific GC/MS system. The requirements for qualitative verification by comparison of mass spectra are as follows:

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

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

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

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

11.3.2 Tentatively Identified Compounds

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

Up to 20 organic compounds of apparent concentration not listed in Attachment A, shall be tentatively identified via a forward library search. Only compounds with responses greater than 10% of the closest IS exhibiting no interference are to be searched.

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

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

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

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

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

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

11.4 Quantitative Analysis

11.4.1 Target Compounds

Target compounds are quantitated by the internal standard technique. The associated internal standards used are listed in Table 5.0 of SW 846 Method 8270A. The EICP areas of the quantitation ions of compounds listed Table 1.0 of SW 846 Method 8270A are used.

The relative response factor (RRF) from the continuing calibration standard

is used to calculate the concentration in the sample. When compound concentrations are below the PQL, but the compound meets identification criteria, report the concentration with a "J" qualifier per client request.

Water Samples

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

where,

Ax =area of the compound quantitation ion

Ais =area of IS quantitation ion

Is =IS amount in nanograms

RRF =continuing calibration standard RRF

Vo =volume of water extracted in ml's

Vi =volume of extract injected in ul's

Vt =volume of the concentrated extract in ul's

Df =dilution factor

Soil Samples

$$\text{Concentration (dry weight basis)} \quad \text{ug/Kg} = \frac{(Ax)(Is)(Vt)(Df)(2.0)}{(Ais)(RRF)(Vi)(Ws)(D)}$$

where,

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

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

Ws = weight of sample extracted in grams

11.4.2 Tentatively Identified Compounds

An estimated concentration for non-target compounds tentatively identified

in the sample shall be determined by the internal standard method. For quantitation, the nearest IS free of interferences shall be used.

The equation for calculating concentrations are the same as in 11.4.1. Total area counts from the total ion chromatograms are used for both the IS and compound. A RRF of 1.0 is assumed.

12.0 CALCULATIONS

12.1 Relative Response Factor (RRF)

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

where,

Ax =area of the compound quantitation ion

Ais =area of IS quantitation ion

Cis =IS concentration

Cx =compound concentration

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

12.2 Percent Relative Standard Deviation (%RSD)

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

12.3 Percent Difference (%D)

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

where,

RRFi =average RRF from the initial calibration

RRFc =RRF from the continuing calibration standard

12.4 Percent Moisture

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

12.5 Target Compound Concentrations

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

12.6 Surrogate Percent Recovery

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

12.7 Matrix Spike Recovery

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

where,

SSR = spiked sample result

SR = sample result

SA = spike added

12.8 Relative Percent Difference

$$RPD = \frac{|(MSR - MSDR)|}{(1/2)(MSR+MSDR)} \times 100$$

where,

MSR = matrix spike recovery

MSDR = matrix spike duplicate recovery

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

12.9 Adjusted Practical Quantitation Limit for Samples

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

where,

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

Df = the dilution factor

13.0 ACCEPTANCE OF DATA

13.1 Method Blank

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

13.2 Surrogates (SU)

Refer to section 10.6 for surrogate acceptance criteria and corrective actions.

13.3 Instrument Performance Check

The criteria for DFTPP is listed in Table 3.0 and in section 10.3.1.

13.4 Internal Standards

The IS acceptance criteria is described in section 10.7.

13.5 Matrix Spike/Matrix Spike Duplicate

The MS/MSD acceptance criteria is described in section 10.4.

14.0 REPORTING OF RESULTS

Data reporting forms are in Appendix A.

14.1 General Information

The following sample suffixes are used:

MS = matrix spike
MSD= matrix spike duplicate
RE = reextracted and/or reanalyzed sample
DL = sample analyzed at a secondary dilution

All semivolatile standards are designated SSTD###, where ### is the concentration level of the standard (i.e. SSTD050).

All semivolatile method blanks are designated SVB###, where ### is a unique numeric designator.

All header field information must be in capital letters.

Instrument ID is common on many of the forms and must include some indication of the manufacturer and/or model of the instrument, and contain addition characters to uniquely identify each instrument (EXTR4 or MSD8).

14.2 Organic Analysis Data Sheet

The header information is completed with the requested information and in the format required.

All results are reported as uncorrected for blank contaminants and to one significant figure if the value is less than 10, and two significant figures if the value is greater than 10.

Data reporting qualifiers are used under the "Q" column and are defined as follows (results below 1 are not reported on the forms):

BQL - Indicates compound was analyzed for but detected at a concentration below the quantitation limit or not detected at all.

J - Indicates an estimated value and is used for reporting all target compound concentrations which are less than the adjusted PQL.

B - This qualifier is used when a compound is found in the associated blank as well as the sample, both target compounds and TIC's use this qualifier

E - This qualifier indicates that a target compound concentration has exceeded the working range of the initial calibration

Other laboratory defined qualifiers may be utilized as long as they are defined in the narrative.

14.3 Semivolatile Organics Analysis Data Sheet Tentatively Identified Compounds

TIC's are reported per client request.

For each tentatively identified compound, report the compound name and estimated concentration.

For each compound which no tentative identification can be made, report the compound as "unknown" along with the estimated concentration. Give further descriptive classification of the unknown compound whenever possible.

Report the total number of TIC's identified per section 11.3.2.

14.4 Surrogate Recovery (Level II only)

All method blank, sample, and MS/MSD surrogate (SU) recoveries are reported on this form to two significant figures if above 10% recovery, and one significant figure if less than 10%.

Any SU recovery outside the control limits must be flagged with an asterisk and the total number out designated in the far right hand column.

Any SU recovery which is reported from a dilution, shall be designated with a "D" flag to indicate diluted out. Recoveries with "D" flags are not counted towards the total number out of criteria.

14.5 Matrix Spike/Matrix Spike Recovery (Level II only)

This form is used to report the results of the analyses of a MS/MSD. The form is matrix specific.

Complete the header and spike information.

Flag all percent recoveries and RPD values outside the QC limits with an asterisk and total the number out at the bottom of the form.

15.0 SUPPLEMENTAL DOCUMENTS

15.1 Copies of IEA-North Carolina reporting sheets for Method 8270 in Attachment A.

16.0 REFERENCES

16.1 SW846, Third Edition, Revision 1, July 1992.

16.2 USEPA Contract Laboratory Program Statement of Work for Organics Analysis, Document Number OLM01.9.

17.0 SUBSTANTIVE REVISIONS

17.1 None from the original issue of this SOP.

17.2 The following modifications have been made to this SOP.
Section 3.3 Method Deviations were added as they appear in this SOP.
Test codes applicable to this SOP were added in Section 2.3.

17.3 See paragraph 10.6.1, Table 5, 10.6.2a, and 10.3.3

Attachment A

IEA
An Aquarion Company

BASE/NEUTRAL/ACID EXTRACTABLES
SW-846 METHOD 8270

IEA Sample Number:	_____	Date Received:	_____
Client Name:	_____	Date Sampled:	_____
Client Project I.D.:	_____	Date Extracted:	_____
Sample Identification:	_____	Date Analyzed:	_____
Matrix:	_____	Analysis By:	_____
Moisture Correction Factor:	_____	Dilution Factor:	_____

Number	Compound	Quantitation Limit (ug/kg)	Results Concentration (ug/kg)
1	Acenaphthene	200	BQL
2	Acenaphthylene	200	BQL
3	Anthracene	200	BQL
4	Benzoic acid	1000	BQL
5	Benzo(a)anthracene	200	BQL
6	Benzo(b)fluoranthene	200	BQL
7	Benzo(k)fluoranthene	200	BQL
8	Benzo(g,h,i)perylene	200	BQL
9	Benzo(a)pyrene	200	BQL
10	Benzyl alcohol	400	BQL
11	bis(2-Chloroethoxy)methane	200	BQL
12	bis(2-Chloroethyl)ether	200	BQL
13	bis(2-Chloroisopropyl)ether	200	BQL
14	bis(2-Ethylhexyl)phthalate	200	BQL
15	4-Bromophenyl phenyl ether	200	BQL
16	Benzyl butyl phthalate	200	BQL
17	4-Chloroaniline	400	BQL
18	2-Chloronaphthalene	200	BQL
19	4-Chloro-3-methylphenol	400	BQL
20	2-Chlorophenol	200	BQL
21	4-Chlorophenyl phenyl ether	200	BQL
22	Chrysene	200	BQL
23	Dibenzo(a,h)anthracene	200	BQL
24	Dibenzofuran	200	BQL
25	Di-n-butylphthalate	200	BQL
26	1,3-Dichlorobenzene	200	BQL
27	1,4-Dichlorobenzene	200	BQL
28	1,2-Dichlorobenzene	200	BQL
29	3,3'-Dichlorobenzidine	400	BQL
30	2,4-Dichlorophenol	200	BQL
31	Diethyl phthalate	200	BQL
32	2,4-Dimethylphenol	200	BQL
33	Dimethyl phthalate	200	BQL
34	4,6-Dinitro-2-methylphenol	1000	BQL
35	2,4-Dinitrophenol	1000	BQL
36	2,4-Dinitrotoluene	200	BQL
37	2,6-Dinitrotoluene	200	BQL
38	Di-n-octylphthalate	200	BQL
39	Fluoranthene	200	BQL

IEA
An Aquarion Company

BASE/NEUTRAL/ACID EXTRACTABLES
SW-846 METHOD 8270

IEA Sample Number:		Date Received:	
Client Name:		Date Sampled:	
Client Project I.D.:		Date Extracted:	
Sample Identification:		Date Analyzed:	
Matrix:		Analysis By:	
Moisture Correction Factor:		Dilution Factor:	

Number	Compound	Quantitation Limit (ug/kg)	Results Concentration (ug/kg)
40	Fluorene	200	BQL
41	Hexachlorobenzene	200	BQL
42	Hexachlorobutadiene	200	BQL
43	Hexachlorocyclopentadiene	200	BQL
44	Hexachloroethane	200	BQL
45	Indeno(1,2,3-cd)pyrene	200	BQL
46	Isophorone	200	BQL
47	2-Methylnaphthalene	200	BQL
48	2-Methylphenol (o-cresol)	200	BQL
49	4-Methylphenol (p-cresol)	200	BQL
50	Naphthalene	200	BQL
51	2-Nitroaniline	1000	BQL
52	3-Nitroaniline	1000	BQL
53	4-Nitroaniline	1000	BQL
54	Nitrobenzene	200	BQL
55	2-Nitrophenol	200	BQL
56	4-Nitrophenol	1000	BQL
57	N-Nitroso-di-n-propylamine	200	BQL
58	N-Nitrosodiphenylamine	200	BQL
59	Pentachlorophenol	1000	BQL
60	Phenanthrene	200	BQL
61	Phenol	200	BQL
62	Pyrene	200	BQL
63	1,2,4-Trichlorobenzene	200	BQL
64	2,4,5-Trichlorophenol	200	BQL
65	2,4,6-Trichlorophenol	200	BQL

Comments:
Sample specific quantitation limits may be calculated by multiplying the quantitation limit by the dilution factor.
BQL = Below Quantitation Limit

Industrial & Environmental Analysts, Inc. (IEA)
WATER SEMIVOLATILE SURROGATE RECOVERIES

IEA Project No.:_____

Sample ID	SURR 1	SURR 2	SURR 3	SURR 4	SURR 5	SURR 6

QC Limits

S1 = Nitrobenzene-d5	(35-114)
S2 = 2-Fluorobiphenyl	(43-116)
S3 = Terphenyl-d14	(33-141)
S4 = Phenol-d5	(10-110)
S5 = 2-Fluorophenol	(21-100)
S6 = 2,4,6-Tribromophenol	(10-123)

FORM WSVSURR Rev. 071591

Industrial & Environmental Analysts, Inc. (IEA)
WATER SEMIVOLATILE MATRIX SPIKE/MATRIX SPIKE DUPLICATE RECOVERY

IEA Project No.:
IEA Reference No.:

COMPOUND	SPIKE ADDED (ug/L)	SAMPLE CONCENTRATION (ug/L)	MS CONCENTRATION (ug/L)	MS % REC #	QC LIMITS REC
Phenol	50	BQL	_____	0	12-89
2-Chlorophenol	50	BQL	_____	0	27-123
1,4-Dichlorobenzene	50	BQL	_____	0	36-97
N-Nitroso-di-n-prop.(1)	50	BQL	_____	0	41-116
1,2,4-Trichlorobenzene	50	BQL	_____	0	39-98
4-Chloro-3-methylphenol	50	BQL	_____	0	23-97
Acenaphthene	50	BQL	_____	0	46-118
4-Nitrophenol	50	BQL	_____	0	10-80
2,4-Dinitrotoluene	50	BQL	_____	0	24-96
Pentachlorophenol	50	BQL	_____	0	9-103
Pyrene	50	BQL	_____	0	26-127

	SPIKE ADDED (ug/L)	MSD CONCENTRATION (ug/L)	MSD % REC #	% RPD #	QC LIMITS RPD	QC LIMITS REC
Phenol	50	_____	0	ERR	42	12-89
2-Chlorophenol	50	_____	0	ERR	40	27-123
1,4-Dichlorobenzene	50	_____	0	ERR	28	36-97
N-Nitroso-di-n-prop.(1)	50	_____	0	ERR	38	41-116
1,2,4-Trichlorobenzene	50	_____	0	ERR	28	39-98
4-Chloro-3-methylphenol	50	_____	0	ERR	42	23-97
Acenaphthene	50	_____	0	ERR	31	46-118
4-Nitrophenol	50	_____	0	ERR	50	10-80
2,4-Dinitrotoluene	50	_____	0	ERR	38	24-96
Pentachlorophenol	50	_____	0	ERR	50	9-103
Pyrene	50	_____	0	ERR	31	26-127

(1) N-Nitroso-di-n-propylamine

COMMENTS:

FORM WASEMMAS REV 012492

Industrial & Environmental Analysts, Inc. (IEA)
BASE/NEUTRAL/ACID EXTRACTABLES SW-846 METHOD 8270
Laboratory Control Spike (LCS)

IEA Project Number: _____ Date Extracted: _____
IEA Sample Number: _____ Date Analyzed: _____
Client Name: _____ Analysis By: _____
Client Project I.D.: _____ Matrix: Water
Sample Identification: SLCS##

Number	Compound	Amount Spiked (NG)	Amount Recovered (NG)	SLCS % REC	Control Limits Lower (%REC)	Upper (%REC)
1	Phenol	100		0	30	112
2	bis(2-Chloroethyl)ether	100		0	12	158
3	2-Chlorophenol	100		0	40	134
4	1,3-Dichlorobenzene	100		0	10	124
5	1,4-Dichlorobenzene	100		0	40	124
6	1,2-Dichlorobenzene	100		0	32	129
7	Benzyl alcohol	100		0	10	145
8	bis(2-Chloroisopropyl)ether	100		0	36	166
9	2-Methylphenol (o-cresol)	100		0	10	136
10	N-Nitroso-di-n-propylamine	100		0	40	138
11	Hexachloroethane	100		0	40	113
12	4-Methylphenol (p-cresol)	100		0	10	132
13	Nitrobenzene	100		0	35	180
14	Isophorone	100		0	21	196
15	2-Nitrophenol	100		0	29	182
16	2,4-Dimethylphenol	100		0	32	119
17	bis(2-Chloroethoxy)methane	100		0	33	184
18	2,4-Dichlorophenol	100		0	39	135
19	Benzoic acid	100		0	10	91
20	1,2,4-Trichlorobenzene	100		0	44	142
21	Naphthalene	100		0	21	133
22	4-Chloroaniline	100		0	10	136
23	Hexachlorobutadiene	100		0	24	116
24	4-Chloro-3-methylphenol	100		0	40	147
25	2-Methylnaphthalene	100		0	10	136
26	Hexachlorocyclopentadiene	100		0	10	110
27	2,4,6-Trichlorophenol	100		0	37	144
28	2,4,5-Trichlorophenol	100		0	10	144
29	2-Chloronaphthalene	100		0	60	118
30	2-Nitroaniline	100		0	10	159
31	Dimethyl phthalate	100		0	10	112
32	Acenaphthylene	100		0	40	145
33	2,6-Dinitrotoluene	100		0	50	158
34	3-Nitroaniline	100		0	10	173
35	Acenaphthene	100		0	47	145
36	2,4-Dinitrophenol	100		0	10	141
37	Dibenzofuran	100		0	10	142
38	4-Nitrophenol	100		0	40	132
39	2,4-Dinitrotoluene	100		0	40	139

Industrial & Environmental Analysts, Inc. (IEA)
BASE/NEUTRAL/ACID EXTRACTABLES SW-846 METHOD 8270
Laboratory Control Spike (LCS)

IEA Project Number: _____ Date Extracted: _____
IEA Sample Number: _____ Date Analyzed: _____
Client Name: _____ Analysis By: _____
Client Project I.D.: _____ Matrix: Water
Sample Identification: SLCS##

Number	Compound	Amount Spiked (NG)	Amount Recovered (NG)	SLCS % REC	Control Limits Lower (%REC)	Upper (%REC)
40	Diethyl phthalate	100		0	10	- 114
41	Fluorene	100		0	59	- 121
42	4-Chlorophenyl phenyl ether	100		0	25	- 158
43	4-Nitroaniline	100		0	10	- 165
44	4,6-Dinitro-2-methylphenol	100		0	10	- 155
45	N-Nitrosodiphenylamine	100		0	10	- 150
46	4-Bromophenyl phenyl ether	100		0	53	- 127
47	Hexachlorobenzene	100		0	10	- 144
48	Pentachlorophenol	100		0	40	- 150
49	Phenanthrene	100		0	54	- 120
50	Anthracene	100		0	27	- 133
51	Di-n-butylphthalate	100		0	10	- 118
52	Fluoranthene	100		0	26	- 137
53	Pyrene	100		0	52	- 115
54	Benzyl butyl phthalate	100		0	10	- 146
55	Benzo(a)anthracene	100		0	33	- 143
56	3,3'-Dichlorobenzidine	100		0	10	- 157
57	Chrysene	100		0	17	- 168
58	bis(2-Ethylhexyl)phthalate	100		0	10	- 145
59	Di-n-octylphthalate	100		0	10	- 146
60	Benzo(b)fluoranthene	100		0	24	- 159
61	Benzo(k)fluoranthene	100		0	10	- 151
62	Benzo(a)pyrene	100		0	17	- 163
63	Indeno(1,2,3-cd)pyrene	100		0	10	- 149
64	Dibenzo(a,h)anthracene	100		0	10	- 152
65	Benzo(g,h,i)perylene	100		0	10	- 155

Comments:

Corresponding Samples:
Filename:

Industrial & Environmental Analysts, Inc. (IEA)
BASE/NEUTRAL/ACID EXTRACTABLES SW-846 METHOD 8270
Laboratory Control Spike (LCS)

IEA Project Number: _____ Date Extracted: _____
IEA Sample Number: _____ Date Analyzed: _____
Client Name: _____ Analysis By: _____
Client Project I.D.: _____ Matrix: Solid
Sample Identification: SLCS

Number	Compound	Amount Spiked (NG)	Amount Recovered (NG)	SLCS % REC	Control Limits Lower (%REC)	Upper (%REC)
1	Phenol	100	0	0	10	112
2	bis(2-Chloroethyl)ether	100	0	0	10	200
3	2-Chlorophenol	100	0	0	23	134
4	1,3-Dichlorobenzene	100	0	0	58	94
5	1,4-Dichlorobenzene	100	0	0	61	90
6	1,2-Dichlorobenzene	100	0	0	10	141
7	Benzyl alcohol	100	0	0	10	153
8	bis(2-Chloroisopropyl)ether	100	0	0	10	136
9	2-Methylphenol (o-cresol)	100	0	0	10	152
10	N-Nitroso-di-n-propylamine	100	0	0	52	93
11	Hexachloroethane	100	0	0	10	138
12	4-Methylphenol (p-cresol)	100	0	0	10	1[illegible]
13	Nitrobenzene	100	0	0	10	1[illegible]
14	Isophorone	100	0	0	10	142
15	2-Nitrophenol	100	0	0	10	160
16	2,4-Dimethylphenol	100	0	0	10	167
17	bis(2-Chloroethoxy)methane	100	0	0	10	144
18	2,4-Dichlorophenol	100	0	0	10	167
19	Benzoic acid	100	0	0	10	178
20	1,2,4-Trichlorobenzene	100	0	0	67	94
21	Naphthalene	100	0	0	10	145
22	4-Chloroaniline	100	0	0	10	117
23	Hexachlorobutadiene	100	0	0	10	150
24	4-Chloro-3-methylphenol	100	0	0	40	145
25	2-Methylnaphthalene	100	0	0	54	93
26	Hexachlorocyclopentadiene	100	0	0	10	121
27	2,4,6-Trichlorophenol	100	0	0	10	159
28	2,4,5-Trichlorophenol	100	0	0	10	159
29	2-Chloronaphthalene	100	0	0	10	147
30	2-Nitroaniline	100	0	0	10	148
31	Dimethyl phthalate	100	0	0	10	150
32	Acenaphthylene	100	0	0	10	142
33	2,6-Dinitrotoluene	100	0	0	10	155
34	3-Nitroaniline	100	0	0	10	120
35	Acenaphthene	100	0	0	63	92
36	2,4-Dinitrophenol	100	0	0	10	133
37	Dibenzofuran	100	0	0	10	143
38	4-Nitrophenol	100	0	0	37	132
39	2,4-Dinitrotoluene	100	0	0	67	95

Industrial & Environmental Analysts, Inc. (IEA)
BASE/NEUTRAL/ACID EXTRACTABLES SW-846 METHOD 8270
Laboratory Control Spike (LCS)

IEA Project Number: _____ Date Extracted: _____
IEA Sample Number: _____ Date Analyzed: _____
Client Name: _____ Analysis By: _____
Client Project I.D.: _____ Matrix: Solid
Sample Identification: SLCS

Number	Compound	Amount Spiked (NG)	Amount Recovered (NG)	SLCS % REC	Control Limits Lower (%REC)	Upper (%REC)
40	Diethyl phthalate	100	0	0	10	- 147
41	Fluorene	100	0	0	10	- 144
42	4-Chlorophenyl phenyl ether	100	0	0	10	- 154
43	4-Nitroaniline	100	0	0	10	- 146
44	4,6-Dinitro-2-methylphenol	100	0	0	10	- 160
45	N-Nitrosodiphenylamine	100	0	0	10	- 153
46	4-Bromophenyl phenyl ether	100	0	0	10	- 151
47	Hexachlorobenzene	100	0	0	10	- 148
48	Pentachlorophenol	100	0	0	29	- 152
49	Phenanthrene	100	0	0	10	- 150
50	Anthracene	100	0	0	10	- 152
51	Di-n-butylphthalate	100	0	0	10	- 148
52	Fluoranthene	100	0	0	10	- 147
53	Pyrene	100	0	0	48	- 121
54	Benzyl butyl phthalate	100	0	0	10	- 155
55	Benzo(a)anthracene	100	0	0	10	- 158
56	3,3'-Dichlorobenzidine	100	0	0	10	- 121
57	Chrysene	100	0	0	10	- 150
58	bis(2-Ethylhexyl)phthalate	100	0	0	10	- 149
59	Di-n-octylphthalate	100	0	0	10	- 152
60	Benzo(b)fluoranthene	100	0	0	10	- 148
61	Benzo(k)fluoranthene	100	0	0	10	- 150
62	Benzo(a)pyrene	100	0	0	10	- 151
63	Indeno(1,2,3-cd)pyrene	100	0	0	10	- 158
64	Dibenzo(a,h)anthracene	100	0	0	10	- 151
65	Benzo(g,h,i)perylene	100	0	0	10	- 151

Comments:

Corresponding Samples:
Filename:

Industrial & Environmental Analysts, Inc. (IEA)
SOIL SEMIVOLATILE MATRIX SPIKE/MATRIX SPIKE DUPLICATE RECOVERY

IEA Project No.: _____
IEA Reference No.: _____

COMPOUND	SPIKE ADDED (ug/kg)	SAMPLE CONCENTRATION (ug/kg)	MS CONCENTRATION (ug/kg)	MS % REC #	QC LIMITS REC
Phenol	_____	BQL	_____	ERR	26-90
2-Chlorophenol	_____	BQL	_____	ERR	25-102
1,4-Dichlorobenzene	_____	BQL	_____	ERR	28-104
N-Nitroso-di-n-prop.(1)	_____	BQL	_____	ERR	41-126
1,2,4-Trichlorobenzene	_____	BQL	_____	ERR	38-107
4-Chloro-3-methylphenol	_____	BQL	_____	ERR	26-103
Acenaphthene	_____	BQL	_____	ERR	31-137
4-Nitrophenol	_____	BQL	_____	ERR	11-114
2,4-Dinitrotoluene	_____	BQL	_____	ERR	28-89
Pentachlorophenol	_____	BQL	_____	ERR	17-109
Pyrene	_____	BQL	_____	ERR	35-142

	SPIKE ADDED (ug/kg)	MSD CONCENTRATION (ug/kg)	MSD % REC #	% RPD #	QC LIMIT RPD	QC LIMIT RE.
Phenol	_____	_____	ERR	ERR	35	26-90
2-Chlorophenol	_____	_____	ERR	ERR	50	25-102
1,4-Dichlorobenzene	_____	_____	ERR	ERR	27	28-104
N-Nitroso-di-n-prop.(1)	_____	_____	ERR	ERR	38	41-126
1,2,4-Trichlorobenzene	_____	_____	ERR	ERR	23	38-107
4-Chloro-3-methylphenol	_____	_____	ERR	ERR	33	26-103
Acenaphthene	_____	_____	ERR	ERR	19	31-137
4-Nitrophenol	_____	_____	ERR	ERR	50	11-114
2,4-Dinitrotoluene	_____	_____	ERR	ERR	47	28-89
Pentachlorophenol	_____	_____	ERR	ERR	47	17-109
Pyrene	_____	_____	ERR	ERR	36	35-142

(1) N-Nitroso-di-n-propylamine

COMMENTS:

12. IEA SOP FOR SW-846 3510 PESTICIDES/PCBs LIQUID MEDIA EXTRACTION

1.0 APPROVALS

The signature of the following individuals indicates that this SOP is complete and meets the requirements specified in corporate Document # QAS00200.NET. In addition, it also signifies that the content meets the specifications of the referenced "Test Code".

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PROPRIETARY DOCUMENT

Laboratory Director_____

Quality Assurance Manager_____

2.0 SCOPE AND APPLICATION

2.1 The objective of this document is to outline the techniques used to extract samples for pesticide analysis using method 3510.

2.2 The following test codes are used to define extractions in accordance with all of the conditions set forth by this SOP:

PE_01 PE_05 PE_07 PE_09

2.3 The document control number for this SOP shall be **SPS01600.NC.**

3.0 SUMMARY OF METHOD

3.1 Pre-determined amounts of samle are measured out and extracted using Method 3510. Upon completion of the extraction, the extract is delivered to GC for analysis.

4.0 INTERFERENCES

4.1 Interferences in the pesticde extraction cannot be detected until analysis is commplete. All possible precautions are taken to insure interference free extracts.

4.2 All glassware is baked to at least 900F.

4.3 All glassware is prerinsed with the extraction solvent prior to use.

5.0 SAFETY

5.1 Chemical resistant polyvinyl gloves should be worn at all times when working with solvents.

5.2 Each analyst has been issued a personal lab coat and it should be worn at all times in the

sample preparation laboratory area.

5.3 MSDS sheets are availble in the lab area for immediate reference in the case of an emergency.

6.0 SAMPLE CONTAINERS, COLLECTION AND PRESERVATION

6.1 Sample Containers

6.1.1 Water samples are collected with 2x1 liter amber glass containers with Teflon coated lid.

6.1.2 Bottles are prepared according to SOP 5.7,"Cleaning Botles Used for Organic Samples."

6.1.3 Sample bottles are never cleaned and reused.

6.2 Sample Collection

6.2.1 Samples are collected as grab samples in the field.

6.2.2 A cooler with either ice or blue ice should be available at the sampling site.

6.2.3 Samples are secured against breakage in the coolers wne kept at 4 deg. C for transport to the laboratory. Samples should arrive at the laboratory the next day following collection.

6.3 Sample Preservation

6.3.1 Samples are preserved by cooling to 4 deg. C.

6.4 Holding Times

6.4.1 Water samples must be extracted within 7 days from sampling.

6.4.2 All extracts must be analyzed within 40 days from date of extraction.

7.0 APPARATUS AND MATERIALS

7.1 Top-Loader balance

7.2 250ml beakers

7.3 2000ml Separatory Funnel

7.4 500ml Kuderna-Danish

7.5 3-ball Snyder Column

7.6 10ml concentrater tube

7.7 75mm funnel

7.8 Drying column

7.9 100ml graduated cylinder

7.10 Tekmar Sonicator

7.11 Whatman 41 filter paper

7.12 1000ul gastight syringe for spiking

7.13 12ml glass vials for extract storage.

8.0 REAGENTS AND STANDARDS PREPARATION

8.1 Reagent Water - water free of interferences at or above MDL of the method. This water is taken from a DI water system and pre-extracted with methylene chloride.

8.2 Methylene Chloride - Fisher Optima Grade from an approved lot.

8.3 Hexane - Fisher Optima Grade from a approved lot.

8.4 Surrogate and Matrix Spiking solutions are prepared by the GC staff according to the SOP for Pesticide/PCB analysis.

8.5 Granular Sodium Sulfate - Fisher ACS grade

8.6 Powdered Sodium Sulfate - Baxter ACS grade

8.7 Glass wool

9.0 CALIBRATION

N/A

10.0 QUALITY CONTROL

10.1 Method blanks consist of a 1L aliquot of reagent water for water samples. These blanks are treated exactly the same as other non QC samples and the frequency is one per batch (a batch is defined as a set of samples equal to or less than 20 in number).

10.2 Blank spikes consist of a 1L aliquot of reagent water for water samles. These blanks are treated exactly the same as other QC samples (sample MS/MSD) and the frequency is one per batch.

10.3 One sample is chosen at random to use for MS/MSD per batch. The MS/MSD are duplicate extractions of the same sample. The only difference is the additonal spiking of the pesticide MS solution.

11.0 SAMPLE PREPARATION AND INSTRUMENTAL PROCEDURES

11.1 Sample extraction

11.1.1 A one liter aliquot of sample is poured into a pre-rinsed two liter separatory funnel. The pesticide surrogate is added at a volume of 1ml per liter of sample used (or 1ml per 200ml of sample for TCLP samples). The pesticide matrix spiking solution is added to the blank spike, matrix spike and matrix spike duplicate at a volume of 1ml per liter of sample in additon to the pesticide surrogate. A separtte TCLP matrix spike is added to TCLP regulatory spike samples only at a volume of 1ml per 200ml.

11.1.2 60ml of methylene chloride is added to the separatory funnel. The separatory funnel is shaken vigorously and vented. The separatory funnel is then placed on the auto-shaker, partially vented, and shaken for two minutes.

11.1.3 The separatory funnel is removed from the shaker and allowed to stand for approximately 10 minutes to allow time for the solvent to separate from the sample. The solvent layer (which is on the bottom) is drained through the drying column into the K-D setup.

11.1.4 Steps 11.1.2 to 11.1.3 are re[eated twp additional times. The drying column is rinsed down with methylene chloride and the extraction portion

is now complete and the concentration is ready to be done.

11.2 Sample Concentration

11.2.1 The K-D setup is placed on the steambath. Approximately 2ml of methylene chloride is added to the Snyder Column to initiate boiling. A carbide boiling chip is also added to the K-D setup prior to the placement on the steambath. Boiling should start immediately and should take between 15 and 20 minutes to get the extract to an approximate volume of 15ml. When the extract is at ~15ml, add 50ml of Hexane to the extract and place back on the steambath. The extract should be boiled off until the extract reached an apparent volume of 4ml. The K-D setup should be removed at this point and cooled.

11.2.2 The extract should be brought to a final volume of 10ml, and transferred into a labeled 12ml vial, and delivered to the GC department for analysis.

12.0 CALCULATIONS

12.1 Dry weight calculations are determined by weighing an aluminum pan (noting the weight, taring the balance, and then weighing out ~3g of sample (noting the weight). This portion of the sample is dried in a laboratory over at 105 deg C for at least 3 hours and then reweighed. the percent moisture is determined by the following calculation:

(Dry wt.- pan wt.)/wet sample wt= % soil

100 - % soil = % moisture

13.0 ACCEPTANCE OF DATA

13.1 Samples that fail the surrogate recoveries listed in the analytical SOP are re-extracted at the request of the labortory director.

14.0 REPORTING OF RESULTS

N/A

15.0 SUPPLEMENTAL DOCUMENTS

15.1 SOP for Pesticide/PCB analyis (GCS00900.NC).

16.0 REFERENCES

16.1 EPA SW-846, 3rd Edition, Method 3510

17.0 SUBSTANTIVE REVISIONS

Original issue

13. IEA SOP FOR SW-846 3550 PESTICIDES/PCBs SOLID MEDIA EXTRACTION

1.0 APPROVALS

The signature of the following individuals indicates that this SOP is complete and meets the requirements specified in corporate Document # QAS00200.NET. In addition, it also signifies that the content meets the specifications of the referenced "Test Code".

Laboratory Director

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Quality Assurance Manager

2.0 SCOPE AND APPLICATION

2.1 The objective of this document is to outline the techniques used to extract samples for pesticide analysis using method 3550.

2.2 The following test codes are used to define extractions in accordance with all of the conditions set forth by this SOP:

PE_02

2.3 The document control number for this SOP shall be **SPS01500.NC.**

3.0 SUMMARY OF METHOD

3.1 Pre-determined amounts of sample are measured out and extracted using Method 3510. Upon completion of the extraction, the extract is delivered to GC for analysis.

4.0 INTERFERENCES

4.1 Interferences in the pesticide extraction cannot be detected until analysis is complete. All possible precautions are taken to insure interference free extracts.

4.2 All glassware is baked to at least 900 deg. F.

4.3 All glassware is prerinsed with the extraction solvent prior to use.

5.0 SAFETY

5.1 Chemical resistant polyvinyl gloves should be worn at all times when working with solvents.

5.2 Each analyst has been issued a personal lab coat and it should be worn at all times in the sample preparation laboratory area.

5.3 MSDS sheets are available in the lab area for immediate reference in the case of an emergency.

6.0 SAMPLE CONTAINERS, COLLECTION AND PRESERVATION

6.1 Sample Containers

6.1.1 Soil samples are collected with 2x500ml amber glass containers with Teflon coated lid.

6.1.2 Bottles are prepared according to SOP 5.7,"Cleaning Bottles Used for Organic Samples."

6.1.3 Sample bottles are never cleaned and reused.

6.2 Sample Collection

6.2.1 Samples are collected as grab samples in the field.

6.2.2 A cooler with either ice or blue ice should be available at the sampling site.

6.2.3 Samples are secured against breakage in the coolers and kept at 4 deg. C for transport to the laboratory. Samples should arrive at the laboratory the next day following collection.

6.3 Sample Preservation

6.3.1 Samples are preserved by cooling to 4 deg. C.

6.4 Holding Times

6.4.1 Soil samples must be extracted within 14 days from sampling.

6.4.2 All extracts must be analyzed within 40 days from date of extraction.

7.0 APPARATUS AND MATERIALS

7.1 Top-Loader balance

7.2 250ml beakers

7.3 500ml Kuderna-Danish

7.4 3-ball Snyder Column

7.5 10ml concentrator tube

7.6 75mm funnel

7.7 Drying column

7.9 100ml graduated cylinder

7.10 Tekmar Sonicator

7.11 Whatman 41 filter paper

7.12 1000ul gastight syringe for spiking

7.13 12ml glass vials for extract storage.

8.0 REAGENTS AND STANDARDS PREPARATION

8.1 Methylene Chloride - Fisher Optima Grade from an approved lot.

8.2 Hexane - Fisher Optima Grade from a approved lot.

8.3 Surrogate and Matrix Spiking solutions are prepared by the GC staff according to the SOP for Pesticide/PCB analysis.

8.4 Granular Sodium Sulfate - Fisher ACS grade

8.5 Powdered Sodium Sulfate - Baxter ACS grade

8.6 Glass wool

9.0 CALIBRATION

N/A

10.0 QUALITY CONTROL

10.1 Method blanks consist of a 30g portion of sodium sulfate. These blanks are treated exactly the same as other non QC samples and the frequency is one per batch (a batch is defined as a set of samples equal to or less than 20 in number).

10.2 Blank spikes consist of a 30g portion sodium sulfate. These blanks are treated exactly the same as other QC samples (sample MS/MSD) and the frequency is one per batch.

10.3 One sample is chosen at random to use for MS/MSD per batch. The MS/MSD are duplicate extractions of the same sample. The only difference is the additional spiking of the pesticide MS solution.

11.0 SAMPLE PREPARATION AND INSTRUMENTAL PROCEDURES

11.1 Sample extraction

11.1.1 A 30g aliquot of sample is weighed into a 250ml beaker. 60g of powdered sodium sulfate is added to the sample and homogenized. The pesticide surrogate is added at a volume of 1ml per 30g of sample. The pesticide matrix spiking solution is added at a volume of 1ml per 30g of sample in addition to the pesticide surrogate for the sample selected for spiking.

11.1.2 100ml of 50/50 methylene chloride/acetone is added to the sample. The sample is sonicated for 90 seconds, removed from the sonicator and poured through a funnel containing Whatman 41 filter paper into a K-D

setup.

11.1.3 11.1.2 is repeated two more times. The funnel is rinsed down with Methylene Chloride. At this point the extraction is complete and the sample is ready for concentration.

11.2 Sample Concentration

11.2.1 The K-D setup is placed on the steambath. Approximately 2ml of methylene chloride is added to the Snyder Column to initiate boiling. A carbide boiling chip is also added to the K-D setup prior to the placement on the steambath. Boiling should start immediately and should take between 15 and 20 minutes to get the extract to an approximate volume of 15ml. When the extract is at ~15ml, add 50ml of Hexane to the extract and place back on the steambath. The extract should be boiled off until the extract reached an apparent volume of 4ml. The K-D setup should be removed at this point and cooled.

11.2.2 The extract should be brought to a final volume of 0.5ml.

11.2.3 Add 0.5ml of acetone to the concentrator tube containing the 0.5ml of hexane.

11.2.4 The extract is then passed through a pre-made Alumina column (3g activated Alumina placed in a 10ml disposable pipet with a glass wool plug at the bottom).

11.2.5 Hexane is added to the column until the eluent reaches a final volume of 10ml.

11.2.6 The extract should be transferred into a 12ml vial, labeled, and delivered to GC for analysis.

12.0 CALCULATIONS

12.1 Dry weight calculations are determined by weighing an aluminum pan (noting the weight, taring the balance, and then weighing out ~3g of sample (noting the weight). This portion of the sample is dried in a laboratory over at 105 deg C for at least 3 hours and then reweighed. the percent moisture is determined by the following calculation:

(Dry wt.- pan wt.)/wet sample wt= % soil

100 - % soil = % moisture

13.0 ACCEPTANCE OF DATA

13.1 Samples that fail the surrogate recoveries listed in the analytical SOP are re-extracted at the request of the laboratory director.

14.0 REPORTING OF RESULTS

N/A

15.0 SUPPLEMENTAL DOCUMENTS

15.1 SOP for Pesticide/PCB analysis (GCS01000.NC)

16.0 REFERENCES

16.1 EPA SW-846, 3rd Edition, Method 3550

17.0 SUBSTANTIVE REVISIONS

Original issue

14. IEA SOP FOR SW-846 8081 PESTICIDES/PCBs IN WATER

1.0 APPROVALS

The signature of the following individuals indicates that this SOP is complete and meets the requirements specified in corporate Document # QAS00200.NET. In addition, it also signifies that the content meets the specifications of the referenced "Test Code".

Laboratory Director

Quality Assurance Manager

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2.0 SCOPE AND APPLICATION

2.1 This method defines the specific steps for analyzing and determining the concentration of various organochlorine pesticides and polychlorinated biphenyls (PCBs) in environmental water samples.

2.2 Refer to section 11.1 of this SOP for parameters.

2.3 The following test codes are addressed in the SOP: GCP_07, GCP_09, GCP_11, GPC_13, and GPC_17.

3.0 SUMMARY OF METHOD

3.1 This method outlines the gas chromatographic procedure for the detection of organochlorine pesticides and polychlorinated biphenyls. Samples are extracted using the proper extraction technique. The extracts are then screened for dilution and analyzed by gas chromatography with an electron capture detector.

3.2 This SOP is based on the following methods:

- EPA Method 8080 (Organochlorine Pesticides and PCBs)
- EPA Method 8000 (Gas Chromatography)
- EPA Method 608 (Organochlorine Pesticides and PCBs)
- USEPA Contract Laboratory Program (CLP) Statement of Work (SOW) 2/88 and CLP SOW 3/90.

4.0 INTERFERENCES

4.1 - Phthalate esters can interfere with pesticide determination; avoid any contact with plastic to best minimize this problem.

- Sulfur is also an interference; this can be removed by performing sulfur clean-up on the extract using TBA-Sulfite. Refer to the Pesticide Extract Sulfur Removal SOP.

5.0 SAFETY

5.1 All compounds listed in Tables 1-6 (see attached documents) along with their respective solvents (i.e., hexane, isooctane, acetone, toluene) are toxic. Non-powdered polyvinyl gloves and safety glasses should be worn at all times when handling any of these solvents and compounds. Also, all work with these chemicals should be performed in a fume hood.

Material Safety Data Sheets (MSDS) for all chemicals used in operations are present in the laboratory for immediate access. Specific MSDS's should be reviewed for a complete knowledge of the toxicity and precautions of each chemical.

6.0 SAMPLE CONTAINERS, COLLECTION AND PRESERVATION

6.1 Sample Containers

* Water samples are collected with 2x1 liter amber glass containers with Teflon-coated liner.

* Bottles are prepared according to SOP 5.7, "Cleaning Bottles Used for Organic Samples".

* Sample bottles are never cleaned and reused.

6.2 Sample Collection

* Samples are collected as grab samples in the field.

* A cooler with either ice or blue ice should be available at the sampling site.

* Samples are secured against breakage in the coolers and kept at 4 deg. C for transport to the laboratory. Samples should arrive at the laboratory the next day following collection.

6.3 Sample Preservation

* Samples are preserved by cooling to 4 deg. C.

6.4 Holding Times

* Water samples must be extracted within 7 days from sampling.

* All extracts must be analyzed within 40 days from date of extraction.

7.0 APPARATUS AND MATERIALS

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

* DB-608 30 meter
  0.53mm ID 0.83um film thickness
  J&W Scientific CAT #125-1730 or equivalent (ie. Restek RTX-35)

* DB5 30 meter
  0.53mm ID 1.5um film thickness
  J&W Scientific CAT #125-5032 or equivalent (ie. Restek RTX-5)

* DB1701 30 meter
  0.53mm ID 1.0um film thickness
  J&W Scientific CAT #125-0732

* Various sizes of syringes, volumetric pipets, volumetric flasks, pipet bulbs

* 0.8 ml and 1.8 ml autosampler crimptop vials and caps.

* Vial crimper

* Sterile Borosilicate glass transfer pipets/transfer bulbs.

* Safety glasses, non-powdered polyvinyl gloves, fume hood.

* Properly cooled refrigerators each for sample and standard storage.

* Standard and Instrument Maintenance logbooks.

* Sample log-in books.

8.0 REAGENTS AND STANDARD PREPARATION

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

8.2 Commercially prepared stock standards can be used if they are certified and pretested by the manufacturer.

8.3 All stock and working standards are stored in amber screw top bottles at 4 deg. C and replaced after 6 months or earlier if necessary.

8.4 **Calibration Stock Standard** is prepared by diluting 1.0 ml of Accustandard Pesticide Mixture-M-001H New Haven, CT (16 compounds)-, 0.5 ml of Restek Pesticide Surrogate Mix - 200 ug/ml CAT #32000 Bellefonte, PA -, 0.6 ml methoxychlor at 1000 ug/ml and 1.2 ml of Endrin Ketone at 500 ug/ml in a prerinsed 100.0 ml volumetric flask with iso-octane. See Table 1 for final concentrations of compounds.

8.5 **Single-component Calibration Standards** at a minimum of 5 concentration levels are prepared through dilution of the calibration stock standard. One concentration level should be near but above the method detection limit (Refer to EPA Method #608). The remaining concentrations should correspond to the linear range of the instrument. See Table 1 for final component concentrations.

8.6 **Multicomponent Standards** are run at a single concentration level. All of these standards are diluted from individual stocks. Each is run after a single component curve. When a multicomponent target compound is identified within a sample, the appropriate standard is analyzed within a 12 hour time window for quantitation purposes. For Multicomponent Stock and Standard concentrations see Table 2.

8.7 **Surrogate Standard** is prepared to monitor the performance of the extraction and analytical system. Samples, standards and blanks are spiked with pesticide surrogate. Two surrogates, 2,4,5,6-tetrachloro-m-xylene (TCX) and Decachlorobiphenyl (DCB), are spiked with the proper amount to have a level of 0.02 ug/ml in the final extract. (See Table 3).

8.8 **Quality Control Check and Matrix Spiking Standard Solutions** are each prepared by diluting 1.0 ml of Accustandard Pesticide S-444 New Haven, CT -, 1.2 ml of Endrin Ketone at 500 ug/ml, 0.6 ml of Methoxychlor at 1000 ug/ml in a 200.0 mL volumetric flask prerinsed and brought to volume with acetone. 1.0 ml is spiked into each aliquot of the sample for the matrix spike and matrix spike duplicate, and to 1.0 L of reagent water for the QC check sample. (See Table 4).

8.9 **Instrument Breakdown Standard** is prepared as a solution of p,p'DDT and Endrin at a concentration near the midlevel concentration of the calibration standards. (See Table 5).

9.0 CALIBRATION

9.1 Analyze the 5 levels of the single-point calibration standard (Table 1) in the order of low to high, followed by the single levels of multicomponent standards. To verify the calibration, linear curves are generated for each single-component analyte using the method calibration function within the HP3350 software.

A midlevel calibration standard is analyzed and the calculated value is obtained against the curve. The calibration is acceptable if the calculated value of the single component analytes are within (+/-) 15% of the expected value. (Other types of curves, such as point-to-point or quadratic curves, may be used if the response of the analytes on a particular instrument fits better.)

9.2 Daily calibration verification is performed to ensure the instrument calibration is still valid. The daily calibration verification consists of the instrument breakdown standard (Section 8.9), the midlevel calibration standard (Section 8.5.1), and the instrument blank standard which consists of clean solvent spiked with both surrogates at 0.02 ug/ml.

9.2.1 The daily calibration verification is acceptable if all of the following are true.

9.2.1.1 The breakdown of Endrin or p'p'DDT is <= 20% based on the presence of Endrin Aldehyde and Endrin Ketone for Endrin, p,p'DDD and p,p'DDE for p,p'DDT. If the breakdown does exceed 20% then corrective action must be taken prior to continuing the calibration verification.

$$\%\ \text{breakdown for p'p'DDT} = \frac{\text{DDD peak area + DDE peak area}}{\text{Total DDT peak area (DDT+DDD+DDE)}} \times 100$$

$$\%\ \text{breakdown for Endrin} = \frac{\text{Endrin aldehyde peak area + Endrin Ketone peak area}}{\text{Total endrin peak area (Endrin+Endrin aldehyde+Endrin Ketone)}} \times 100$$

9.2.1.2 The midpoint calibration standard must have all compounds (+/-) 15% of their expected value. Because of the low concentration of pesticide standards injected on a GC/EC, column adsorption may be a problem when the GC has not been operated for a day. Therefore, the GC column should be primed by injecting a pesticide standard mixture approximately 20 times more concentrated than the midlevel standard.

If the midpoint calibration standard fails, then it can be rerun. If it fails two consecutive times, for any reason, a new calibration curve is to be run.

9.2.1.3 Instrument blank standard is run after the midlevel calibration standard and prior to any samples to ensure the instrument and its autosampler are clean.

9.3 When analyzing samples, any extract that contains a target compound that exceeds the high level calibration must be diluted to within the calibration range. Multicomponent targets must also be diluted so that the largest peak in the multicomponent does not exceed the response of the largest peak in the high level single-component calibration standard.

9.4 Calculations:

1) Quantitation of single-component target analytes: The calculation of targets is done by the HP3350 software against the calibration curve using the following external standard calculation:

$$\text{Amount of y} = \frac{\text{(Corrected Response)y}}{\text{Vol. Ratio}} \times \frac{\text{Dilution factor}}{100}$$

where:

(Corrected Response)y is the Measured Response (area or height) of peak y after correction by the calibration curve.

Vol. Ratio is the ratio of the volume of sample injected in this run to the volume of calibration mix injected in the calibration run. The default value is 1, meaning that the volumes are the same.

Dilution factor is the percent dilution factor, Default = 100. Any change in extract dilution is entered here.

2) Multi-component analytes are calculated by adding the areas of 5 of the largest peaks common to the sample and the standard and using this combined area in the following calculation:

Samp. Conc. ug/L =

$$\frac{X}{Y} \times \frac{\text{Final Extract volume, ml}}{\text{Initial Samp. volume, L}} \times \text{std. conc. ug/ml} \times \text{Dilution factor}$$

where:

X = the combined area of 5 peaks from sample.
Y = the combined area of 5 peaks from std.

10.0 QUALITY CONTROL

10.1 Refer to Table 6 for Practical Quantitation Limits (PQL's) for all compounds.

10.2 A continuing calibration standard, breakdown standard and instrument blank are initially run daily. The calibration standard must be within (+/-) 15% of expected value versus the curve; breakdown must be < = 20% each for Endrin and p,p'DDT, and the blank must contain no peaks > 1/2 PQL for all compounds.

10.3 Matrix spike (MS) and matrix spike duplicates (MSDs) are analyzed within every set of 10 samples or less. Recoveries must be within the limits listed in Section 13.5 of this SOP. If these criteria are not met, but the blank spike data meet all of the recovery criteria, then the MS/MSD are documented as having matrix interferences. If the blank spike fails, check for instrument and/or column related problems and reanalyze the spikes. If the problem is corrected, the samples are reanalyzed. If these spikes still fail the sample batch is reextracted.

10.4 A QC check sample is analyzed every 10 samples and must pass all single-components within (+/-) 15% of the expected value versus the curve; otherwise, the following samples must be reanalyzed.

10.5 A method blank must be analyzed with each extracted batch of samples. This should have no target compounds above PQL's.

10.6 Surrogates must fall within retention time windows. The %-recovery in samples, spikes, blanks is advisory from 60-150%.

10.7

Daily Calibration Check Sample	One per day
Breakdown Standard	One per day
Instrument Blank	One per day (more if necessary)
MS/MSD	One pair every 20 samples
QC Check	Every 20 samples
Surrogates	Every sample and standard
Method Blank	One per batch
Blank Spike	One per 20 samples

11.0 SAMPLE PREPARATION AND INSTRUMENTAL PROCEDURES

11.1 One HP5890 Series II GC with dual EC's and 7673A Twin Tower Autosampler is currently being used to run Pesticides/PCBs under this SOP. Instruments, columns and conditions are as follows:

a)

Front Injection port:	30 meter DB-1701 (See Section 7.1)
Column flow:	4.0 ml/min He
Detector Make-up Flow:	55 ml/min P-5
Injector Temperature:	Tracks oven temperature +3 deg. C
Detector Temperature:	325 deg. C

Temperature Programming:
Initial Temperature: 160 deg. C Hold 1.0 min.
Initial Ramp: 3 degrees/min to 200 deg. C, Hold 3.0 min.
Final Ramp and Temp.: 15 degrees/min to 270 deg. C, Hold 16 min.

b)

Back Injection Port	RTX-35 (See Section 7.1)
Column flow:	5 ml/min He
Detector Make-up Flow:	55 ml/min P-5
Injector Temperature:	Tracks oven temperature +3 deg. C
Detector Temperature:	325 deg. C

Temperature Programming:
Initial Temperature: 160 deg. C, Hold 1.0 min.
Initial Ramp: 3 degrees/min to 200 deg. C, Hold 3.0 min.
Final Ramp and Temp.: 15 degrees/min to 270 deg. C, Hold 16 min.

11.2 Aqueous sample preparation for pesticides/PCBs:

11.2.1 **Brief Summary:**

A 1 liter sample aliquot is spiked with the surrogate and extracted with methylene chloride at a pH between 5 and 9. Using either the separatory funnel extraction method or the continuous liquid-liquid extraction method is acceptable. The methylene chloride extract is dried and solvent exchanged to hexane and adjusted to a final volume of 10 mL. The sample extraction must be completed within 7 days of sample collection.

A 1 uL aliquot of the sample extract is injected into the gas chromatograph (GC). The compounds are separated by the GC. Compound identification is performed by the comparison of GC retention times to those of known analytical standards. Quantitative analysis is performed by the comparison of compound peak height or peak areas to those of an analytical standard at a known concentration.

11.2.2 **Sample Extraction**

See sample preparation SOP's for Pesticides in Water by Method 8080.

11.3 **Data System:**

HP1000 Analytical Data System with A-400 CPU, running RTE-A Operating System and HP3350A Laboratory Automation System Software for data collection and reduction.

12.0 CALCULATIONS

See Section 9.4.1 & 9.4.2 of this document.

12.1.0 **Calculation of % Difference:**

The following formula is used to calculate % difference in the calculated versus expected values of standards.

$$\% \text{ Difference} = \frac{|\text{Calculated conc.} - \text{expected conc.}|}{\text{Expected conc.}} \times 100$$

12.1.1 **Calculation of spike and QC check recoveries:**

The following calculation is used for spiked sample recoveries.

$$\% \text{ Recoveries} = \frac{\text{Amount recovered}}{\text{Amount added}} \times 100\%$$

13.0 ACCEPTANCE OF DATA

13.1 **Daily Calibration Check Standard.**

Verification of the calibration curve with the midlevel single-component calibration standard is obtained if the calculated concentration of all the compounds is (+/-) 15% of the expected value.

13.2 **Breakdown Check.**

Instrument Breakdown of DDT and Endrin is considered under control if the % Breakdown of each analyte is $<= 20\%$.

13.3 **Instrument Blank.**

The instrument blank is used to verify that the analytical system is free of contaminants. The instrument blank shall be free of any target compounds above quantitation limits and shall not contain any unusual interferences.

13.4 **Method Blanks.**

Method blanks are extracted with every batch of up to 20 samples to ensure that there is no contamination from the extraction process. The method blanks shall be free of any target compounds above quantitation limits and shall not contain any unusual interferences.

13.5 **Matrix Spikes, Matrix Spike Duplicates, and QC Checks.**

MS, MSD, and QC Checks are extracted with every batch of up to 20 samples to verify extraction efficiencies. Acceptance criteria are listed below: (From 40 CFR, Table 3, QC Acceptance Criteria, Method 608)

PARAMETER	RANGE FOR P (%)
Aldrin	42-122
alpha-BHC	37-134
beta-BHC	17-147
delta-BHC	19-140
gamma-BHC	32-127
4,4'-DDD	31-141
4,4'-DDE	30-145
4,4'-DDT	25-160
Dieldrin	36-146
Endosulfan I	45-153
Endosulfan II	D-202
Endosulfan Sulfate	26-144
Endrin	30-147
Heptachlor	34-111
Heptachlor epoxide	37-142

where:

P = % Recovery

D = Detected, result must be greater than 0

14.0 REPORTING OF RESULTS

14.1 Included with the attachments to this SOP is a results sheet that provides information on the following items:

* IEA ID for sample.
* Client ID for sample.
* Date of extraction.
* Date of analysis.
* Surname of analyst.
* List of names of target compounds.
* List of quantitation limits.
* List of concentration data for each compound
* A comments section to describe various events during analysis (e.g., dilution factors, matrix interferences, etc.).
* Units of measure are (ug/L).
* Two significant figures are employed.
* BQL = Below Quantitation Limit. No estimated values are provided unless specifically requested by the client in advance.

14.2 Three different levels of reports are provided for this method:

LEVEL I QC This deliverables package includes data for:

(1) Analytical results for samples.

LEVEL II QC In addition to analytical results for samples, this deliverables package includes data for:

(1) Blank Analysis
(2) Matrix spike/Matrix spike duplicate
(3) Surrogate recoveries
(4) Complete sampling chain-of-custody documentation

LEVEL III QC In addition to analytical results for samples, this deliverables package includes data for:

(1) Method summary
(2) Blank analysis
(3) Matrix spike/Matrix spike duplicate
(4) Surrogate recoveries
(5) Complete sampling and laboratory chain-of-custody documentation
(6) Documentation of extractions and/or dilutions
(7) Second column confirmation as applicable
(8) GC/MS confirmation as applicable, including:

* Tuning data for DFTPP
* Pesticide standard spectra
* Method summary

15.0 SUPPLEMENTAL DOCUMENTS

15.1 SOP for Cleaning Bottles Used for Organic Samples, SOP 5.7, Rev. 3, July 31, 1991.

15.2 SOP for Pesticide Extract Sulfur Removal

15.3 SOP for Pesticides in Water Extraction by Method 8080.

15.4 Tables attached include the following:

(1) Table 1.-Single Component Calibration Concentrations
(2) Table 2.-Multi Component Concentration
(3) Table 3.-Surrogate Mix
(4) Table 4.-QC Check and Matrix Spike
(5) Table 5.-Breakdown Standard Concentration
(6) Table 6.-Practical Quantitation Limits (PQL)

15.5 Pesticides/PCBs EPA 8080 Water Compound List Result Sheet (IEA/NC).

16.0 REFERENCES

16.1 "Method 8000 - Gas Chromatography", EPA SW846, 3RD Edition.

"Method 8080 - Organochlorine Pesticides and PCBs", EPA SW846, 3RD Edition.

"Methods of Organic Chemical Analysis of Municipal and Industrial Wastewater", Federal Register Vol. 49, No. 209, October 26, 1984.

Organic Analysis: Multi-Media, Multi-Concentration IFB-CLP, 2/88.

Organic Analysis: Multi-Media, Multi-Concentration IFB-CLP, 3/90.

17.0 SUBSTANTIVE REVISIONS

17.1 Revisions to this method include:

Multi-component target compounds, such as toxaphene, technical chlordane, and the Aroclors, are analyzed using a single-point calibration. EPA Method 8080 requires at least a five-point calibration for each target compound. Production level work is simply not feasible using multi-level calibrations for the two multi-component pesticides and seven multi-response Aroclors.

An additional revision is that our QC check solution does not routinely contain multi-component pesticides or PCBs due to peak interferences between the multi-component chromatograms and single-component peaks.

TABLE 1 - Single Component Calibration Concentrations, ug/mL

Analyte	Stock	Cal A	Cal B	Cal C	Cal D	Cal E
Aldrin	1.0	0.005	0.01	0.025	0.05	0.10
Dieldrin	2.0	0.01	0.02	0.05	0.10	0.20
p,p'DDT	6.0	0.03	0.06	0.15	0.30	0.60
p,p'DDE	2.0	0.01	0.02	0.05	0.10	0.20
p,p'DDD	6.0	0.03	0.06	0.15	0.30	0.60
Endosulfan I	2.0	0.01	0.02	0.05	0.10	0.20
Endosulfan II	2.0	0.01	0.02	0.05	0.10	0.20
Endosulfan Sulfate	6.0	0.03	0.06	0.15	0.30	0.60
Endrin	2.0	0.01	0.02	0.05	0.10	0.20
Endrin Aldehyde	6.0	0.03	0.06	0.15	0.30	0.60
Heptachlor	1.0	0.005	0.01	0.025	0.05	0.10
Heptachlor Epoxide	1.0	0.005	0.01	0.025	0.05	0.10
alpha-BHC	1.0	0.005	0.01	0.025	0.05	0.10
beta-BHC	1.0	0.005	0.01	0.025	0.05	0.10
gamma-BHC	1.0	0.005	0.01	0.025	0.05	0.10
delta-BHC	1.0	0.005	0.01	0.025	0.05	0.10
Methoxychlor	6.0	0.03	0.06	0.15	0.30	0.60
Endrin Ketone	6.0	0.03	0.06	0.15	0.30	0.60
TCX	1.0	0.005	0.01	0.025	0.05	0.10
DCB	1.0	0.005	0.01	0.025	0.05	0.10

TABLE 2 - Multi-Component Concentration, ug/mL

Multi-Component	Stock	Working Standard
AR 1660	1.0	0.10
AR 1221	1.0	0.20
AR 1232	1.0	0.10
AR 1242	1.0	0.10
AR 1248	1.0	0.10
AR 1254	1.0	0.10
Technical Chlordane	50.0	0.20
Toxaphene	10.0	0.50

TABLE 3 - Surrogate Mix, ug/mL

Surrogate	Stock	Mix for Extractions (added to each sample and spike)
TCX	2.0	0.20
DCB	2.0	0.20

TABLE 4 - QC Check and Matrix Spike, ug/mL

Analyte	Stock	Standard for Extractions
Aldrin	1.0	0.20
Dieldrin	1.0	0.20
p,p'DDT	5.0	1.0
p,p'DDE	1.0	0.2
p,p'DDD	5.0	1.0
Endosulfan I	1.0	0.20
Endosulfan II	5.0	1.0
Endosulfan Sulfate	5.0	1.0
Endrin	5.0	1.0
Endrin Aldehyde	5.0	1.0
Heptachlor	1.0	0.20
Heptachlor Epoxide	1.0	0.20
alpha-BHC	1.0	0.20
beta-BHC	1.0	0.20
gamma-BHC	1.0	0.20
delta-BHC	1.0	0.20
Methoxychlor	10.0	2.0
Endrin Ketone	5.0	1.0

TABLE 5 - Breakdown Std Conc., ug/mL

Compound	Stock (individual compound)	Standard (working)
Endrin	1.0	0.1
p,p'DDT	1.0	0.1

TABLE 6 - Practical Quantitation Limits (PQL)

Analyte	PQL, ug/L	Results, ug/L (2 sig fig)
alpha-BHC	0.05	BQL unless above PQL
beta-BHC	0.05	BQL unless above PQL
delta-BHC	0.05	BQL unless above PQL
gamma-BHC (Lindane)	0.05	BQL unless above PQL
Heptachlor	0.05	BQL unless above PQL
Aldrin	0.05	BQL unless above PQL
Heptachlor Epoxide	0.05	BQL unless above PQL
Endosulfan I	0.05	BQL unless above PQL
Dieldrin	0.10	BQL unless above PQL
p,p'DDE	0.10	BQL unless above PQL
Endrin	0.10	BQL unless above PQL
Endosulfan II	0.10	BQL unless above PQL
p,p'DDD	0.10	BQL unless above PQL
Endosulfar Sulfate	0.10	BQL unless above PQL
p,p'DDT	0.10	BQL unless above PQL
Methoxychlor	0.50	BQL unless above PQL
Toxaphene	1.0	BQL unless above PQL
AR 1016	0.50	BQL unless above PQL
AR 1221	0.50	BQL unless above PQL
AR 1232	0.50	BQL unless above PQL
AR 1242	0.50	BQL unless above PQL
AR 1248	0.50	BQL unless above PQL
AR 1254	1.0	BQL unless above PQL
AR 1260	1.0	BQL unless above PQL
Tech. Chlordane	0.50	BQL unless above PQL
Endrin Aldehyde	0.10	BQL unless above PQL
Endrin Ketone	0.10	BQL unless above PQL

15. IEA SOP FOR SW-846 8081 PESTICIDES/PCBs IN SOIL

1.0 APPROVALS

The signature of the following individuals indicates that this SOP is complete and meets the requirements specified in corporate Document # QAS00200.NET. In addition, it also signifies that the content meets the specifications of the referenced "Test Code".

Laboratory Director

UNCONTROLLED COPY
PROPRIETARY DOCUMENT

Quality Assurance Manager

2.0 SCOPE AND APPLICATION

2.1 This method defines the specific steps for analyzing and determining the concentration of various organochlorine pesticides and polychlorinated biphenyls (PCBs) in environmental soil samples.

2.2 Refer to section 11.1 of this SOP for parameters.

2.3 The following test codes are addressed in the SOP: GCP_10, GCP_12, and GCP_14.

3.0 SUMMARY OF METHOD

3.1 This method outlines the gas chromatographic procedure for the detection of organochlorine pesticides and polychlorinated biphenyls. Samples are extracted using the proper extraction technique. The extracts are then screened for dilution and analyzed by gas chromatography with an electron capture detector.

3.2 This SOP is based on the following methods:

- EPA Method 8080 (Organochlorine Pesticides and PCBs)
- EPA Method 8000 (Gas Chromatography)
- EPA Method 608 (Organochlorine Pesticides and PCBs)
- USEPA Contract Laboratory Program (CLP) Statement of Work (SOW) 2/88 and CLP SOW 3/90.

4.0 INTERFERENCES

4.1 - Phthalate esters can interfere with pesticide determination; avoid any contact with plastic to best minimize this problem.

- Sulfur is also an interference; this can be removed by performing sulfur clean-up on the extract using TBA-Sulfite. Refer to the Pesticide Extract Sulfur Removal SOP.

5.0 SAFETY

5.1 All compounds listed in Tables 1-6 (see attached documents) along with their respective solvents (i.e., hexane, isooctane, acetone, toluene) are toxic. Non-powdered polyvinyl gloves and safety glasses should be worn at all times when handling any of these solvents and compounds. Also, all work with these chemicals should be performed in a fume hood.

Material Safety Data Sheets (MSDS) for all chemicals used in operations are present in the laboratory for immediate access. Specific MSDS's should be reviewed for a complete knowledge of the toxicity and precautions of each chemical.

6.0 SAMPLE CONTAINERS, COLLECTION AND PRESERVATION

6.1 Sample Containers

* Soil samples are collected with 250 ml or 500 ml amber glass containers with Teflon-coated liner.

* Bottles are prepared according to SOP 5.7, "Cleaning Bottles Used for Organic Samples".

* Sample bottles are never cleaned and reused.

6.2 Sample Collection

* Samples are collected as grab samples in the field.

* A cooler with either ice or blue ice should be available at the sampling site.

* Samples are secured against breakage in the coolers and kept at 4 deg. C for transport to the laboratory. Samples should arrive at the laboratory the next day following collection.

6.3 Sample Preservation

* Samples are preserved by cooling to 4 deg. C.

6.4 Holding Times

* Soil samples must be extracted within 14 days from sampling.

* All extracts must be analyzed within 40 days from date of extraction.

7.0 APPARATUS AND MATERIALS

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

* DB-608 30 meter
  0.53mm ID 0.83um film thickness
  J&W Scientific CAT #125-1730 or equivalent (ie. Restek RTX-35)

* DB5 30 meter
  0.53mm ID 1.5um film thickness
  J&W Scientific CAT #125-5032 or equivalent (ie. Restek RTX-5)

* DB1701 30 meter
  0.53mm ID 1.0um film thickness
  J&W Scientific CAT #125-0732

* Various sizes of syringes, volumetric pipets, volumetric flasks, pipet bulbs

* 0.8 ml and 1.8 ml autosampler crimptop vials and caps.

* Vial crimper

* Sterile Borosilicate glass transfer pipets/transfer bulbs.

* Safety glasses, non-powdered polyvinyl gloves, fume hood.

* Properly cooled refrigerators each for sample and standard storage.

* Standard and Instrument Maintenance logbooks.

* Sample log-in books.

8.0 REAGENTS AND STANDARD PREPARATION

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

8.2 Commercially prepared stock standards can be used if they are certified and pretested by the manufacturer.

8.3 All stock and working standards are stored in amber screw top bottles at 4 deg. C and replaced after 6 months or earlier if necessary.

8.4 **Calibration Stock Standard** is prepared by diluting 1.0 ml of Accustandard Pesticide Mixture-M-001H New Haven, CT (16 compounds)-, 0.5 ml of Restek Pesticide Surrogate Mix - 200 ug/ml CAT #32000 Bellefonte, PA -, 0.6 ml methoxychlor at 1000 ug/ml and 1.2 ml of Endrin Ketone at 500 ug/ml in a prerinsed 100.0 ml volumetric flask with iso-octane. See Table 1 for final concentrations of compounds.

8.5 **Single-component Calibration Standards** at a minimum of 5 concentration levels are prepared through dilution of the calibration stock standard. One concentration level should be near but above the method detection limit (Refer to EPA Method #608). The remaining concentrations should correspond to the linear range of the instrument. See Table 1 for final component concentrations.

8.6 **Multicomponent Standards** are run at a single concentration level. All of these standards are diluted from individual stocks. Each is run after a single component curve. When a multicomponent target compound is identified within a sample, the appropriate standard is analyzed within a 12 hour time window for quantitation purposes. For Multicomponent Stock and Standard concentrations see Table 2.

8.7 **Surrogate Standard** is prepared to monitor the performance of the extraction and analytical system. Samples, standards and blanks are spiked with pesticide surrogate. Two surrogates, 2,4,5,6-tetrachloro-m-xylene (TCX) and Decachlorobiphenyl (DCB), are spiked with the proper amount to have a level of 0.02 ug/ml in the final extract. (See Table 3).

8.8 **Quality Control Check and Matrix Spiking Standard Solutions** are each prepared by diluting 1.0 ml of Accustandard Pesticide S-444 New Haven, CT -, 1.2 ml of Endrin Ketone at 500 ug/ml, 0.6 ml of Methoxychlor at 1000 ug/ml in a 200.0 mL volumetric flask prerinsed and brought to volume with acetone. 1.0 ml is spiked into each aliquot of the sample for the matrix spike and matrix spike duplicate, and to 1.0 L of reagent water for the QC check sample. (See Table 4).

8.9 **Instrument Breakdown Standard** is prepared as a solution of p,p'DDT and Endrin at a concentration near the midlevel concentration of the calibration standards. (See Table 5).

9.0 CALIBRATION

9.1 Analyze the 5 levels of the single-point calibration standard (Table 1) in the order of low to high, followed by the single levels of multicomponent standards. To verify the calibration, linear curves are generated for each single-component analyte using the method calibration function within the HP3350 software.

A midlevel calibration standard is analyzed and the calculated value is obtained against the curve. The calibration is acceptable if the calculated value of the single component analytes are within (+/-) 15% of the expected value. (Other types of curves, such as point-to-point or quadratic curves, may be used if the response of the analytes on a particular instrument fits better.)

9.2 Daily calibration verification is performed to ensure the instrument calibration is still valid. The daily calibration verification consists of the instrument breakdown standard (Section 8.9), the midlevel calibration standard (Section 8.5.1), and the instrument blank standard which consists of clean solvent spiked with both surrogates at 0.02 ug/ml.

9.2.1 The daily calibration verification is acceptable if all of the following are true.

9.2.1.1 The breakdown of Endrin or p'p'DDT is <= 20% based on the presence of Endrin Aldehyde and Endrin Ketone for Endrin, p,p'DDD and p,p'DDE for p,p'DDT. If the breakdown does exceed 20% then corrective action must be taken prior to continuing the calibration verification.

$$\%\ \text{breakdown for p'p'DDT} = \frac{\text{DDD peak area + DDE peak area}}{\text{Total DDT peak area (DDT+DDD+DDE)}} \times 100$$

$$\%\ \text{breakdown for Endrin} = \frac{\text{Endrin aldehyde peak area + Endrin Ketone peak area}}{\text{Total endrin peak area (Endrin+Endrin aldehyde+Endrin Ketone)}} \times 100$$

9.2.1.2 The midpoint calibration standard must have all compounds (+/-) 15% of their expected value. Because of the low concentration of pesticide standards injected on a GC/EC, column adsorption may be a problem when the GC has not been operated for a day. Therefore, the GC column should be primed by injecting a pesticide standard mixture approximately 20 times more concentrated than the midlevel standard.

If the midpoint calibration standard fails, then it can be rerun. If it fails two consecutive times, for any reason, a new calibration curve is to be run.

9.2.1.3 Instrument blank standard is run after the midlevel calibration standard and prior to any samples to ensure the instrument and its autosampler are clean.

9.3 When analyzing samples, any extract that contains a target compound that exceeds the high level calibration must be diluted to within the calibration range. Multicomponent targets must also be diluted so that the largest peak in the multicomponent does not exceed the response of the largest peak in the high level single-component calibration standard.

9.4 Calculations:

1) Quantitation of single-component target analytes: The calculation of targets is done by the HP3350 software against the calibration curve using the following external standard calculation:

$$\text{Amount of y} = \frac{\text{(Corrected Response)y}}{\text{Vol. Ratio}} \times \frac{\text{\% Dilution factor}}{100}$$

where:

(Corrected Response)y is the Measured Response (area or height) of peak y after correction by the calibration curve.

Vol. Ratio is the ratio of the volume of sample injected in this run to the volume of calibration mix injected in the calibration run. The default value is 1, meaning that the volumes are the same.

For soil samples, the percent dilution factor is calculated as follows to achieve the correct quantitated result in ug/Kg:

$$\text{\% Dilution Factor} \times \frac{1000}{\text{Dry Weight of Sample (g)}} \times \text{Extract Dilution.}$$

$$\text{- Dry Weight} = \text{Wet Weight Extracted} \times \frac{100 - \text{\% Moisture}}{100}$$

2) Multi-component analytes are calculated by adding the areas of 5 of the largest peaks common to the sample and the standard and using this combined area in the following calculation:

Samp. Conc. ug/Kg =

$$\frac{X}{Y} \times \frac{\text{Final Extract volume, ml}}{\text{Initial Samp. weight, g}} \times \text{std. conc. ug/ml} \times \frac{1000\ g}{1\ Kg} \times \text{Dilution factor}$$

where:

X = the combined area of 5 peaks from sample.
Y = the combined area of 5 peaks from std.

10.0 QUALITY CONTROL

10.1 Refer to Table 6 for Practical Quantitation Limits (PQL's) for all compounds.

10.2 A continuing calibration standard, breakdown standard and instrument blank are initially run daily. The calibration standard must be within (+/-) 15% of expected value versus the curve; breakdown must be < = 20% each for Endrin and p,p'DDT, and the blank must contain no peaks > 1/2 PQL for all compounds.

10.3 Matrix spike (MS) and matrix spike duplicates (MSDs) are analyzed within every set of 10 samples or less. Recoveries must be within the limits listed in Section 13.5 of this SOP. If these criteria are not met, but the blank spike data meet all of the recovery criteria, then the MS/MSD are documented as having matrix interferences. If the blank spike fails, check for instrument and/or column related problems and reanalyze the spikes. If the problem is corrected, the samples are reanalyzed. If these spikes still fail the sample batch is reextracted.

10.4 A QC check sample is analyzed every 10 samples and must pass all single-components within (+/-) 15% of the expected value versus the curve; otherwise, the following samples must be reanalyzed.

10.5 A method blank must be analyzed with each extracted batch of samples. This should have no target compounds above PQL's.

10.6 Surrogates must fall within retention time windows. The %-recovery in samples, spikes, blanks is advisory from 60-150%.

10.7

Daily Calibration Check Sample	One per day
Breakdown Standard	One per day
Instrument Blank	One per day (more if necessary)
MS/MSD	One pair every 20 samples
QC Check	Every 20 samples
Surrogates	Every sample and standard
Method Blank	One per batch
Blank Spike	One per 20 samples

11.0 SAMPLE PREPARATION AND INSTRUMENTAL PROCEDURES

11.1 One HP5890 Series II GC with dual EC's and 7673A Twin Tower Autosampler is currently being used to run Pesticides/PCBs under this SOP. Instruments, columns and conditions are as follows:

a)

Front Injection port:	30 meter DB-1701 (See Section 7.1)
Column flow:	4.0 ml/min He
Detector Make-up Flow:	55 ml/min P-5
Injector Temperature:	Tracks oven temperature +3 deg. C
Detector Temperature:	325 deg. C

Temperature Programming:
Initial Temperature: 160 deg. C Hold 1.0 min.
Initial Ramp: 3 degrees/min to 200 deg. C, Hold 3.0 min.
Final Ramp and Temp.: 15 degrees/min to 270 deg. C, Hold 16 min.

b)

Back Injection Port	RTX-35 (See Section 7.1)
Column flow:	5 ml/min He
Detector Make-up Flow:	55 ml/min P-5
Injector Temperature:	Tracks oven temperature +3 deg. C
Detector Temperature:	325 deg. C

Temperature Programming:
Initial Temperature: 160 deg. C, Hold 1.0 min.
Initial Ramp: 3 degrees/min to 200 deg. C, Hold 3.0 min.
Final Ramp and Temp.: 15 degrees/min to 270 deg. C, Hold 16 min.

11.2 Solid sample preparation for pesticides/PCBs:

11.2.1 **Brief Summary:**

A 30 gram sample aliquot is spiked with the surrogate and extracted with a 50/50 mix of methylene chloride and acetone using the sonication extraction method. The methylene chloride/acetone extract is dried and solvent exchanged to hexane and adjusted to a final volume of 10 mL. The sample extraction must be completed within 14 days of sample collection.

A 1 uL aliquot of the sample extract is injected into the gas chromatograph (GC). The compounds are separated by the GC. Compound identification is performed by the comparison of GC retention times to those of known analytical standards. Quantitative analysis is performed by the comparison of compound peak height or peak areas to those of an analytical standard at a known concentration.

11.2.2 Sample Extraction

See sample preparation SOP's for Pesticides in Solid by Method 8080.

11.3 Data System:

HP1000 Analytical Data System with A-400 CPU, running RTE-A Operating System and HP3350A Laboratory Automation System Software for data collection and reduction.

12.0 CALCULATIONS

See Section 9.4.1 & 9.4.2 of this document.

12.1.0 Calculation of % Difference:

The following formula is used to calculate % difference in the calculated versus expected values of standards.

$$\% \text{ Difference} = \frac{|\text{Calculated conc.} - \text{expected conc.}|}{\text{Expected conc.}} \times 100$$

12.1.1 Calculation of spike and QC check recoveries:

The following calculation is used for spiked sample recoveries.

$$\% \text{ Recoveries} = \frac{\text{Amount recovered}}{\text{Amount added}} \times 100\%$$

13.0 ACCEPTANCE OF DATA

13.1 Daily Calibration Check Standard.

Verification of the calibration curve with the midlevel single-component calibration standard is obtained if the calculated concentration of all the compounds is (+/-) 15% of the expected value.

13.2 Breakdown Check.

Instrument Breakdown of DDT and Endrin is considered under control if the % Breakdown of each analyte is $<= 20\%$.

13.3 **Instrument Blank.**

The instrument blank is used to verify that the analytical system is free of contaminants. The instrument blank shall be free of any target compounds above quantitation limits and shall not contain any unusual interferences.

13.4 **Method Blanks.**

Method blanks are extracted with every batch of up to 20 samples to ensure that there is no contamination from the extraction process. The method blanks shall be free of any target compounds above quantitation limits and shall not contain any unusual interferences.

13.5 **Matrix Spikes, Matrix Spike Duplicates, and QC Checks.**

MS, MSD, and QC Checks are extracted with every batch of up to 20 samples to verify extraction efficiencies. Acceptance criteria are listed below: (From 40 CFR, Table 3, QC Acceptance Criteria, Method 608)

PARAMETER	RANGE FOR P (%)
Aldrin	42-122
alpha-BHC	37-134
beta-BHC	17-147
delta-BHC	19-140
gamma-BHC	32-127
4,4'-DDD	31-141
4,4'-DDE	30-145
4,4'-DDT	25-160
Dieldrin	36-146
Endosulfan I	45-153
Endosulfan II	D-202
Endosulfan Sulfate	26-144
Endrin	30-147
Heptachlor	34-111
Heptachlor epoxide	37-142

where:

P = % Recovery
D = Detected, result must be greater than 0

14.0 REPORTING OF RESULTS

14.1 Included with the attachments to this SOP is a results sheet that provides information on the following items:

* IEA ID for sample.
* Client ID for sample.
* Date of extraction.
* Date of analysis.
* Surname of analyst.
* List of names of target compounds.
* List of quantitation limits.
* List of concentration data for each compound
* A comments section to describe various events during analysis (e.g., dilution factors, matrix interferences, etc.).
* Units of measure are (ug/L).
* Two significant figures are employed.
* BQL = Below Quantitation Limit. No estimated values are provided unless specifically requested by the client in advance.

14.2 Three different levels of reports are provided for this method:

LEVEL I QC This deliverables package includes data for:

(1) Analytical results for samples.

LEVEL II QC In addition to analytical results for samples, this deliverables package includes data for:

(1) Blank Analysis
(2) Matrix spike/Matrix spike duplicate
(3) Surrogate recoveries
(4) Complete sampling chain-of-custody documentation

LEVEL III QC In addition to analytical results for samples, this deliverables package includes data for:

(1) Method summary
(2) Blank analysis
(3) Matrix spike/Matrix spike duplicate
(4) Surrogate recoveries
(5) Complete sampling and laboratory chain-of-custody documentation
(6) Documentation of extractions and/or dilutions
(7) Second column confirmation as applicable
(8) GC/MS confirmation as applicable, including:

* Tuning data for DFTPP
* Pesticide standard spectra
* Method summary

15.0 SUPPLEMENTAL DOCUMENTS

15.1 SOP for Cleaning Bottles Used for Organic Samples, SOP 5.7, Rev. 3, July 31, 1991.

15.2 SOP for Pesticide Extract Sulfur Removal

15.3 SOP for Pesticides in Solid Extraction by Method 8080.

15.4 Tables attached include the following:

(1) Table 1.-Single Component Calibration Concentrations
(2) Table 2.-Multi Component Concentration
(3) Table 3.-Surrogate Mix
(4) Table 4.-QC Check and Matrix Spike
(5) Table 5.-Breakdown Standard Concentration
(6) Table 6.-Practical Quantitation Limits (PQL)

15.5 Pesticides/PCBs EPA 8080 Soil Compound List Result Sheet (IEA/NC).

16.0 REFERENCES

16.1 "Method 8000 - Gas Chromatography", EPA SW846, 3RD Edition.

"Method 8080 - Organochlorine Pesticides and PCBs", EPA SW846, 3RD Edition.

"Methods of Organic Chemical Analysis of Municipal and Industrial Wastewater", Federal Register Vol. 49, No. 209, October 26, 1984.

Organic Analysis: Multi-Media, Multi-Concentration IFB-CLP, 2/88.

Organic Analysis: Multi-Media, Multi-Concentration IFB-CLP, 3/90.

17.0 SUBSTANTIVE REVISIONS

17.1 Revisions to this method include:

Multi-component target compounds, such as toxaphene, technical chlordane, and the Aroclors, are analyzed using a single-point calibration. EPA Method 8080 requires at least a five-point calibration for each target compound. Production level work is simply not feasible using multi-level calibrations for the two multi-component pesticides and seven multi-response Aroclors.

An additional revision is that our QC check solution does not routinely contain multi-component pesticides or PCBs due to peak interferences between the multi-component chromatograms and single-component peaks.

TABLE 1 - Single Component Calibration Concentrations, ug/mL

Analyte	Stock	Cal A	Cal B	Cal C	Cal D	Cal E
Aldrin	1.0	0.005	0.01	0.025	0.05	0.10
Dieldrin	2.0	0.01	0.02	0.05	0.10	0.20
p,p'DDT	6.0	0.03	0.06	0.15	0.30	0.60
p,p'DDE	2.0	0.01	0.02	0.05	0.10	0.20
p,p'DDD	6.0	0.03	0.06	0.15	0.30	0.60
Endosulfan I	2.0	0.01	0.02	0.05	0.10	0.20
Endosulfan II	2.0	0.01	0.02	0.05	0.10	0.20
Endosulfan Sulfate	6.0	0.03	0.06	0.15	0.30	0.60
Endrin	2.0	0.01	0.02	0.05	0.10	0.20
Endrin Aldehyde	6.0	0.03	0.06	0.15	0.30	0.60
Heptachlor	1.0	0.005	0.01	0.025	0.05	0.10
Heptachlor Epoxide	1.0	0.005	0.01	0.025	0.05	0.10
alpha-BHC	1.0	0.005	0.01	0.025	0.05	0.10
beta-BHC	1.0	0.005	0.01	0.025	0.05	0.10
gamma-BHC	1.0	0.005	0.01	0.025	0.05	0.10
delta-BHC	1.0	0.005	0.01	0.025	0.05	0.10
Methoxychlor	6.0	0.03	0.06	0.15	0.30	0.60
Endrin Ketone	6.0	0.03	0.06	0.15	0.30	0.60
TCX	1.0	0.005	0.01	0.025	0.05	0.10
DCB	1.0	0.005	0.01	0.025	0.05	0.10

TABLE 2 - Multi-Component Concentration, ug/mL

Multi-Component	Stock	Working Standard
AR 1660	1.0	0.10
AR 1221	1.0	0.20
AR 1232	1.0	0.10
AR 1242	1.0	0.10
AR 1248	1.0	0.10
AR 1254	1.0	0.10
Technical Chlordane	50.0	0.20
Toxaphene	10.0	0.50

TABLE 3 - Surrogate Mix, ug/mL

Surrogate	Stock	Mix for Extractions (added to each sample and spike)
TCX	2.0	0.20
DCB	2.0	0.20

TABLE 4 - QC Check and Matrix Spike, ug/mL

Analyte	Stock	Standard for Extractions
Aldrin	1.0	0.20
Dieldrin	1.0	0.20
p,p'DDT	5.0	1.0
p,p'DDE	1.0	0.2
p,p'DDD	5.0	1.0
Endosulfan I	1.0	0.20
Endosulfan II	5.0	1.0
Endosulfan Sulfate	5.0	1.0
Endrin	5.0	1.0
Endrin Aldehyde	5.0	1.0
Heptachlor	1.0	0.20
Heptachlor Epoxide	1.0	0.20
alpha-BHC	1.0	0.20
beta-BHC	1.0	0.20
gamma-BHC	1.0	0.20
delta-BHC	1.0	0.20
Methoxychlor	10.0	2.0
Endrin Ketone	5.0	1.0

TABLE 5 - Breakdown Std Conc., ug/mL

Compound	Stock (individual compound)	Standard (working)
Endrin	1.0	0.1
p,p'DDT	1.0	0.1

TABLE 6 - Practical Quantitation Limits (PQL)

Analyte	PQL, ug/L	Results, ug/L (2 sig fig)
alpha-BHC	0.05	BQL unless above PQL
beta-BHC	0.05	BQL unless above PQL
delta-BHC	0.05	BQL unless above PQL
gamma-BHC (Lindane)	0.05	BQL unless above PQL
Heptachlor	0.05	BQL unless above PQL
Aldrin	0.05	BQL unless above PQL
Heptachlor Epoxide	0.05	BQL unless above PQL
Endosulfan I	0.05	BQL unless above PQL
Dieldrin	0.10	BQL unless above PQL
p,p'DDE	0.10	BQL unless above PQL
Endrin	0.10	BQL unless above PQL
Endosulfan II	0.10	BQL unless above PQL
p,p'DDD	0.10	BQL unless above PQL
Endosulfar Sulfate	0.10	BQL unless above PQL
p,p'DDT	0.10	BQL unless above PQL
Methoxychlor	0.50	BQL unless above PQL
Toxaphene	1.0	BQL unless above PQL
AR 1016	0.50	BQL unless above PQL
AR 1221	0.50	BQL unless above PQL
AR 1232	0.50	BQL unless above PQL
AR 1242	0.50	BQL unless above PQL
AR 1248	0.50	BQL unless above PQL
AR 1254	1.0	BQL unless above PQL
AR 1260	1.0	BQL unless above PQL
Tech. Chlordane	0.50	BQL unless above PQL
Endrin Aldehyde	0.10	BQL unless above PQL
Endrin Ketone	0.10	BQL unless above PQL

Industrial & Environmental Analysts, Inc. (IEA)

WATER PESTICIDE
LABORATORY CONTROL SAMPLE (LCS)

IEA Project No.: _____ Date Analyzed: _____
IEA Sample No.: _____ Dilution Factor: _____
IEA QC Sample No.: _____

Compound	SPIKE ADDED (ug/L)	LCS CONCENTRATION (ug/L)	LCS % REC #	%Recovery Limits
alpha-BHC	0.20	XX	0	41-126
gamma-BHC	0.26	XX	0	60-127
Heptachlor	0.20	XX	0	61-111
Aldrin	0.20	XX	0	64-113
beta-BHC	0.20	XX	0	17-147
delta-BHC	0.20	XX	0	19-140
Heptachlor epoxide	0.20	XX	0	76-128
Endosulfan I	0.20	XX	0	87-143
4,4'-DDE	0.20	XX	0	46-145
Dieldrin	0.20	XX	0	75-146
Endrin	1.0	XX	0	58-133
4,4'-DDD	2.0	XX	0	69-141
Endosulfan II	2.0	XX	0	53-152
4,4'-DDT	1.0	XX	0	46-134
Endrin aldehyde	1.0	XX	0	10-200
Methoxychlor	2.0	XX	0	47-142
Endosulfan sulfate	1.0	XX	0	26-144
Endrin ketone	0.50	XX	0	10-200

Comments:
BQL = Below Quantitation Limit
C = Coeluting peaks on column used for quantitation.
Corresponding Samples:

Industrial & Environmental Analysts, Inc. (IEA)

SOIL PESTICIDE
LABORATORY CONTROL SAMPLE (LCS)

IEA Project No.:
IEA Sample No.:
IEA QC Sample No.:

Date Analyzed:
Dilution Factor:

Compound	SPIKE ADDED (ug/Kg)	LCS CONCENTRATION (ug/Kg)	LCS % REC #	%Recovery Limits
alpha-BHC	13	_____	0	43-105
gamma-BHC	13	_____	0	52-108
Heptachlor	13	_____	0	52-111
Aldrin	13	_____	0	42-122
beta-BHC	13	_____	0	63-125
delta-BHC	13	_____	0	19-140
Heptachlor epoxide	13	_____	0	54-121
Endosulfan I	13	_____	0	66-138
4,4'-DDE	13	_____	0	61-143
Dieldrin	13	_____	0	57-146
Endrin	67	_____	0	55-126
4,4'-DDD	67	_____	0	53-127
Endosulfan II	67	_____	0	47-121
4,4'-DDT	67	_____	0	25-160
Endrin aldehyde	67	_____	0	10-200
Methoxychlor	130	_____	0	63-133
Endosulfan sulfate	67	_____	0	26-144
Endrin ketone	67	_____	0	10-200

Comments:
BQL = Below Quantitation Limit
Corresponding Samples:

Industrial & Environmental Analysts, Inc. (IEA)

WATER PESTICIDE
MATRIX SPIKE / MATRIX SPIKE DUPLICATE

IEA Project No.: _____ Date Analyzed: _____
IEA Sample No.: _____ Dilution Factor: _____
IEA QC Sample No.: _____

Compound	SPIKE ADDED (ug/L)	SAMPLE CONCENTRATION (ug/L)	MS CONCENTRATION (ug/L)	MS % REC #	%Recovery Limits
alpha-BHC	0.20	BQL	0.00	0.00	37-134
gamma-BHC	0.26	BQL	0.00	0.00	32-127
Heptachlor	0.20	BQL	0.00	0.00	34-111
Aldrin	0.20	BQL	0.00	0.00	42-122
beta-BHC	0.20	BQL	0.00	0.00	17-147
delta-BHC	0.20	BQL	0.00	0.00	19-140
Heptachlor epoxide	0.20	BQL	0.00	0.00	37-142
Endosulfan I	0.20	BQL	0.00	0.00	45-153
4,4'-DDE	0.20	BQL	0.00	0.00	30-145
Dieldrin	0.20	BQL	0.00	0.00	36-146
Endrin	1.0	BQL	0.00	0.00	30-147
4,4'-DDD	1.0	BQL	0.00	0.00	31-141
Endosulfan II	1.0	BQL	0.00	0.00	10-200
4,4'-DDT	1.0	BQL	0.00	0.00	25-160
Endrin aldehyde	1.0	BQL	0.00	0.00	10-200
ethoxychlor	2.0	BQL	0.00	0.00	25-160
dosulfan sulfate	1.0	BQL	0.00	0.00	26-144
ndrin ketone	1.0	BQL	0.00	0.00	10-200

Comments:
BQL = Below Quantitation Limit
Corresponding Samples:

Industrial & Environmental Analysts, Inc. (IEA)

SOIL PESTICIDE
MATRIX SPIKE / MATRIX SPIKE DUPLICATE

IEA Project No.: _____ Date Analyzed: _____
IEA Sample No.: _____ Dilution Factor: 1.0
IEA QC Sample No.: _____

Compound	SPIKE ADDED (ug/Kg)	SAMPLE CONCENTRATION (ug/Kg)	MS CONCENTRATION (ug/Kg)	MS % REC #	%Recovery Limits
alpha-BHC	16	BQL	0	0	37-134
gamma-BHC	16	BQL	0	0	32-127
Heptachlor	16	BQL	0	0	34-111
Aldrin	16	BQL	0	0	42-122
beta-BHC	16	BQL	0	0	17-147
delta-BHC	16	BQL	0	0	19-140
Heptachlor epoxide	16	BQL	0	0	37-142
Endosulfan I	16	BQL	0	0	45-153
4,4'-DDE	16	BQL	0	0	30-145
Dieldrin	16	BQL	0	0	36-146
Endrin	80	BQL	0	0	30-147
4,4'-DDD	80	BQL	0	0	31-141
Endosulfan II	80	BQL	0	0	10-200
4,4'-DDT	80	BQL	0	0	25-160
Endrin aldehyde	80	BQL	0	0	10-200
Methoxychlor	160	BQL	0	0	25-160
Endosulfan sulfate	80	BQL	0	0	26-144
Endrin ketone	40	BQL	0	0	10-200

Comments:
BQL = Below Quantitation Limit
Corresponding Samples:

Industrial & Environmental Analysts, Inc. (IEA)

WATER SURROGATE RECOVERY
TCX and DCB

IEA Project ID:

IEA Sample No.	Amount added	% RECOVERY TCX	% RECOVERY DCB
____	0.20	____	____
____	0.20	____	____
____	0.20	____	____
____	0.20	____	____
____	0.20	____	____
____	0.20	____	____
____	0.20	____	____
____	0.20	____	____

	Advisory Limits %
TCX: Tetrachloro-m-xylene	60 - 150
DCB: Decachlorobiphenyl	60 - 150

16. IEA SOP FOR SW-846 8151 HERBICIDES IN WATER

1.0 APPROVALS

The signatures of the following individuals indicate that this SOP is complete and meets the requirements specified in corporate document # QAS00200.NET. In addition, it signifies that the content meets the specifications of the referenced "Test Code".

Laboratory Director

Quality Assurance Manager

2.0 SCOPE AND APPLICATION

2.1 This method outlines the gas chromatographic procedure for detecting and determining the concentration of chlorinated phenoxy acid herbicides in environmental water samples.

2.2 See Table 1 for Target Compounds.

2.3 The following test codes are addressed in the SOP:

GCH_01 GCH_04 GCH_05 GCH_07 GCH_09

3.0 SUMMARY OF METHOD

3.1 This method defines the steps for analyzing environmental water samples for chlorinated phenoxy acid herbicides. The samples are acidified, then extracted to remove the herbicide residues from the environmental matrix. The residue is hydrolyzed to an acid and solvent washed to remove extraneous material. The acid is then derivitized to methyl esters which are analyzed by gas chromatography with an electron capture detector.

3.2 This SOP is based on the following methods:

-EPA SW-846 Method 8150 (Chlorinated Herbicides)
-EPA SW-846 Method 8000 (Gas Chromatography)

4.0 INTERFERENCES

4.1 Organic acids, especially chlorinated acids, cause the most direct interference with this method. Phenols, including chlorophenols, may also interfere with this procedure.

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

4.3 The herbicides, being strong organic acids, react readily with alkaline substances and may be lost during extraction or analysis. Glassware, glass wool and sodium sulfate must be acidified with sulfuric acid prior to use to avoid this problem.

5.0 SAFETY

5.1 All compounds listed in Tables 1 - 3 (see attached documents) along with their respective solvents (i.e., hexane, iso-octane, acetone, toluene) and the derivitizing agents used in this method are toxic. Non-powdered polyvinyl gloves and safety glasses should be worn at all times when handling any of these materials. All work with these chemicals should be performed in a fume hood.

5.2 Material Safety Data Sheets (MSDS) for all chemicals used in this procedure are present in the laboratory for immediate access. Specific MSDS should be reviewed for a complete knowledge of the toxicity and precaution of each chemical.

6.0 SAMPLE CONTAINERS, COLLECTION AND PRESERVATION

6.1 Sample Containers

6.1.1 Water samples are collected with 2x1 liter amber glass containers with Teflon-coated liners.

6.1.2 Level 1 certified bottleware is purchased precleaned.

6.1.3 Sample bottles are never cleaned and reused.

6.2 Sample Collection

6.2.1 Samples are collected as grab samples in the field.

6.2.2 A cooler with either ice or blue ice should be available at the sampling site.

6.2.3 Samples are secured against breakage in the coolers and kept at 4 deg. C for transport to the laboratory. Samples should arrive at the laboratory within 24 hours of sample collection.

6.3 Sample Preservation

6.3.1 Samples are preserved by cooling to 4 deg. C.

6.4 Holding Times

6.4.1 Water samples must be extracted within 7 days from sampling.

6.4.2 All extracts must be analyzed within 40 days from the date of extraction.

7.0 APPARATUS AND MATERIALS

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

7.1.1 DB-608 30 meter
0.053 mm ID 0.83 um film thickness
J&W Scientific CAT #125-1730 or equivalent (i.e., Restek RTX-35).

7.1.2 DB-5 30 meter
0.53 mm ID 1.5 um film thickness
J&W Scientific CAT #125-5032 or equivalent (i.e., Restek RTX-5).

7.1.3 DB-1701 30 meter
0.53 mm ID 1.0 um film thickness
J&W Scientific CAT #125-0732.

7.2 Various sizes of syringes, volumetric pipets, volumetric flasks, pipet bulbs.

7.3 0.8 mL and 1.8 mL autosampler crimptop vials and caps.

7.4 Vial crimper.

7.5 Sterile Borosillicate glass transfer pipets/transfer bulbs.

7.6 Safety glasses, non-powdered polyvinyl gloves, fume hood.

7.7 Properly cooled refrigerators each for sample and standard storage.

8.0 REAGENTS AND STANDARD PREPARATION

8.1 Solvents: Hexane, Acetone, Toluene, Iso-octane (2,2,4-trimethylpentane) should be pesticide grade or equivalent.

8.2 Commercially prepared stock standards can be used if they are certified and pretested by the manufacturer. All standards received are documented in the Stock Standard Receipt Logbook (Attachment III).

8.3 All stock and working standards are stored in amber screw top bottles at 4 deg. C and replaced after 6 months or earlier if necessary.

8.4 Calibration stock standard for the extracted herbicide standard is prepared by adding the following to a 100 mL volumetric flask then bringing up to volume with acetone.

2.0 mL 2,4-D (2,4-Dichlorophenoxyacetic Acid) at 1000 ug/mL
400 uL 2,4,5-T (2,4,5-Trichlorophenoxyacetic Acid) at 500 ug/mL
200 uL Silvex (2-(2,4,5-Trichlorophenoxy)propionic Acid) at 1000 ug/mL
2.0 mL 2,4-DB (4-(2,4-Dichlorophenoxy)butyric acid) at 1000 ug/mL

These solutions are purchased from Accustandard, Inc. See Table 1 for final concentrations.

8.5 Single-component Calibration Standards at a minimum of 5 concentration levels are prepared through dilution of the extracted herbicide standard. One concentration level should be near but above the method detection limit. The remaining concentrations should correspond to the linear range of the instrument. See Table 4 for final component concentrations.

8.6 Surrogate standard is prepared to monitor the performance of the extraction and analytical system. Samples, blanks and spikes all have surrogate solution spiked prior to extraction. The herbicide surrogate solution is prepared by dissolving 0.050 g of 2,4-DB (4-(2,4-Dichlorophenoxy)butyric Acid, Aldrich Chemical Co.) in 50 mL of a solvent mixture consisting of 50% iso-octane, 40% Ethyl Acetate and 10% Acetone. From this 1000 ug/mL stock, a working solution at 10.0 ug/mL is prepared by diluting 1.0 mL of the stock to final volume of 100 mL in acetone.

8.7 Matrix Spike solution for the extracted herbicide matrix spike and matrix spike duplicate is prepared by adding the following to a 50 mL volumetric flask then bringing to volume with acetone.

200 uL 2,4-D (2,4-Dichlorophenoxyacetic Acid) at 1000 ug/mL
500 ul 2,4,5-T (2,4,5-Trichlorophenoxyacetic Acid) at 500 ug/mL
40 uL Silvex (2-(2,4,5-Trichlorophenoxy)propionic Acid at 1000 ug/mL

These solutions are purchased from Accustandard, Inc.

8.8 All standard preparations are documented in the Standard Preparation Logbook (Attachment I).

9.0 CALIBRATION

9.1 A herbicide calibration standard is prepared by adding 1.0 mL of the Herbicide standard solution (Section 8.6) to 1.0 L of acidified reagent water. This standard goes through the extraction and esterification process with each batch of samples. The standard is then

diluted to 5 single-component calibration levels. For final standard concentration see Table 4.

9.2 Analyze the 5 levels of the calibration standard in the order of low to high. To verify the calibration, response factors (RF's) are generated for each compound and the linearity (%RSD) of each is checked. If the %RSD is <20%, linearity through the origin is assumed and an average response factor can be used for concentration calculations. If linearity does not meet 20%, then linear curves are generated for each component using the method calibration function within the HP3350 software. The curves are prepared based on an origin included first order linear regression. See Section 12 for the calculations.

9.3 Daily calibration verification is performed to ensure the instrument calibration is still valid. The daily calibration verification consists of the midlevel calibration standard and the instrument blank standard which consists of clean solvent spiked with the surrogate.

9.4 A midlevel calibration standard is analyzed and the calculated value is obtained against the average RF or curve. The calibration is acceptable if the calculated value of the analytes are within +/- 15% of the expected value.

9.4.1 If the midlevel calibration standard fails, then it can be rerun using a fresh calibration standard. If it fails two consecutive times, for any reason, a new calibration curve is run.

9.5 An instrument blank standard is run after the midlevel calibration standard and prior to any samples to ensure the instrument and its autosampler are clean. Blank criteria are defined in Section 10.2.

9.6 When analyzing samples, any extract that contains a target compound that exceeds the high level calibration must be diluted to within the calibration range.

9.7 After calibration verification, the herbicide extracts are set up to run on the gas chromatograph in the following sequence:

Herbicide Mid-level Standard
Instrument Blank
Herbicide Extraction Blank
Samples (up to 10)
Herbicide Mid-level Standard
Samples (up to 10)
LCS*
MS*
MSD*

*These QA samples may also be placed in other locations in the sequence.

The midlevel standard is run after every 10 or fewer samples. All compounds in the standard must yield values (+/-) 15% of their expected value to verify the continued working conditions of the instrument.

If the midlevel standard fails, then it can be rerun. If it fails two consecutive times, for any reason, a new calibration curve is to be run.

10.0 QUALITY CONTROL

10.1 Refer to Table 1 for Practical Quantitation Limits (PQL's) for all compounds.

10.2 A continuing calibration (midlevel) standard and instrument blank are initially run daily. The calibration standard must be within +/- 15% of expected value; and the blank must contain no peaks > PQL for all compounds. If the instrument blank does not meet criteria the autosampler, syringes and solvents must be checked for possible sources of interference and the system demonstrated to be clean before analyses can proceed.

10.3 A Laboratory Control Sample (LCS) is analyzed with every set of 20 samples or less. Recoveries are listed in Table 2. If these criteria are not met, then all samples in the batch are re-extracted for the affected components.

10.4 A Matrix spike (MS) and matrix spike duplicates (MSDs) is analyzed within every set of 20 samples or less. Recoveries are listed on Table 3. If these criteria are not met, but the laboratory control sample (LCS) meets all of the recovery criteria, then the MS/MSD are documented as having matrix interferences. If the LCS fails the criteria listed in Table 2, then all samples in that batch are reextracted.

10.5 A midlevel standard is analyzed every 10 samples and must pass all components within +/- 15% of the expected value; otherwise, the preceding samples must be reanalyzed.

10.6 A method blank must be analyzed with each extracted batch of samples. This should have no target compounds above PQL's. The method blank must be analyzed on each instrument where samples from the batch are analyzed. If the method blank contains target compounds above PQL's, it should be re-analyzed to eliminate the possibility of carry-over from a highly contaminated extract which may have run previously. If the contamination is confirmed the extraction batch associated with the method blank is to be re-extracted and re-analyzed.

10.7 Surrogate recovery limits are 30-140%. When surrogate recovery from a sample or method blank is <30% or >140%, check (1) calculations to locate possible errors, (2) fortifying solutions for degradation, (3) contamination, and (4) instrument performance. If these steps do not reveal the cause of the problem, reanalyze the extract. If sample extract reanalysis meets the surrogate recovery criterion, report only data for the reanalyzed extract. If sample extract reanalysis continues to fail the recovery criterion,

the sample should be re-extracted and re-analyzed to determine if sample matrix caused the surrogate failure.

10.8 Retention Time Windows

10.8.1 Retention time windows are determined by making three injections of all standard mixtures throughout the course of a 72 hour period. Serial injections over less than a 72 hour period result in retention time windows that are too tight.

10.8.2 Calculate the standard deviation of the three retention times for each component. Plus or minus three times the standard deviation of the retention times for each standard will be used to define the retention time window; however, the experience of the analyst should weigh heavily on the interpretation of the chromatograms.

10.8.3 In those cases where the standard deviation for a particular standard is zero, the laboratory must substitute the standard deviation of a close eluting, similar compound to develop a valid retention time window.

10.8.4 The laboratory must calculate retention time windows for each standard on each GC and whenever a new GC column is installed. The data must be retained by the laboratory and a copy provided to the QA department.

10.9 A Method Detection Limit (MDL) determination is required by this method and is performed on an annual basis in accordance with the SOP for Conducting MDL Studies, IEA Doc# QAS02000.NET.

11.0 SAMPLE PREPARATION AND INSTRUMENTAL PROCEDURES

11.1 Two GC's are currently being used to run Herbicides under this SOP.

11.1.1 One HP5890 Series II GC with dual EC's and 7673A Twin Tower Autosampler with columns and conditions as follows:

11.1.1.1	Front Injection port:	30 meter DB-1701 (See Section 7.1)
	Column flow:	4.0 mL/min He
	Detector Make-up Flow	55 mL/min P-5
	Injector Temperature Tracks	oven temperature +3 deg. C
	Detector Temperature	325 deg. C
	Temperature Programming:	
	Initial Temperature:	150 deg. C Hold 3.0 min
	Initial Ramp:	4 deg/min to 220 deg. C, Hold 0.0 min.
	Final Ramp and Temp.:	6 deg./min. to 270 deg. C, Hold 18 minutes

11.1.1.2

Back Injection Port	RTX-35 (See Section 7.1)
Column Flow	4.0 mL/min. He
Detector Make-up Flow:	55 mL/min. P-5
Injector Temperature:	Tracks oven temperature +3 deg. C
Detector Temperature:	325 deg. C
Temperature Programming:	
Initial Temperature:	150 deg. C, Hold 3.0 min.
Initial Ramp:	4 deg./min. to 220 deg. C, Hold 0.0 min.
Final Ramp and Temp:	6 deg./min. to 270 deg. C, Hold 18 min.

11.1.2 One Tracor 540 with a single ECD and a 30 meter RTX-35 (See Section 7.1) with the following conditions:

Column Flow:	5.0 mL/min. He
Detector Make-up Flow	85 mL/min. P-5
Injector Temperature:	225 deg. C
Detector Temperature:	250 deg. C
Temperature Programming:	
Initial Temperature:	180 deg. C, Hold 10 min.
Initial Ramp:	10 deg./min.
Final Temp:	220 deg. C, Hold 5.0 minutes.

11.2 Sample Extraction: See sample preparation SOP for Herbicides in water by Method 8150, IEA Doc# SPS01001.NC.

11.3 Data System:

HP1000 Analytical Data System with A-400 CPU, running RTE-A Operating System and HP3350A Laboratory Automation System Software for data collection and reduction.

11.4 All analysis are documented in the GC Herbicides Runlog (Attachment II).

12.0 CALCULATIONS

12.1 Quantitation using average RF's: the RF for each compound is calculated as

$$\frac{\text{Area of peak}}{\text{Mass injected (ng)}} = \text{RF} \qquad \text{Avg RF} = \frac{RF1+RF2+RF3+RF4+RF5}{5}$$

12.2 Determination of sample concentration (waters)

$$\frac{\text{Area of peak}}{\text{Avg RF}} \times \frac{\text{Final extract vol.}}{\text{Initial sample vol.}} \times \text{Dilution Factor (DF)} = \text{sample conc. ug/L}$$

12.3 Quantitation using calibration curves: The calculation of targets is done by the HP3350 software against the calibration curve using the following calculation:

Quantitation of target analytes: The calculation of targets is done by the HP3350 software against the calibration curve using the following external standard calculation:

$$\text{Amount of y} = \frac{\text{(Corrected Response)y}}{\text{Vol. Ratio}} \times \frac{\text{Dilution factor}}{100}$$

where:

(Corrected Response)y is the Measured Response (area or height) of peak y after correction by the calibration curve.

Vol. Ratio is the ratio of the volume of sample injected in this run to the volume of calibration mix injected in the calibration run. The default value is 1, meaning that the volumes are the same.

Dilution factor is the percent dilution factor, Default = 100. Any change in extract dilution is entered here.

12.4 Calculation of surrogate, LCS, continuing calibration standard recoveries:

$$\%\text{Recovery} = \frac{\text{Amount recovered}}{\text{Amount Added}} \times 100\%$$

12.5 Calculation of % Difference:

$$\%\ \text{Difference} = \frac{|\ \text{Calculated conc. - expected conc.}\ |}{\text{Expected conc.}} \times 100\%$$

13.0 ACCEPTANCE OF DATA

13.1 Verification of the calibration curve with the midlevel single-component calibration standard is obtained if the calculated concentration of all the compounds is within +/- 15% of the expected value.

13.2 The instrument blank is used to verify that the analytical system is free of contaminants. The instrument blank shall be free of any target compounds above quantitation limits and shall not contain any unusual interferences.

13.3 Method blanks are extracted with every batch of up to 20 samples to ensure that there is no contamination from the extraction process. The method blanks shall be free of any target compounds above quantitation limits and shall not contain any unusual interferences.

13.4 Matrix Spike and Matrix Spike Duplicates:
Matrix spike and matrix spike duplicates are extracted and analyzed with every batch of up to 20 samples. Method limits are listed in Table 3.

13.4 Laboratory Control Samples:
Laboratory Control Samples are extracted and analyzed with every batch of up to 20 samples. Method limits are listed in Table 2.

13.6 Midlevel Calibration Standards:
A midlevel calibration standard must be analyzed after every set of 10 samples or less. All analytes in the standard must yield values within (+/-) 15% of their expected value verses the calibration curve.

14.0 REPORTING OF RESULTS

14.1 Units of measure are mg/L
Two significant figures are employed.
BQL = Below Quantitation Limit. No estimated values are provided unless specifically requested by the client in advance.

14.2 A Level I report of results provides information on the following items to the client for samples and method blanks:

IEA ID for sample.
Client ID for sample.
Date of extraction.
Date of analysis.
Surname of analyst.
List of target compounds.
List of quantitation limits.
List of concentration data for each compound.
A comments section to describe various events during analysis (e.g., dilution factors, matrix interferences, etc.).
Copy of Sampling COCs.

14.3 A Level II report includes all data in Section 14.2 plus QC summary reports for surrogate and MS/MSD recovery.

14.4 A Level III report includes all data in Section 14.3 plus all raw data, laboratory COCs and a project narrative.

15.0 SUPPLEMENTAL DOCUMENTS

15.1 SOP for Conducting MDL Studies, IEA Doc# QAS02000.NET.

15.2 SOP for Extracting Herbicide Water Samples by Method 8150, IEA Doc# SPS01001.NC.

16.0 REFERENCES

16.1 "Test Methods for Evaluating Solid Waste", SW-846 Third Edition, September 1986, USEPA, Method 8150.

16.2 "Test Methods for Evaluating Solid Waste", SW-846 Third Edition, September 1986, USEPA, Method 8000.

17.0 SUBSTANTIVE REVISIONS

17.1 This procedure has been revised to include a 5-point calibration curve for each target compound. (10/27/92)

17.2 Logbook reference added in Section 8.2. Addendum to Section 11.2: IEA Doc# added to referenced SOP. Sections 14.3 and 14.4 added to describe Level II and Level III report packages. Section 15.2 revised to reflect current SOP and document control number.

TABLE 1
Practical Quantitation Limits (PQL)

Analyte	PQL, mg/L	Results, mg/L (2 sig.fig.)
Dinoseb	0.0020	BQL unless above PQL
2,4 - D	0.0020	BQL unless above PQL
Silvex	0.00040	BQL unless above PQL
2,4,5 - T	0.0010	BQL unless above PQL

TABLE 2
Laboratory Control Sample Recovery Limits

Analyte	Spike Added, mg/L	Recovery Limits
2,4 - D	0.0040	70 - 120%
Silvex	0.00080	64 - 120%
2,4,5 - T	0.0020	28 - 110%

TABLE 3
Matrix Spike/Matrix Spike Duplicate Recovery Limits

Analyte	Spike Added, mg/L	Recovery Limits	RPD Limits
2,4 - D	0.0040	70 - 120%	35%
Silvex	0.00080	64 - 120%	35%
2,4,5 - T	0.0020	28 - 110%	35%

TABLE 4
Calibration Concentrations, ug/mL

Analyte	LEVEL I	LEVEL II	LEVEL III	LEVEL IV	LEVEL V
2,4-D	0.20	0.40	1.0	1.5	2.0
Silvex	0.020	0.040	0.10	0.15	0.20
2,4,5-T	0.020	0.040	0.10	0.15	0.20
2,4-DB	0.20	0.40	1.0	1.5	2.0

Attachment I

GC Semi-Volatile Runlog

Instrument-*

Method: 3510/3550 **Other:**_____

Curve: _____ **Batch:** _____

Filename	IEA Sample ID.	Date	DF	Dilution Preparation/Comment	Analyst

Surr Fail:___ IS Fail:___ Tot Inj:___ Suc Inj:___ Billables:___

Secondary Review By:_____ Date:_____

Attachment II

STANDARDS LOGBOOK

Standard Name: _____
Location: _____

Lot Code #: _____
Solvent Used: _____
Volume of Solvent Used: _____

Prepared By: _____
Date Prepared: _____
Expiration Date: _____

Stock Name	Vendor	Stock Lot # / Lot Code #	Logbook / Page #	Stock Conc	Amt Added	Final Vol
1.						
2.						
3.						
4.						
5.						

Calculations:

Analyte	Final Analyte Concentration	Analyte	Final Analyte Concentration

Comments: _____

Secondary Review By: _____ **Date:** _____

Attachment III

Stock Standards Receiving Log
GC/GCMS Laboratory

Date Received	Analyst	Standard Name	Stock/Standard Code Lot #	Vendor	Expiration Date	Comments

Secondary Review By:_____ Date:_____

17. QUANTERRA WEST SACRAMENTO, CA (FORMERLY ENSECO) SOP FOR SW-846 8290 DIOXIN/FURAN ANALYSIS

OFFICIAL COPY

STANDARD OPERATING PROCEDURE

Subject or Title: Page 1 of 86
Method 8290-Polychlorinated Dioxins & Furans by HRGC/HRMS

SOP No.:	Revision No.:
LM-CAL-3001	1.0

Supersedes: Original of December 18, 1989

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Prepared By:	Date:
Mark Bechthold/Ron Bobel	June 30, 1993
Management Approval:	Date:
[signature]	3/10/94
QA Officer Approval:	Date:
[signature]	3-10-94

1. SCOPE AND APPLICATION

1.1 This method provides procedures for the detection and quantitative measurement of 2,3,7,8-tetrachlorodibenzo-p-dioxin (2,3,7,8-TCDD), polychlorinated dibenzo-p-dioxins (tetra- through octachlorinated homologs; PCDs), and polychlorinated dibenzofurans (tetra- through octachlorinated homologs; PCDFs) in a variety of environmental matrices and at part-per-trillion (ppt) concentrations. The analytical method calls for the use of high-resolution gas chromatography and high-resolution mass spectrometry (HRGC/HRMS) on purified sample extracts. Table 1 lists the various sample types covered by this analytical protocol, the 2,3,7,8-TCDD-based method calibration limits (MCLs) and other germane information. Analysis of a one-tenth aliquot of the sample permits measurement of concentrations up to 10 times the upper MCL (Table 1). Samples containing concentrations of specific congeners (PCDDs and PCDFs) considered within the scope of this method that are greater than the upper MCL must be analyzed by a protocol designed for such concentrations of specific congeners. An optional method for reporting the analytical results using a 2,3,7,8-TCDD toxicity equivalency factor (TEF) is described.

1.2 The sensitivity of this method is dependent upon the level of interferences within a given matrix.

1.3 This method is designed for use by analysts who are experienced with residue analysis and skilled in high-resolution gas chromatography/high-resolution mass spectrometry (HRGC/HRMS).

2. SUMMARY OF METHOD

2.1 This procedure uses matrix-specific extraction, analyte-specific cleanup, and high-resolution capillary column gas chromatography/high-resolution mass spectrometry (HRGC/HRMS) techniques.

2.2 If interferences are encountered, the method provides selected cleanup procedures to aid the analyst in their elimination. A simplified analysis flow chart is shown in Figure 1 of the Appendix.

2.3 A specified amount (see Table 1) of soil, sediment, fly ash, water, sludge (including paper pulp), still-bottom, fuel oil, chemical reactor residue, or fish tissue, is spiked with a solution containing specified amounts of each of the nine isotopically (^{13}C) labeled PCDDs/PCDFs listed in Column 1 of Table 2. The sample is then extracted according to a matrix-specified extraction procedure. The extraction procedures are: a) toluene (or benzene) Soxhlet extraction for soil, sediment, fly ash samples and aqueous sludges; b) methylene chloride liquid-liquid extraction for water samples; c) dilution of small sample aliquot in hexane for fuel oils and still-bottoms; and d) cyclohexane/methylene chloride extraction for fish tissue; e) ethanol/water extraction for paper pulp. The decision for the selection of an extraction process for chemical reactor residue samples is based on the appearance (consistency, viscosity) of the samples. Generally they can be handled according to the procedure used for still-bottom (or chemical sludge) samples.

2.4 The extracts are submitted to an acid-base washing treatment and dried. Following a solvent exchange step, the residue is cleaned up by column chromatography on acid base silica, acid alumina and carbon on silica. The preparation of the final extract for HRGC/HRMS analysis is accomplished by adding, to the concentrated carbon column eluate, 10 uL (depending on the matrix type) of a tetradecane solution containing 100 pg/uL of each of the two recovery standards ^{13}C-1,2,3,4-TCDD and ^{13}C-,1,2,3,7,8,9-HxCDD (Table 2). The former is used to determine the percent recoveries of tetra-and pentachlorinated PCDD/PCDF congeners while the latter is used for the determination of hexa-, hepta- and octa-chlorinated PCDD/PCDF congeners percent recoveries.

2.5 One to two uL of the concentrated extract are injected into an HRGC/HRMS system capable of performing selected ion monitoring at resolving powers of at least 10,000 (10 percent valley definition).

2.6 The identification of OCDD and nine of the fifteen 2,3,7,8-substituted congeners (Table 3), for which a ^{13}C-labeled standard is available in the sample fortification and recovery standard solutions (Table 2), is based on their elution at their exact retention time (-1 to +3 seconds from the respective internal or recovery standard signal) and simultaneous detection of the two most abundant ions in the molecular ion region. The remaining six 2,3,7,8-substituted congeners (i.e., 2,3,4,7,8-PeCDF, and 1,2,3,4,7,8-HxCDD; 1,2,3,6,7,8-HxCDF; 1,2,3,7,8,9-HxCDF; 2,3,4,6,7,8-HxCDF, and 1,2,3,4,7,8,9-HpCDF), for which no carbon-labeled internal standards are available in the sample fortification solution, and all other identified PCDD/PCDF congeners are identified by their relative retention times falling within their respective PCDD/PCDF retention time windows, as established by using a GC column performance evaluation solution, and the simultaneous detection of the two most abundant ions in the molecular ion region. The identification of OCDF is based on its retention time relative to ^{13}C-OCDD and the simultaneous detection of the two most abundant ions in the molecular ion region. Confirmation is based on a comparison of the ratio of the integrated ion abundance of the molecular ion species to their theoretical abundance ratio.

2.7 Quantification of the individual congeners, total PCDDs and total PCDFs is achieved in conjunction with the establishment of a multipoint (five points) calibration curve for each homolog, during which each calibration solution is analyzed once.

2.8 In some instances, samples may be spiked using 1613 standards at 1613 specified levels, and may be quantitated using a 1613 calibration curve.

3. DEFINITIONS

3.1 Polychlorinated dibenzo-p-dioxins (PCDDS) and polychlorinated dibenzofurans (PCDFs): compounds (Figure 2) that contain from one to eight chlorine atoms. The fifteen 2,3,7,8-substituted PCDDs (totaling 75) and PCDFs (totaling 135) are shown in Table 3. The number of isomers at different chlorination levels is shown in Table 4.

3.2 Homologous series: Defined as a group of chlorinated dibenzodioxins or dibenzofurans having a specific number of chlorine atoms.

3.3 Isomer: Defined by the arrangement of chlorine atoms within an homologous series. For example, 2,3,7,8-TCDD is a TCDD isomer.

3.4 Congener: Any isomer of any homologous series.

3.5 Internal Standard: An internal standard is a ^{13}C-labeled analog of a congener chosen from the compounds listed in Table 3 and of OCDD. Internal standards are added to all samples including method blanks and quality control samples before extraction, and they are used to measure the concentration of the analytes. Nine internal standards are used in this method. There is one for each of the dioxin and furan homologs (except for OCDF) with the degree of chlorination ranging from four to eight.

3.6 Recovery Standard: Recovery standards (two) are used to determine the percent recoveries for the isotopically labeled PCDDs and PCDFs. The ^{13}C-1,2,3,4-TCDD is used to measure the percent recoveries of the tetra- and pentachlorinated dioxins and furans while ^{13}C-1,2,3,7,8,9-HxCDD permits the recovery determination of the hexa-, hepta- and octachlorinated homologs. They are added to the final sample extract before HRGC/HRMS analysis. Furthermore, ^{13}C-1,2,3,7,8,9-HxCDD is used for the identification of the unlabeled analog present in sample extracts.

3.7 High-Resolution Concentration Calibration Solutions (Table 5): Solutions (tetradecane) containing known amounts of 17 selected PCDDs and PCDFs, nine internal standards (^{13}C-labeled PCDDs/PCDFs), and two carbon-labeled recovery standards; the set of five solutions is used to determine the instrument response of the unlabeled analytes relative to the internal standards and of the internal standards relative to the recovery standards.

3.8 Sample Fortification Solution (Table 2): A solution (isooctane or toluene) containing the nine internal standards, which is used to spike all samples before extraction and cleanup.

3.9 Recovery Standard Solution (Table 2): A tetradecane solution containing the two recovery standards, which is added to the final sample extract before HRGC/HRMS analysis.

3.10 Field Blank: A portion of a sample representative of the matrix under consideration, which is free of any PCDDs/PCDFs.

3.11 Laboratory Method Blank: A blank prepared in the laboratory and carried through all analytical procedure steps except the addition of a sample aliquot to the extraction vessel.

3.12 Rinsate: A portion of solvent used to rinse sampling equipment. The rinsate is analyzed to demonstrate that samples were not contaminated during sampling.

3.13 GC Column Performance Check Mixture: A tetradecane solution containing a mixture of selected PCDD/PCDF standards including the first and last eluters for each homologous series, which is used to demonstrate continued acceptable performance of the capillary column (i.e., ≤25 percent valley separation of 2,3,7,8-TCDD from all the other 21 TCDD isomers) and to define the homologous PCDD/PCDF retention time windows.

3.14 Performance Evaluation Materials: Representative sample portions containing known amounts of certain unlabeled PCDD/PCDF congeners (in particular the ones having a 2,3,7,8-substitution pattern). Representative interferences may be present. PEMs are obtained from the EPA EMSL-LV and submitted to potential contract laboratories, must analyze these and obtain acceptable results before being awarded a contract for sample analyses (see IFB Pre-Award Bid Confirmations). PEMs are also included as unspecified ("blind") quality control (QC) samples in any sample batch submitted to a laboratory for analysis.

3.15 Relative Response Factor: Response of the mass spectrometer to a known amount of a native analyte relative to a known amount of an internal standard, or a known amount of internal standard to a known amount of a recovery standard.

3.16 Estimated Level of Method Blank Contamination: The response from a signal occurring in the homologous PCDD/PCDF retention time windows, at any of the masses monitored, is used to calculate the level of contamination in the method blank. The results from such calculations must be reported along with the data obtained on the samples belonging to the batch associated with the method blank.

3.17 Batch: A group of samples processed at the same time.

3.18 Sample Rerun: Extraction of another portion of the sample followed by extract cleanup and extract analysis.

3.19 Extract Reanalysis: Analysis by HRGC/HRMS of another aliquot of the final extract.

3.20 Mass Resolution Check: Standard method used to demonstrate a static resolving power of 10,000 minimum (10 percent valley definition).

3.21 Method Calibration Limits (MCLs): For a given sample size, a final extract volume, and the lowest and highest concentration calibration solutions, the lower and upper MCLs delineate the region of quantification for which the HRGC/HRMS system was calibrated with standard.

3.22 Matrix Spike (MS): A sample which is spiked with a known amount of the matrix spike fortification solution (this exhibit, Section 3.24) prior to the extraction step. The recoveries of the matrix spike compounds are determined; they are used to estimate the effect of the sample matrix upon the analytical methodology.

3.23 Matrix Spike Duplicate (MSD): A second portion of the same sample as used in the matrix spike analysis and which is treated like the matrix spike sample.

3.24 Matrix Spike Fortification Solution: Solution used to prepare the MS and MSD samples. It contains all unlabeled analytes listed in Table 5. The solution also contains all internal standards used in the sample fortification solution per the method.

4. COMMENTS

4.1 Solvents, reagents, glassware and other sample processing hardware may yield discrete artifacts or elevated baselines that may cause misinterpretation of the chromatographic data. All of these materials must be demonstrated to be free from interferents under the conditions of analysis by running laboratory method blanks. Analysts should avoid using PVC gloves.

4.2 The use of high-purity reagents and solvents helps minimize interference problems. Purification of solvents by distillation in all-glass systems may be necessary.

4.3 Reuse of glassware is to be minimized to avoid the risk of contamination.

4.4 Interferents co-extracted from the sample will vary considerably from matrix to matrix. PCDDs and PCDFs are often associated with other interfering chlorinated substances such as polychlorinated biphenyls (PCBs), polychlorinated diphenyl ethers (PCDPEs), polychlorinated naphthalenes, and polychlorinated xanthenes that may be found at concentrations several orders of magnitude higher than the analytes of interest. Retention times of target analytes must be verified using reference standards. These values must correspond to the retention time windows established. While certain clean-up techniques are provided as part of this method, unique samples may require additional cleanup steps to achieve lower detection limits.

4.5 A high-resolution capillary column (60 m DB-5) is used to resolve as many PCDD and PCDF isomers as possible; however, no single column is known to resolve all isomers. The use of several capillary columns will, in fact, be necessary during the determination of the toxicity equivalency factors (TEFs) this exhibit, Section 14.7).

5. SAFETY ISSUES

5.1 2,3,7,8-TCDD is identified as a carcinogen, teratogen, and mutagen. Other PCDDs and PCDFs containing chlorine atoms in positions 2,3,7,8 are known to have toxicities comparable to that of 2,3,7,8-TCDD.

5.2 The analyst should note that finely divided dry soils contaminated with PCDDs and PCDFs are particularly hazardous because of the potential for inhalation and ingestion. Such samples are to be processed in a confined environment, such as a hood or a glove box. Laboratory personnel handling these types of samples should also wear masks fitted with charcoal filter absorbent media to prevent inhalation of dust.

5.3 Safety practices described in Sections 5.4 through 5.5 are adapted from EPA Method 613, Section 4 (July 1982 version).

5.4 The toxicity or carcinogenicity of each reagent used in this method is not precisely defined; however, each chemical compound should be treated as a potential health hazard. From this viewpoint, exposure to these chemicals must be kept to a minimum by whatever means available. The laboratory is responsible for maintaining a current awareness file of OSHA regulations regarding the safe handling of the chemicals specified in this method. A reference file of Material Safety Data Sheets (MSDS) should also be made available to all personnel involved in the chemical analysis. Personnel are expected to read pertinent MSDS's before handling chemicals or samples.

5.5 Each laboratory must develop a strict safety program for the handling of 2,3,7,8-TCDD. The laboratory practices listed below are recommended.

5.5.1 Contamination of the laboratory will be minimized by conducting the manipulations in a fume hood.

5.5.2 The effluents of sample splitters for the gas chromatograph and roughing pumps on the HRGC/HRMS system should pass through either a column of activated charcoal or be bubbled through a trap containing oil or high-boiling alcohols.

5.5.3 Liquid waste should be dissolved in methanol or ethanol and irradiated with ultraviolet light at a wavelength less than 290 nm for several days (use F 40 BL lamps or equivalent). Using this analytical method, analyze the liquid wastes and dispose of the solutions when 2,3,7,8-TCDD can no longer be detected.

5.6 Some of the following precautions were issued by Dow Chemical U.S.A. (revised 11/78) for safe handling of 2,3,7,8-TCDD in the laboratory and amended for use in conjunction with this method.

5.6.1 The following statements on safe handling are as complete as possible on the basis of available toxicological information. The precautions for safe handling and use are necessarily general in nature since detailed, specific recommendations can be made only for the particular exposure and circumstances of each individual use. Assistance in evaluating the health hazards of particular plant conditions may be obtained from certain consulting laboratories and from State Departments of Health or of Labor, many of which have an industrial health service. The 2,3,7,8,-TCDD isomer is extremely toxic to certain kinds of laboratory animals. However, it has been handled for years without injury in analytical laboratories. Techniques used in handling radioactive and infectious materials are applicable to 2,3,7,8-TCDD.

5.6.1.1 Protective Equipment: Throw-away plastic gloves, apron or lab coat, safety glasses and laboratory hood adequate for radioactive work.

5.6.1.2 Training: Workers must be trained in the proper method of removing contaminated gloves and clothing without contacting the exterior surfaces.

5.6.1.3 Personal Hygiene: Thorough washing of hands and forearms after each manipulation and before breaks (coffee, lunch, and shift).

5.6.1.4 Confinement: Isolated work area, posted with signs, segregated glassware and tools, plastic-backed absorbent paper on benchtops.

5.6.1.5 Waste: Good technique includes minimizing contaminated waste. Plastic bag liners should be used in waste cans.

5.6.1.6 Disposal of Hazardous Wastes: Refer to the November 7, 1986 issue of the Federal Register on Land Ban Rulings for details concerning the handling of dioxin-containing wastes.

5.6.1.7 Decontamination: Personnel - any mild soap with plenty of scrubbing action. Glassware, tools and surfaces - Chlorothene NU solvent (Trademark of the Dow Chemical Company) is the least toxic solvent shown to be effective. Satisfactory cleaning may be accomplished rinsing with Chlorothene, then washing with any detergent and water. Dish water may be disposed to the sewer after percolation through a charcoal bed filter. It is prudent to minimize solvent wastes because they require special disposal through commercial sources that are expensive.

5.6.1.8 Laundry: Clothing known to be contaminated should be disposed with the precautions prescribed under "Disposal of Hazardous Wastes." Laboratory coats or other clothing worn in 2,3,7,8-TCDD work area may be laundered. Clothing should be collected in plastic bags.

5.6.1.9 Wipe Tests: A useful method of determining cleanliness of work surfaces and tools is to wipe the surface with a piece of filter paper, extract the filter paper and analyze the extract. (See Appendix)

5.6.1.10 Inhalation: Any procedure that may produce airborne contamination must be carried out with good ventilation. Gross losses to a ventilation system must not be allowed. Handling of the dilute solutions normally used in analytical and animal work presents no significant inhalation hazards except in case of an accident.

5.6.1.11 Accidents: Remove contaminated clothing immediately, taking precautions not to contaminate skin or other articles. Wash exposed skin vigorously and repeatedly until medical attention is obtained.

6. APPARATUS

6.1 High-Resolution Gas Chromatograph/High-Resolution Mass Spectrometer/Data System (HRGC/HRMS/DS).

6.1.1 The GC must be equipped for temperature programming, and all required accessories must be available, such as syringes, gases, and capillary columns. The GC injection port must be designed for capillary columns. The use of splitless injection techniques is recommended. On-Column 1-uL injections can be used on the 60-m DB-5 column. The use of a moving needle injection port is also acceptable. When using the method described in this protocol, a 2-uL injection volume is used consistently (i.e., the injection volumes for all extracts, blanks, calibration solutions and the performance check samples are 2 uL). One-uL injections are allowed; however, laboratories are encouraged to remain consistent throughout the analyses by using the same injection volume at all times.

6.1.2 Gas Chromatograph/Mass Spectrometer (GC/MS) Interface--The GC/MS interface components should withstand 350° C. The interface must be designed so that the separation of 2,3,7,8-TCDD from the other TCDD isomers achieved in the gas chromatographic column is not appreciably degraded. Cold spots or active surfaces (adsorption sites) in the GC/MS interface can cause peak tailing and peak broadening. It is recommended that the GC column be fitted directly into the mass spectrometer ion source without being exposed to the ionizing electron beam. Graphite ferrules should be avoided in the injection port because they may adsorb the PCDDs and PCDFs. Vespel(TM) or equivalent ferrules are recommended.

6.1.3 Mass Spectrometer--The static resolving power of the instrument must be maintained at a minimum of 10,000 (10 percent valley). The mass spectrometer must be operated in a selected ion monitoring (SIM) mode with a total cycle time (including the voltage reset time) of one second or less (this exhibit, Section 9.1.4.1). At a minimum, the ions listed in Table 6 for each of the five SIM descriptors must be monitored. Note that the PeCDF masses (M+2 & M+4) are also monitored in the first descriptor. This is because the first PeCDF isomer elutes prior to elution of the final tetra isomer. The selection (Table 6) of the molecular ions M and M+2 for ^{13}C-HxCDF and ^{13}C-HpCDF rather than M+2 and M+4 (for consistency) is to eliminate, even under high-resolution mass spectrometric conditions, interferences occurring in these two ion channels for samples containing high levels of native HxCDDs and HpCDDs. It is important to maintain the same set of ions for both calibration and sample extract analyses. The selection of the lock-mass ion is left to the performing laboratory. The recommended mass spectrometer tuning conditions (this exhibit, Section 8.2.3) are based on the groups of monitored ions shown in Table 6.

6.1.4 Data System -- A dedicated data system is employed to control the rapid multiple ion monitoring process and to acquire the data. Quantification data (peak areas or peak heights) and SIM traces (displays of intensities of each ion signal being monitored including the lock-mass ion as a function of time) must be acquired during the analyses and stored. Quantifications may be reported based upon computer-generated peak areas or upon measured peak heights (chart recording). The data system must be capable of acquiring data at a minimum of 10 ions in a single scan. It is also recommended to have a data system capable of switching to different sets of ions (descriptors) at specified times during an HRGC/HRMS acquisition. The data system should be able to provide hard copies of individual ion chromatograms for selected gas chromatographic time intervals. It should also be able to acquire mass-spectral peak profiles (this exhibit, Section 8.2.4) and provide hard copies of peak profiles to demonstrate the required resolving power. The data system should also permit the measurement of noise on the base line.

6.2 GC Column

In order to have an isomer-specific determination for 2,3,7,8-TCDD and to allow the detection of OCDD/OCDF within a reasonable time interval in one HRGC/HRMS analysis, the 60-m DB-5 fused-silica capillary column is recommended. Minimum acceptance criteria must be demonstrated and documented (this exhibit, Section 8.1). At the beginning of each 12-hour period (after mass resolution is demonstrated) during which sample extracts or concentration calibration solutions will be analyzed, column operating conditions must be attained for the required separation on the column to be used for samples. Operating conditions known to produce acceptable results with the recommended column are shown in Table 7.

6.3 Miscellaneous Equipment and Materials

The following list of items does not necessarily constitute an exhaustive compendium of the equipment needed for this analytical method.

6.3.1 Nitrogen evaporation apparatus with variable flow rate.

6.3.2 Balances capable of accurately weighing to 0.01 g and 0.0001 g.

6.3.3 Centrifuge.

6.3.4 Water bath, equipped with concentric ring covers and capable of maintaining temperature control within +/- 2° C.

6.3.5 Stainless steel or glass containers large enough to hold contents of one-pint sample containers.

6.3.6 Glove box.

6.3.7 Drying oven.

6.3.8 Stainless steel spoons and spatulas.

6.3.9 Laboratory hoods.

6.3.10 Pipets, disposable, Pasteur, 150 mm long x 5 mm ID.

6.3.11 Pipets, disposable, serological, 10 mL, for the preparation of the carbon column specified in Section 7.1.2.

6.3.12 Reacti-vial, 2 mL, silanized amber glass.

6.3.13 Stainless steel meat grinder with a 3- to 5-mm hole size inner plate.

6.3.14 Separatory funnels, 125 mL.

6.3.15 Kuderna-Danish concentrator, 500 mL, fitted with 10-mL concentrator tube and three-ball Snyder column.

6.3.16 Teflon(TM) boiling chips (or equivalent), washed with DCM before use.

6.3.17 Chromatographic column, glass, 300 mm x 10.5 mm, fitted with Teflon(TM) stopcock.

6.3.18 Adaptors for concentrator tubes.

6.3.19 Glass fiber filters.

6.3.20 Dean-Stark trap, 5 or 10 mL, with T-joints, condenser and 125-mL flask.

6.3.21 Continuous liquid-liquid extractor.

6.3.22 All-glass Soxhlet apparatus, 500-mL flask.

6.3.23 Glass funnels, sized to hold 170 mL of liquid.

6.3.24 Desiccator.

6.3.25 Solvent reservoir (125 mL), Kontes; 12.35 cm diameter (special order item), compatible with gravity carbon column.

6.3.26 Rotary evaporator with a temperature-controlled water bath.

6.3.27 High-speed tissue homogenizer, equipped with an EN-8 probe or equivalent.

6.3.28 Glass wool, extracted with methylene chloride, dried and stored in a clean glass jar.

NOTE: Reuse of glassware should be minimized to avoid the risk of contamination. All glassware that is reused must be scrupulously cleaned as soon as possible after use, applying the following procedure:

6.4 Proper cleaning of glassware is extremely important because glassware may not only contaminate the samples, but may also remove the analytes of interest by absorption on the glassware surface.

6.4.1 Glassware should be rinsed with solvent and washed with a detergent solution as soon after use as is practical. Sonication of glassware containing a detergent solution for approximately 30 seconds may aid in cleaning. Glassware with removable parts, particularly separatory funnels with Teflon stopcocks, must be disassembled prior to detergent washing.

6.4.2 After detergent washing, glassware should be immediately rinsed with acetone, toluene, hexane, and then methylene chloride.

6.4.3 Do not bake reusable glassware in an oven as a routine part of cleaning. Baking may be warranted after particularly dirty samples are encountered, but should be minimized, as repeated baking of glassware may cause the formation of active sites on the glass surface that will irreversibly absorb PCDDs/ PCDFs.

6.4.4 Immediately prior to use, Soxhlet extraction glassware should be pre-extracted with toluene for approximately 3 hours.

7. REAGENTS AND STANDARDS

7.1 Column Chromatography Reagents

7.1.1 Silica Gel - Kieselgel 60 or equivalent, activate for >12 hours at 130°C before use. Store at 130°C in covered flask.

7.1.2 Acid Alumina - Bio-Rad Ag-4 or equivalent, activate for >12 hours at 130°C before use. Store at 130°C in covered flask.

7.1.3 Basic Alumina - Bio-Rad Ag-10 or equivalent, kiln at 600°C for >24 hours before use. Store at 130°C in covered flask. DO NOT USE IF OLDER THAN 5 DAYS!

7.1.4 Carbopack/silica gel - Mix 3.6g carbopack (Supelco 1-0257) and 16.4 g activated silica gel; (alternatively, prepare AX-21/silica gel (5%/95%); i.e., combine 5 g precleaned AX-21 with 95 g silica gel). Activate mix for >12 hours at 130°C before use. Store at 130°C in covered flask.

7.1.5 44% HSO/silica gel - Mix 24mL conc. H_2SO_4 and 56g activated silica gel. Stir and shake until free flowing. Store at room temperature.

7.1.6 33% NaOH/silica gel - Mix 34mL 1N NaOH and 67g activated silica gel. Stir and shake until free flowing. Store at room temperature.

7.2 Reagents

7.2.1 Sulfuric acid, concentrated, ACS grade, specific gravity 1.84.

7.2.2 Potassium hydroxide, ACS grade, 20 percent (w/v) in distilled water.

7.2.3 Distilled water demonstrated to be free of interferents

7.2.4 Potassium carbonate, anhydrous, analytical reagent.

7.2.5 Silica gel.

7.3 Desiccating Agent

7.3.1 Sodium sulfate, granular, anhydrous; use as such.

7.4 Solvents

7.4.1 High-purity, distilled-in-glass or highest available purity: Methylene chloride, hexane, benzene, methanol, tetradecane, isooctane, toluene, cyclohexane, and acetone.

7.5 Calibration Solutions

7.5.1 High-Resolution Concentration Calibration Solutions (Table 5) -- Five tetradecane solutions containing unlabeled (totaling 17) and carbon-labeled (totaling 11) PCDDs and PCDFs at known concentrations used to calibrate the instrument. The concentration ranges are homolog dependent, with the lowest values associated with the tetra chlorinated dioxins and furans (1.0 pg/uL) and the highest for the octachlorinated congeners (1000 pg/uL).

7.5.2 Individual isomers that make up the high-resolution concentration calibration solutions are obtained from commercial sources and prepared in the laboratory. These standards are traceable back to EPA-supplied standard solutions.

7.5.3 Store the calibration solutions in capped test tubes and at room temperature in the dark.

7.6 GC Column Performance Check Solution

This solution contains the first and last eluting isomers for each homologous series from tetra- through hepta-chlorinated congeners. The solution also contains a series of other TCDD isomers for the purpose of documenting the chromatographic resolution. The ^{13}C-2,3,7,8-TCDD is also present. The laboratory is required to use tetradecane as the solvent and adjust the volume so that the final concentration does not exceed 100 pg/uL per congener. Table 8 summarizes the qualitative composition (minimum requirement) of this performance evaluation solution.

7.7 Sample Fortification Solution (Matrix Spike Mix)

This isooctane (or toluene) solution contains the nine internal standards at the nominal concentrations that are listed in Table 2. The solution contains at least one carbon-labeled standard for each homologous series, and it is used to measure the concentrations of the native substances. (Note that ^{13}C-OCDF is not present in the solution.)

7.8 Recovery Standard Solution

This tetradecane solution contains two recovery standards (^{13}C-1,2,3,4-TCDD and ^{13}C-1,2,3,7,8,HxCDD). An appropriate volume of this solution will be spiked into each sample extract before the final concentration step and HRGC/HRMS analysis.

8. SYSTEM PERFORMANCE CRITERIA

System performance criteria are presented below. The laboratory may use the recommended GC column described in Section 6.2. It must be documented that all applicable system performance criteria specified in Section 8.1 were met before analysis of any sample is performed. Table 7 provides recommended GC conditions that can be used to satisfy the required criteria. Figure 4 provides a typical 12-hour analysis sequence. A GC column performance check is only required at the beginning of each 12-hour period during which samples are analyzed.

8.1 GC Column Performance

8.1.1 Inject 2 uL of the column performance check solution and acquire selected ion monitoring (SIM) data as described in Section 6.1.3 within a total cycle time of $\leq$ 1 second.

8.1.2 The chromatographic separation between 2,3,7,8-TCDD and the peaks representing any other TCDD isomers must be resolved with a valley of $\leq$ 25 percent (Figure 5), where

$$\text{Valley Percent} = (x/y)\ (100)$$

x = measured as in Figure 5 from the 2,3,7,8-closest TCDD eluting isomer, and

y = the peak height of 2,3,7,8-TCDD.

It is the responsibility of the laboratory to verify the conditions suitable for the appropriate resolution of 2,3,7,8-TCDD from all other TCDD isomers. The GC column performance check solution also contains the known first and last PCDD/PCDF eluters under the conditions specified in this protocol. Their retention times are used for qualitative and quantitative purposes. The peak for 2,3,7,8-TCDD must be labeled on the chromatograms. The chromatograms showing the first and last eluters of a homologous series must be included.

8.1.3 The retention times for the switching of SIM ions characteristic of one homologous series to the next higher homologous series must be indicated in the SICP. Accurate switching at the appropriate times is absolutely necessary for accurate monitoring of these compounds.

8.2 Mass Spectrometer Performance

8.2.1 The mass spectrometer must be operated in the electron ionization mode. A static resolving power of at least 10,000 (10 percent valley definition) must be demonstrated at appropriate masses before any analysis is performed. Corrective actions must be implemented whenever the resolving power does not meet the requirement.

8.2.2 Chromatography time for PCDDs and PCDFs exceeds the long-term mass stability of the mass spectrometer. Because the instrument is operated in the high-resolution mode, mass drifts of a few ppm (e.g., 5 ppm in mass) can have serious adverse effects on instrument performance. Therefore, a mass-drift correction is mandatory. To that effect, it is recommended to select a lock-mass ion from the reference compound (PFK is recommended) used for tuning the mass spectrometer. The selection of the lock-mass ion is dependent on the masses of the ions monitored within each descriptor. Table 6 offers some suggestions for the lock-mass ions. However, an acceptable lock-mass ion at any mass between the lightest and heaviest ion in each descriptor can be used to monitor and correct mass drifts. The level of the reference compound (PFK) metered into the ion chamber during HRGC/HRMS analyses should be adjusted so that the amplitude of the most intense selected lock-mass ion signal (regardless of the descriptor number) does not exceed 10 percent of the full-scale deflection for a given set of detector parameters. Under those conditions, sensitivity changes that might occur during the analysis can be more effectively monitored.

NOTE: Excessive PFK (or any other reference substance) may cause noise problems and contamination of the ion source resulting in downtime for source cleaning.

8.2.3 By using a PFK molecular leak, tune the instrument to meet minimum required resolving power of 10,000 (10 percent valley) at m/z 304.9824 (PFK) or any other reference signal close to m/z 303.9016 (from TCDF). By using the peak matching unit and the aforementioned PFK reference peak, verify that the exact mass of m/z 380.9760 (PFK) is within 5 ppm of the required value. Note that the selection of the low- and high-mass ions must be such that they provide the largest voltage jump performed in any of the five mass descriptors (Table 6).

8.2.4 Documentation of the instrument resolving power must then be accomplished by recording the peak profile of the high-mass reference signal (m/z 380.9760) obtained during the above peak matching experiment by using the low-mass PFK ion at m/z 304.9824 as a reference. The minimum resolving power of 10,000 must be demonstrated on the high-mass ion while it is transmitted at a lower accelerating voltage than the low-mass reference ion, which is transmitted at full sensitivity. The format of the peak profile representation (Figure 6) must allow manual determination of the resolution, i.e., the horizontal axis must be a calibrated mass scale (amu or ppm per division). The result of the peak width measurement (performed at 5 percent of the maximum, which corresponds to the 10-percent valley definition) must appear on the hard copy and cannot exceed 100 ppm at m/z 380.9760 (or 0.038 amu at that particular mass).

9. CALIBRATION

9.1 Initial Calibration

Initial calibration is required before any samples are analyzed for PCDDs and PCDFs. Initial calibration is also required if any routine calibration (this exhibit, Section 9.3) does not meet the required criteria listed in Section 9.4 (this exhibit).

9.1.1 Five high-resolution concentration calibration solutions, listed in Table 5, must be used for the initial calibration.

9.1.2 Tune the instrument with PFK as described in Section 8.2.3.

9.1.3 Inject 2 uL of the GC column performance check solution and acquire SIM mass spectral data as described earlier in Section 8.1. The total cycle time must be $\leq$ 1 second. The laboratory must not perform any further analysis until it is demonstrated and documented that the criterion listed in Section 8.1.2 is met.

9.1.4 By using the same GC and mass spectrometer conditions that produced acceptable results with the column performance check solution, analyze a 2-uL portion of each of the five concentration calibration solutions once with the following mass spectrometer operating parameter.

9.1.4.1 The total cycle time for data acquisition must be 1 second. The total cycle time includes the sum of all dwell times and voltage reset times.

9.1.4.2 Acquire SIM data for all the ions listed in the five descriptors of Table 6.

9.1.4.3 The ratio of integrated ion current for the ions appearing in Table 9 (homologous series quantification ions) must be within the indicated control limits (set for each homologous series).

9.1.4.4 The ratio of integrated ion current for the ions belonging to the carbon-labeled internal and recovery standards must be within the control limits stipulated in Table 9.

NOTE: Sections 9.1.4.3 and 9.1.4.4 require that 17 ion ratios from Section 9.1.4.3 and 11 ion ratios from Section 9.1.4.4 be within the specified control limits simultaneously in one run. It is the laboratory's responsibility to take corrective action if the ion abundance ratios are outside the limits.

9.1.4.5 For each SICP and for each GC signal corresponding to the elution of a target analyte and of its labeled standards, the signal-to-noise ratio (S/N) must be better than or equal to 10. This measurement is suggested for any GC peak that has an apparent S/N of less than 5:1. The result of the calculation must appear on the SICP above the GC peak in question.

9.1.4.6 Referring to Table 10, calculate the 17 relative response factors (RRF) for unlabeled target analytes [RRF(n); n=1 to 17] relative to their appropriate internal standards (Table 5) and the nine RRFs for the labeled ^{13}C internal standards [RRF(m); m=18 to 26] relative to the two recovery standards according to the following formulae:

$$RRF(n) = \frac{A_x \times Q_{is}}{Q_x \times A_{is}}$$

$$RRF(m) = \frac{A_{is} \times Q_{rs}}{Q_{is} \times A_{rs}}$$

where:

A_x = sum of the integrated ion abundances of the quantitation ions (Tables 6 and 10) for unlabeled PCDDs/PCDFs,

A_{is} = sum of the integrated ion abundances of the quantitation ions (Tables 6 and 10) for the labeled internal standards,

A_{rs} = sum of the integrated ion abundances of the quantitation ions (Tables 6 and 10) for the labeled recovery standards,

Q_{is} = quantity of the internal standard injected (pg),

Q_{rs} = quantity of the recovery standard injected (pg), and

Q_x = quantity of the unlabeled PCDD/PCDF analyte injected (pg).

The RRF(n) and RRF(m) are dimensionless quantities; the units used to express Q_{is}, Q_{rs}, and Q_x must be the same.

9.1.4.7 Calculate the $\overline{RRF}(n)$s and their respective percent relative standard deviations (%RSD) for the five calibration solutions:

$$\overline{RRF}(n) = 1/5 \sum_{j=1}^{5} RRF_j(n)$$

where n represents a particular PCDD/PCDF (2,3,7,8-substituted) congener (n = 1 to 17; Table 10), and j is the injection number (or calibration solution number; j = 1 to 5).

9.1.4.8 The relative response factors to be used for the determination of the concentration of total isomers in a homologous series (Table 10) are calculated as follows:

9.1.4.8.1 For congeners that belong to a homologous series containing only one isomer (e.g., OCDD and OCDF) or only one 2,3,7,8-substituted isomer (Table 4; TCDD, PeCDD, HpCDD, and TCDF), the mean RRF used will be the same as the mean RRF determined in Section 9.1.4.7.

NOTE: The calibration solutions do not contain ^{13}C-OCDF as an internal standard. This is because a minimum resolving power of 12,000 is required to resolve the $[M+6]^+$ ion of ^{13}C-OCDF from the $[M+2]^+$ ion of OCDD (and $[M+4]^+$ from ^{13}C-OCDF with $[M]^+$ of OCDD). Therefore, the RRF for OCDF is calculated relative to ^{13}C-OCDD.

9.1.4.8.2 For congeners that belong to a homologous series containing more than one 2,3,7,8-substituted isomer (Table 4), the mean RRF used for those homologous series will be the mean of the RRFs calculated for all individual 2,3,7,8-substituted congeners using the equation below:

$$\overline{RRF}(k) = \frac{1}{t} \sum_{n=1}^{t} RRF_n$$

where

k = 27 to 30 (Table 10), with 27 = PeCDF; 28 = HxCDF; 29 = HxCDD; and 30 = HpCDF,

t = total number of 2,3,7,8-substituted isomers present in the calibration solutions (Table 5) for each homologous series (e.g., two for PeCDF, four for HxCDF, three for HxCDD, two for HpCDF).

NOTE: Presumably, the HRGC/HRMS response factors of different isomers within a homologous series are different. However, this analytical protocol will make the assumption that the HRGC/HRMS responses of all isomers in a homologous series that do not have the 2,3,7,8-substitution patterns are the same as the responses of one or more of the 2,3,7,8-substituted isomer(s) in that homologous series.

9.1.4.9 Relative response factors [$\overline{RRF}(m)$] to be used for the determination of the percent recoveries for the nine internal standards are calculated as follows:

$$RRF(m) = \frac{A_{is}^{m} \times Q_{rs}}{Q_{is}^{m} \times A_{rs}}$$

$$\overline{RRF}(m) = \frac{1}{5} \sum_{j=1}^{5} RRF_j(m),$$

where:

m = 18 to 26 (congener type) and j = 1 to 5 (injection number),

A_{is}^{m} = sum of the integrated ion abundances of the quantitation ions (Tables 6 and 10) for a given internal standard (m = 18 to 26),

A_{rs} = sum of the integrated ion abundances of the quantitation ions (Tables 6 and 10) for a given internal standard (m = 18 to 26),

Q_{rs} & Q_{is}^{m} = quantities of, respectively, the recovery standard (rs) and a particular internal standard (m) injected (pg),

$RRF(m)$ = relative response factor of a particular internal standard (m) relative to an appropriate recovery standard, as determined from one injection, and

$\overline{RRF}(m)$ = calculated mean relative response factor of a particular internal standard, as determined from the five initial calibration injections (j).

9.2 Criteria For Acceptable Calibration

The criteria listed below for acceptable calibration must be met before the analysis is performed.

9.2.1 The percent relative standard deviations for the mean response factors [RRF(n) and RRF(m)] from the 17 unlabeled standards must not exceed ±20 percent, and those for the nine labeled reference compounds must not exceed ±30 percent.

9.2.2 The signal/noise ratio (S/N) for the GC signals present in every SICP (including the ones for the labeled standards) must be ≥ 10.

9.2.3 The isotopic ratios (Table 9) must be within the specified control limits.

NOTE: If the criterion for acceptable calibration listed in Section 9.2.1 (this exhibit) is met, the analyte-specific RRF can then be considered independent of the analyte quantity for the calibration concentration range. The mean RRFs will be used for all calculations until the routine calibration criteria (this exhibit, Section 9.4) are no longer met. At such time, new mean RRFs will be calculated from a new set of injections of the calibration solutions.

9.3 Routine Calibration (Continuing Calibration Check)

Routine calibrations must be performed at the beginning of a 12-hour period after successful mass resolution and GC resolution performance checks.

9.3.1 Inject 2 uL of the concentration calibration solution HRCC-3 containing 10 pg/uL of tetra-and pentachlorinated congeners, 25 pg/uL of hexa- and heptachlorinated congeners, 50 pg/uL of octachlorinated congeners, and the respective internal and recovery standards (Table 5). By using the same HRGC/HRMS conditions as used in Sections 6.1.3 and 6.2 (this exhibit), determine and document an acceptable calibration as provided in Section 9.4 (this exhibit).

9.4 Criteria for Acceptable Routine Calibration

The following criteria must be met before further analysis is performed. If these criteria are not met, corrective action must be taken.

9.4.1 The measured RRFs [RRF(n) for the unlabeled standards] obtained during the routine calibration runs must be within 20 percent of the mean values established during the initial calibration (this exhibit, Section 9.1.4.7).

9.4.2 The measured RRFs [RRF(m) for the labeled standards] obtained during the routine calibration runs must be within 30 percent of the mean values established during the initial calibration (this exhibit, Section 9.1.4.9).

9.4.3 The ion-abundance ratios (Table 9) must be within the allowed control limits.

9.4.4 If either one of the above criteria (this exhibit, Sections 9.4.1 and 9.4.2) is not satisfied, the entire initial calibration process (this exhibit, Section 9.1) must be repeated. If the ion-abundance ratio criterion (this exhibit, Section 9.4.3) is not satisfied, refer to the note in Section 9.1.4.4 (this exhibit) for resolution.

NOTE: An initial calibration must be carried out whenever the HRCC-3, the sample fortification or the recovery standard solution is replaced by a new solution from a different lot.

10. SAMPLE COLLECTION, PRESERVATION, CONTAINERS, HOLDING TIMES, TREATMENTS, AND ANCILLARY DETERMINATIONS

10.1 The sample collection, shipping, handling, and chain-of-custody procedures are not described in this document. Sample collection personnel will, to the extent possible, homogenize samples in the field before filling the sample containers. This should minimize or eliminate the necessity for sample homogenization in the laboratory. The analyst should make a judgment, based on the appearance of the sample, regarding the necessity for additional mixing. If the sample is clearly non-homogeneous, the entire contents should be transferred to a glass or stainless steel pan for mixing with a stainless steel spoon or spatula before removal of a sample portion for analysis.

10.2 Grab and composite samples must be collected in glass containers.

Conventional sampling practices must be followed. The bottle must not be prewashed with sample before collection. Sampling equipment must be free of potential sources of contamination.

10.3 Grinding or Blending of Fish Samples.

If not otherwise specified by the EPA, the whole fish (frozen) should be blended or ground to provide a homogeneous sample. The use of a stainless steel meatgrinder with a 3- to 5-mm hole size inner plate is recommended. In some circumstances, analysis of fillet or specific organs of fish may be requested by the EPA. If so requested by the EPA, the above whole fish requirement is superseded.

10.4 With the exception of the fish tissues, which must be stored at -20°C, all samples must be stored at 4°C, extracted within 30 days and completely analyzed within 45 days of collection.

10.5 Phase Separation

10.5.1 On a routine basis, very wet soil and sediments may be air dried prior to percent moisture determination and extraction procedures.

10.5.2 Non-routinely, phase separation on very wet (>25 percent water) soil and sediment samples may be accomplished as follows. Place a 50-g portion in a suitable centrifuge bottle and centrifuge for 30 minutes at 2,000 rpm. Remove the bottle and mark the interface level on the bottle. Estimate the relative volume of each phase. With a disposable pipet, transfer the liquid layer into a clean bottle. Mix the solid with a stainless steel spatula and remove a portion to be weighed and analyzed (percent moisture determination, extraction). Return the remaining solid portion to the original sample bottle (empty) or to a clean sample bottle that is properly labeled, and store it as appropriate. Analyze the solid phase by using only the soil and sediment method. Take note of and report the estimated volume of liquid before disposing of the liquid as a liquid waste.

CAUTION: Finely divided soils and sediments contaminated with PCDDs/PCDFs are hazardous because of the potential for inhalation or ingestion of particles containing PCDDs/PCDFs (including 2,3,7,8-TCDD). Such samples should be handled in a confined environment (i.e., a closed hood or a glove box).

10.6 Soil, Sediment or Paper Sludge (Pulp) Percent Moisture Determination.

The percent moisture of soil or sediment samples showing detectable levels (see note below) of at least one 2,3,7,8-substituted PCDD/PCDF congener is determined according to the following recommended procedure.

Generally, depending on sample availability, a 5-10 g sample, weighed to three significant figures, is used for % solids determination. The sample is then dried to constant weight at 100°C in an adequately ventilated oven. Weigh the dried solid to three significant figures. Calculate and report the percent moisture on the appropriate form. Do not use this solid portion of the sample for extraction, but instead dispose of it as hazardous waste.

NOTE: The lower MCLs (Table I) may be used to estimate the minimum detectable levels.

$$\text{Percent Moisture} = \frac{\text{Weight of wet soil - Weight of dry soil}}{\text{Weight of wet soil}} \times 100$$

10.7 Fish Tissue Lipid Content Determination

The percent lipid of fish samples showing detectable levels of at least one 2,3,7,8-substituted PCDD/PCDF congener is determined as follows:

Use a separate portion (2 g) of the ground frozen fish sample. Blend it with 6 g anhydrous sodium sulfate, pour the mixture into a 1-cm i.d. glass column and extract the lipids by passing two 25-mL portions of methylene chloride through the column and collecting the extract in a tared 100-mL round-bottom flask. Concentrate the extract on a rotary evaporator until constant weight is attained. The percent lipid is calculated using the following expression:

$$\text{Percent lipid} = \frac{\text{Weight of residue from extraction (in g)}}{\text{Weight of fish tissue portion (in g)}} \times 100$$

Dispose of the lipid residue as a hazardous waste if the results of the analysis indicate the presence of PCDDs or PCDFS.

11. EXTRACTION AND CLEANUP PROCEDURES

11.1 Internal Standard Addition. Use a portion of 1 g to 1000 g (typical sample size requirements for each type of matrix are given in Section 11.2 of this exhibit and in Table 1) of the sample to be analyzed. Transfer the sample portion to a tared flask and determine its weight. Add an appropriate quantity of the sample fortification mixture to the sample. A 100 uL aliquot of fortification mixture is added to all samples, regardless of sample size. As an example, for ^{13}C-2,3,7,8-TCDD, a 10-g soil sample requires the addition of 1000 pg of ^{13}C-2,3,7,8-TCDD to give the requisite 100 ppt fortification level.

11.2 Extraction

11.2.1 Sludge - Paper Pulp Sludges are generally air-dried and ground. Because of the drying procedure, a Dean-Stark water separator may, or may not, be used for extraction. Extraction is generally done by Soxhlet with 200-300 mL of Ethanol/Toluene 68:32.

Non-Paper Pulp Sludges are extracted with 200-300 mL of toluene.

Soxhlet sample for a minumum of 16 hrs. Cool the sample, filter the toluene (or benzene) extract, if needed, through a glass-fiber filter, or equivalent, into a round-bottom flask. Rinse the filter with 10 mL toluene (or benzene), and combine the extract and rinsate. Concentrate the combined solutions to near dryness on a rotary evaporator at 50°C (toluene) or a Kuderna-Danish (KD) apparatus (benzene). Use of an inert gas to concentrate the extract is also permitted. Proceed with Section 11.2.4. below.

11.2.2 Still-Bottom/Fuel Oil. All organic liquids and solids that will dissolve in a solvent will be treated as a solvent dilution. Dissolve 1-2mL of sample in an appropriate solvent; then dilute with 125mL of Hexane. Spike with appropriate Internal Standards and proceed with section 11.3

11.2.3 Fly Ash. Extract fly ash samples by placing a sample portion (e.g., 10 g) and an equivalent amount of anhydrous sodium sulfate in a Soxhlet extraction apparatus charged with 200-300 mL toluene (or benzene), and extract for 16 hours using a three cycle/hour schedule. Cool and filter the toluene (or benzene) extract through a glass-fiber filter into a 500-mL round-bottom flask. Rinse the filter with 5 mL toluene (or benzene). Concentrate the combined toluene (or benzene) solutions to near dryness on a rotary evaporator (toluene) at 50°C or a KD apparatus (benzene). Proceed with Section 11.2.5.4 below.

11.2.4 Soil. Add anhydrous sodium sulfate to the soil sample portion in a ratio of 2 to 1 (e.g. 20g sodium sulfate to 10g of sample) and mix thoroughly with a stainless steel spatula. After breaking up any lumps, place the soil/sodium sulfate mixture in the Soxhlet apparatus on top of a glass-wool plug (the use of an extraction thimble is optional). Add 200 to 250 mL benzene (or toluene) to the Soxhlet apparatus and reflux for 16 hours. The solvent must cycle completely through the system at least three times per hour. Proceed with Section 11.2.5.4.

11.2.5 Aqueous Samples. Mark the water meniscus on the side of the 1-L sample bottle for later determination of the exact sample volume. Pour the entire sample (approximately 1-L) into a 2-L separatory funnel. Proceed with Section 11.2.5.1 (this exhibit).

NOTE: A continuous liquid-liquid extractor may be used in place of a separatory funnel when experience with a sample from a given source indicates that a serious emulsion problem will result or an emulsion is encountered when using a separatory funnel. Add 60 mL methylene chloride to the sample bottle, seal, and shake for 30 seconds to rinse the inner surface. Transfer the solvent to the extractor. Repeat the sample bottle rinse with an additional 50- to 100-mL portion of methylene chloride and add the rinsate to the extractor. Add 200 to 500 mL methylene chloride to the distilling flask, add sufficient reagent water to ensure proper operation, and extract for 24 hours. Allow to cool, then detach the distilling flask. Proceed with Section 11.2.5.3.

11.2.5.1 Add 60 mL methylene chloride to the sample bottle, seal, and shake for 30 seconds to rinse the inner surface. Transfer the solvent to the separatory funnel and extract the sample by shaking the funnel for two minutes with periodic venting. Allow the organic layer to separate from the water phase for a minimum of 10 minutes. If the emulsion interface between layers is more than one-third the volume of the solvent layer, the analyst must employ mechanical techniques to complete the phase separation. Extraction is repeated two additional times with DCM.

11.2.5.2 Determine the original sample volume by filling the sample bottle to the mark with water and transferring the water to a 1000-mL graduated cylinder. Record the sample volume to the nearest 5 mL.

11.2.5.3 Dry extract with sodium sulfate: Place glass wool in a precleaned filter funnel. Rinse glass wool with DCM and load funnel with DCM-rinsed Na_2SO_4. Pour extract through Na_2SO_4 to remove water. Rinse Na_2SO_4 with fresh DCM and collect in round bottom flask.

11.2.5.4 Transfer the extract to a 500-mL round-bottom, add 100 ul of tetradecane and concentrate on a rotary evaporator or TurboVap.

11.3 Partition the extract in 50-125 mL of hexane against 40 mL concentrated sulfuric acid in a separatory funnel. Shake for two minutes. Remove and discard the sulfuric acid layer (bottom). Repeat the acid washing until no color is visible in the acid layer (perform a maximum of four acid washings).

11.4 Partition the extract against 50 mL distilled H_2O (w/v). Shake for two minutes. Remove and discard the aqueous layer (bottom).

11.5 Partition the extract using 50 ml of 10 Normal Sodium Hydroxide. Shake for two minutes. Remove and discard the aqueous layer (bottom). Repeat the base washing until no color is visible in the bottom layer (perform a maximum of four base washings). Strong base is known to degrade certain PCDDs/PCDFs, so contact time must be minimized.

11.6 Partition the extract against 50 mL of distilled H_2O. Shake for two minutes. Remove and discard the aqueous layer (bottom). Dry the extract by pouring it through a funnel containing anhydrous sodium sulfate and collect it in a round-bottom flask. Rinse the sodium sulfate with two 15-mL portions of hexane, add the rinsates to the flask, and concentrate the hexane solution to near dryness on a rotary evaporator (35°C water bath), making sure all traces of toluene (when applicable) are removed. (Use of blow-down with an inert gas to concentrate the extract is also permitted.)

11.7 Proceed with the following Clean-up Steps:

For NON paper pulp mill solids & effluents proceed with clean-up step exhibit "A".

For paper pulp mill solids & effluents proceed with exhibit "B".

EXHIBIT A

IFB COLUMN CLEANUP

Use 15 mm column for top column
Use 11 mm column for bottom column

	# of 15mL capsfull
1 cm Na_2SO_4	1
2g Silica gel	2
4g 44% H_2SO_4/Silica gel	2
1g Silica gel	1
2g 33% 1M NaOH/silica gel	1
1g Silica gel	1
glasswool	

1 cm Na_2SO_4

8g Acid alumina

glasswool

-Pre-rinse both columns with hexane 40 mL Top and 20 mL Bottom
-Put one column above the other
-Add extract to the top column - Rinse Extract Vessel 2 times with 1 mL ea. of Hexane and add to column.
-Elute the top column directly onto the bottom column with 90mL hexane
-Elute the bottom column with 20 mL of hexane - discard in proper waste stream
-Elute with 20mL of 20% $MeCl_2$/hexane
-Collect eluate in Turbo vap tube
-Use tetradecane and N_2 as appropriate per flow chart

Proceed with 11.8

EXHIBIT B

NCASI Paper Pulp IFB COLUMN CLEANUP

Use 15 mm column for top column
Use 11 mm column for bottom column

	# of 15mL capsfull
1 cm Na_2SO_4	1
2g Silica gel	2
8g 44% H_2SO_4/Silica gel	4
1g Silica gel	1
4g 33% 1M NaOH/silica gel	2
1g Silica gel	1
glasswool	

1 cm Na_2SO_4

8g Acid alumina

glasswool

-Pre-rinse both columns with hexane 40 mL Top and 20 mL Bottom
-Put one column above the other
-Add extract to the top column w/hexane - Rinse Extract Vessel 2 times with 1 mL ea. of Hexane and add to column
-Elute the top column directly onto the bottom column with 120mL hexane (60 x 2)
-Elute the bottom column with 20 mL of hexane - discard in proper waste stream
-Elute with 23mL of 20% $MeCl_2$/hexane
-Collect eluate in Turbo vap tube
-Use tetradecane and N_2 as appropriate per flow chart

11.8 Carbon Column Cleanup - Prepare an AX-21 Carbon & Silica Gel column as described in EXHIBIT D, Page 41.

EXHIBIT D
CARBON COLUMN CLEANUP
SPECIAL D2

-Cut off both ends of a 10 mL pipet.
-Push a glasswool plug down to the 6 mL mark.
-Add 1g of 5% AX-21/silica and top with another glasswool plug.

-Pre-elute with 5 mL 1:1 $MeCl_2$:cyclohexane. Direction "A"
-Turn over and pre-elute with 5 mL 1:1 MeCl:cyclohexane in direction "B"..
-Discard pre-eluates.

-Dilute extract to 1 mL with hexane and transfer to the column.
-Rinse sample vial onto the column with 2 x 2 mL 1:1 $MeCl_2$:cyclohexane

-Elute with: 6mL 1:1 $MeCl_2$:cyclohexane
5mL 75:20:5 $MeCl_2$:MeOH:Benzene

-DISCARD ELUATES

-Turn the column over and elute with 25 mL toluene in direction "A".

-N_2 or roto-vap to NEAR dryness and proceed to next step.

11.9 Extraction and Purification Procedures for Biota (Stallings SOP)

Column #1 & #2 - Use approximately 850 mL of Solvent A (1:1 cyclohexane:DCM) per sample: 700 mL through the column, 100 mL into the jar with the sample before spiking, and 50 mL to rinse the jar.

NOTE: (Add 30 g anhydrous sodium sulfate to a 10-g portion of a homogeneous tissue sample and mix thoroughly with a stainless steel spatula. After breaking up any lumps, prepare to add the tissue/sodium sulfate mixture to the top of column). Use a 2 mL pipet to stir up the sample. Then pour the spiked sample through a large funnel onto the #1 dry column, using 50mL to rinse the sample jar. Keep the small column wet with Solvent A. When the solvent reaches the top cap of the small column (#2), fill the small column to the top and place the cap on tightly. When the Solvent A reaches the top of the solid material in the column, pour 700 mL of solvent A (in two portions) onto the column. Drain into a 1 L clear glass jar (CGJ).

2 cm Na_2SO_4

column #2 cap

30 g
K silicate

30 g
silica gel

15 g K silicate

7 g silica gel

2 cm Na_2SO_4

Column #1: Flex Column number 420400-2550
Column #2: Flex Column number 420400-1530

Disposal of columns: Air out the columns in a hood. Take the used columns #1 & #2 and place them in a doubled garbage bag. Seal with red tape and dispose of in the lab pack.

Column #3 - Close the stopcock. Put glasswool in the bottom of the column. Weigh 10 g of AX-21/silica gel in a 150 mL beaker, add 75 mL of toluene, mix, and pour into column #3. Rinse beaker with toluene. After the packing settles, drain the toluene down to the top of the packing material. Rinse the sides of the column with MeOH and drain down to 2 cm above the top of the packing material. Put a plug of glasswool on top of the column. Condition the column with 80 mL of MeOH, dicard the eluant. Then elute the column with 80 mL of Solvent A and discard.

Add Cleanup Recovery Standard (CRS) to the samples. Then add entire extract from column 2 to the top of column 3. Drain samples through the column and discard the eluate. Drain 75 mL of Solvent A through the column, discard. Drain 50 mL 75:20 DCM:MeOH through the column, discard.

Turn the column over and put a filter funnel and 250 mL round bottom flask containing 100 uL C$_{14}$ under the column. Insert the reservoir column and fill with 75ml of Toluene. Elute the packed column, collecting the toluene in the 250 mL round bottm flask. Carefully blow out the toulene in the packing material with a squeeze bulb.

glasswool

10 g PX-21

glasswool

Proceed with columns #4 and #5

Column #4 & #5 - Use a 5 mL pipet with a glasswool plug to estimate the acid alumina for column #5. Place the plug at the 4.5 mL mark and measure up to the Zero mark, making sure to pack the material by tapping on the pipet. Once you have 4.5 mL of acid alumina, pour into column #5. Add a little over 1/4 inch of DCM rinsed Na_2SO_4 and place the cap back on the column. Insert a pasteur pipet containing a small glass woolplug (column #4) into the small hole in the cap of column #5. Add about 1 cm of acid silica into column #4. Add about 1 cm K-silicate on top of the acid silica.

Set up test tube rack, raised with a 1 inch board, with 8 mL test tubes and 40 mL VOA vials. The first 40 mL VOA vial is for the 23 mL of pre-rinse, the 8 mL test tube is for the post-rinse, and the 2nd 40 mL VOA vial is for the 37 mL of remaining eluate. Label the containers with the appropriate CAL ID#.

At this point, sample extracts from column 3 should be roto-vap'd down to the tetradecane and brought up to 5 mL in hexane. Set up samples (250 mL round bottom) in front of the columns.

Solvents: 2% DCM:Hexane
5% DCM:Hexane
8% DCM:Hexane
Hexane

Place first 40 mL VOA vial underneath each of the column #5's. Transfer all of sample to column #4. Fill column #4 to the top with hexane. Add another 5 mL to the sample round bottom. After the solvent in column #4 reaches the Na_2SO_4, transfer the 5 mL in the round bottom onto the column. Blow out column #4 using a red pipette bulb (discard column #4). Rinse the reusable reservoir (yellow top) with hexane and insert it into the top of column #5. Add 10 mL of 2% solvent. Transfer the labels from the round bottom to the second 40 mL VOA vial.

The first VOA vial should contain two 5 mL hexane rinses and the 10 mL of 2% solvent. Cap and archive the rinses.

Place the second 40 mL VOA vial under the columns; eluate and collect:
Add 5 mL 2% solvent
Add 15 mL 5% solvent
Add 20 mL 8% solvent

Remove the VOA vial. Place a labeled 8 mL test tube under the column #5 and elute with 5 mL of DCM. Archive as a post rinse. Label test tube with the appropriate CAL ID#.

Reduce the volume of the second VOA vial with N_2 to a small puddle. Transfer the sample to a 2 mL conical vial including 0.5 mL rinse of the VOA vial. Do not blow the sample to dryness in the 40 mL VOA vial.

Column #4: Pasteur pipet
Column #5: Flex Column number 420400-0730

Proceed with 12.1

12. ANALYTICAL PROCEDURES

12.1 With a stream of dry, purified nitrogen, reduce the extract volume to 10 uL. Add 10 uL of the recovery standard solution (Table 2).

12.2 Inject a 1 or 2uL aliquot of the extract into the GC, operated under the conditions previously used (this exhibit, Section 6.2) to produce acceptable results with the performance check solution.

12.3 Acquire SIM data according to Section 6.1.3 (this exhibit). Use the same acquisition and mass spectrometer operating conditions previously used to determine the relative response factors (this exhibit, Sections 9.1.4.6 through 9.1.4.9). Ions characteristic for polychlorinated diphenyl ethers are included in the descriptors listed in Table 6. Their presence is to monitor their interference during the characterization of PCDFs.

12.4 Identification Criteria

For a gas chromatographic peak to be identified as a PCDD or PCDF, it must meet all of the following criteria:

12.4.1 Retention Times.

12.4.1.1 For 2,3,7,8-substituted congeners, which have an isotopically labeled internal or recovery standard present in the sample extract (this represents a total of 10 congeners including OCDD; Tables 2 and 3), the retention time (at maximum peak height) of the sample components (i.e., the two ions used for quantitation purposes listed in Table 6) must be within -1 and +3 seconds of the retention time of the peak for the isotopically labeled internal or recovery standard at m/z corresponding to the first characteristic ion (of the set of two; Table 6) to obtain a positive identification of these nine 2,3,7,8-substituted PCDDs/PCDFs and OCDD.

12.4.1.2 For 2,3,7,8-substituted compounds that do not have an isotopically labeled internal standard present in the sample extract (this represents a total of six congeners), the relative retention time (relative to the appropriate internal standard) must fall within 0.005 relative retention time units of the relative retention times measured in the daily routine calibration. Identification of OCDF is based on its retention time relative to ^{13}C-OCDD as determined from the daily routine calibration results.

12.4.1.3 For non-2,3,7,8-substituted compounds (tetra through octa; totaling 119 congeners), the retention time must be within the corresponding homologous retention time windows established by analyzing the column performance check solution.

12.4.1.4 The ion current responses for both ions used for quantitative purposes (e.g., for TCDDs: m/z 319.8465 and 321.8936) must reach a maximum simultaneously (± 2 seconds).

12.4.1.5 The ion current responses for both ions used for the labeled standards (e.g., for ^{13}C-TCDD: m/z 331.9368 and m/z 333.9339) must reach a maximum simultaneously (± 2 seconds).

12.4.2 Ion Abundance Ratios

12.4.2.1 The integrated ion current for the two ions used for quantitation purposes must have a ratio between the lower and upper limits established for the homologous series to which the peak is assigned. See Sections 9.1.4.3 and 9.1.4.4 (this exhibit) and Table 9 for details.

12.4.3 Signal-To-Noise Ratio

12.4.3.1 All ion current intensities must be ≥2.5 times noise level for positive identification of the PCDD/PCDF compound or a group of coeluting isomers. Figure 7 describes the procedure to be followed for the determination of the S/N.

12.4.4 Polychlorinated Diphenyl Ether Interferences

12.4.4.1 In addition to the above criteria, the identification of a GC peak as a PCDF can only be made if no signal having a S/N $\geq$2.5 is detected, at the same retention time ($\pm$2 seconds), in the corresponding polychlorinated diphenyl ether (PCDPE, Table 6) channel.

13. QA/QC REQUIREMENTS

13.1 Performance Check Solutions

13.1.1 At the beginning of each 12-hour period during which samples are to be analyzed, aliquots of the 1) GC column performance check solution and 2) high-resolution concentration calibration solution No. 3 (HRCC-3) shall be analyzed to demonstrate adequate GC resolution and sensitivity, response factor reproducibility, and mass range calibration, and to establish the PCDD/PCDF retention time windows. A mass resolution check shall also be performed to demonstrate adequate mass resolution using an appropriate reference compound (PFK is recommended). If the required criteria are not met, remedial action must be taken before any samples are analyzed.

13.1.2 Deviations from criteria specified for the GC performance check or for the mass resolution check check invalidates all positive sample data collected between analysis of the performance check solution; extracts from those positive samples shall be reanalyzed.

If the routine calibration run fails at the beginning of a 12-hour shift, the instructions in Section 9.4.4 must be followed.

13.1.3 The GC column performance check mixture, high-resolution concentration calibration solutions, and the sample fortification solutions may be obtained from the EMSL-CIN. However, if not available from the EMSL-CIN, standards can be obtained from other sources, and solutions can be prepared in the laboratory. Concentrations of all solutions containing 2,3,7,8-substituted PCDDs/PCDFs, which are not obtained from the EMSL-CIN, must be verified by comparison with the EPA standard solutions that are available from the EMSL-CIN.

13.2 Blanks

13.2.1 Method Blank (MB)

One method blank is required per batch or per 20 samples, whichever is more frequent. For the method blank use all reagents, standards, equipment, apparatus, glassware and solvents that would be used for a sample. If the accompanying samples are aqueous, use methylene chloride-rinsed distilled water as a matrix. (There is no matrix for the MB if samples are nonaqueous.) Take the MB through all steps detailed in the analytical procedure.

13.2.1.1 The method blank must contain the same amount of ^{13}C-labeled internal standards that is added to samples before extraction.

13.2.1.1.1 If method blank contamination is present, check solvents, reagents, fortification solutions, apparatus and glassware to locate and eliminate the source of contamination before any further samples are extracted and analyzed.

13.2.1.1.2 If new batches of reagents or solvents contain interfering contaminants, purify or discard them.

13.2.2 Field Blanks

Each batch of samples may contain a field blank sample of uncontaminated soil, sediment or water that is to be fortified before analysis.

13.2.2.1 Fortified Field Blank

13.2.2.1.1 Weigh a 10-g portion or use 1 L (for aqueous samples) of the specified field blank sample and add 100 uL of the solution containing the nine internal standards (Table 2) diluted with 1.5 mL acetone.

13.2.2.1.2 Extract by using the procedures described in Section 11.2. As applicable, add 10 uL of the recovery standard solution and analyze a 2-uL aliquot of the concentrated extract.

13.2.2.2 Calculate the concentration of 2,3,7,8-substituted PCDDs/PCDFs and the percent recovery of the internal standards.

13.2.2.2.1 Extract and analyze a new simulated fortified field blank whenever new lots of solvents or reagents are used for sample extraction or for column chromatographic procedures.

13.2.2.3 Rinsate Samples

13.2.2.3.1 In addition to the field blank, a batch of samples may include a rinsate, which is a portion of the solvent (usually trichloroethylene) that was used to rinse sampling equipment. The rinsate is analyzed to assure that the samples were not contaminated by the sampling equipment.

13.2.2.3.2 The rinsate sample must be fortified like a regular sample.

13.2.2.3.3 Take a 100-mL (± 0.5 mL) portion of the sampling equipment rinse solvent (rinsate sample), filter, if necessary, and add 100 uL of the solution containing the nine internal standards (Table 2).

13.2.2.3.4 Using appropriate methods, concentrate to approximately 10 mL.

13.2.2.3.5 Just before analysis, add 10 uL tetradecane recovery standard solution (Table 2), and reduce the volume to a final volume of 20 uL, as necessary. No column chromatography is required.

13.2.2.3.6 Analyze an aliquot following the same procedures used to analyze samples.

13.2.2.3.7 Report percent recovery of the internal standard and the presence of any PCDD/PCDF compounds in pg-mL of rinsate solvent.

13.3 Surrogate spiked into samples. A surrogate compound ^{37}Cl-2,3,7,8-TCDD may be spiked into all samples and QC samples for this method. The spike concentration is 1.0 ng per sample. This surrogate is spiked following extraction and just prior to cleanup as a "cleanup recovery standard", in order to monitor relative loss of internal standard during both extraction and cleanup.

13.4 Duplicates

13.4.1 In each batch of samples, locate the sample specified for duplicate analysis, and prepare and analyze a second 10-g soil or sediment sample portion or 1-L water sample, or an appropriate amount of the type of matrix under consideration.

13.4.1.1 The results of the laboratory duplicates (percent recovery and concentrations of 2,3,7,8-substituted PCDD/PCDF compounds) should agree within 25 percent relative difference. Report all results.

13.4.1.2 Recommended actions to help locate problems:

13.4.1.2.1 Verify satisfactory instrument performance.

13.4.1.2.2 If possible, verify that no error was made while weighing the sample portions.

13.4.1.2.3 Review the analytical procedures with the performing laboratory personnel.

13.5 Matrix Spike and Matrix Spike Duplicate (MS and MSD)

13.5.1 Locate the sample for the MS and MSD analyses (the sample may be labeled "double volume").

13.5.2 Add an appropriate volume of the matrix spike fortification solution, adjusting the fortification level as specified in Table 1, under IS Spiking Levels.

13.5.3 Analyze the MS and MSD samples as described in Section 12.

13.5.4 The results obtained from the MS and MSD samples (percent recovery and concentrations of 2,3,7,8-substituted PCDDs/PCDFs) must agree within 20 percent relative difference.

13.6 Percent Recovery of the Internal Standards

For each sample, method blank and rinsate, calculate the percent recovery. The percent recovery should be between 40 percent and 135 percent for all 2,3,7,8-substituted internal standards.

NOTE: A low or high percent recovery for a blank does not require discarding the analytical data but it may indicate a potential problem with future analytical data.

13.7 Identification Criteria

13.7.1 If either one of the identification criteria appearing in Sections 12.4.1.1 through 12.4.1.4, is not met for an homologous series, it is reported that the sample does not contain unlabeled 2,3,7,8-substituted PCDD/PCDF isomers for that homologous series at the calculated detection limit.

13.7.2 If the first initial identification criteria are met, but the criteria appearing in Sections 12.4.1.5 and 12.4.2.1, are not met, that sample is presumed to contain interfering contaminants. This must be noted on the analytical report form, and the sample should be rerun or the extract reanalyzed.

14. CALCULATIONS

14.1 For gas chromatographic peaks that have met the criteria outlined in Section 12.4, calculate the concentration of the PCDD or PCDF compounds using the formula:

$$C_X = \frac{A_X \times Q_{is}}{A_{is} \times W \times \overline{RRF}(n)}$$

where:

C_X = concentration of unlabeled PCDD/PCDF congeners (or group of coeluting isomers within an homologous series) usually in pg/g or pg/L,

A_X = sum of the integrated ion abundances of the quantitation ions (Table 6) for the unlabeled PCDDs/PCDFs,

A_{is} = sum of the integrated ion abundances of the quantitation ions (Table 6) for the labeled internal standards,

Q_{is} = quantity, in pg, of the internal standard added to the sample before extraction,

W = Sample size in g (if solid) or L (if liquid).

$\overline{RRF}(n)$ = Calculated mean relative response factor for the analyte

[$\overline{RRF}(n)$ with n = 1 to 17; Section 9.1.4.7, this exhibit].

If the analyte is identified as one of the 2,3,7,8-substituted PCDDs or PCDFs, $\overline{RRF}(n)$ is the value calculated using the equation in Section 9.1.4.7. However, if it is a non-2,3,7,8-substituted congener, the $\overline{RRF}(k)$ value is the one calculated using the equation in Section 9.1.4.8.2 [$\overline{RRF}(k)$ with k = 27 to 30.].

14.2 Calculate the percent recovery of the nine internal standards measured in the sample extract, using the formula:

$$\text{Internal Standard Percent Recovery} = \frac{A_{is} \times Q_{rs}}{Q_{is} \times A_{rs} \times \overline{RRF}(m)} \times 100$$

where

A_{is} = sum of the integrated ion abundances of the quantitation ions (Table 6) for the labeled internal standard,

A_{rs} = sum of the integrated ion abundances of the quantitation ions (Table 6) for the labeled recovery standard; the selection of the recovery standard depends on the type of congeners (see Table 5, footnotes),

Q_{is} = Quantity, in pg, of the internal standard added to the sample before extraction,

Q_{rs} = Quantity, in pg, of the recovery standard added to the cleaned-up sample residue before HRGC/HRMS analysis, and

$\overline{RRF}(m)$ = calculated mean relative response factor for the labeled internal standard relative to the appropriate (see Table 5, footnotes) recovery standard. This represents the mean obtained in Section 9.1.4.9 [$\overline{RRF}(m)$ with m = 18 to 26].

14.3 If the concentration in the 10-uL or 50-uL final extraction of any of the fifteen 2,3,7,8-substituted PCDD/PCDF compounds (Table 3) exceeds the upper method calibration limits (MCL) listed in Table 1 (e.g., 200 pg/uL for TCDD in a 10g soil), the linear range of response versus concentration may have been exceeded, a reanalysis of the sample (using one tenth aliquot) should be undertaken. The volumes of the internal and recovery standard solutions should remain the same as described for the sample preparation (this exhibit, Section 11.1 to 11.9.2). For the other congeners (including OCDD), however, report the measured concentration and indicate that the value exceeds the MCL.

14.4 The total concentration for each homologous series of PCDD and PCDF is calculated by summing up the concentrations of all positively identified isomers of each homologous series. Therefore, the total should also include the 2,3,7,8-substituted congeners. The total number of GC signals included in the homologous total concentration value may be specified in the report.

14.5 Sample-Specific Estimated Detection Limit

The sample-specific estimated detection limit (EDL) is the concentration of a given analyte required to produce a signal with a peak height of at least 2.5 times the background signal level. An EDL is calculated for each 2,3,7,8-substituted congener that is not identified, regardless of whether or not other non-2,3,7,8-substituted isomers are present. Two methods of calculation can be used, as follows, depending on the type of response produced during the analysis of a particular sample.

14.5.1 Samples giving a response for both quantitation ions (Tables 6 and 9) that is less than 2.5 times the background level.

14.5.1.1 Use the expression for EDL (specific 2,3,7,8-substituted PCDD/PCDF) below to calculate an EDL for each absent 2,3,7,8-substituted PCDD/PCDF (i.e., S/N <2.5). The background level is determined by measuring the range of the noise (peak to peak) for the two quantitation ions (Table 6) of a particular 2,3,7,8-substituted isomer within an homologous series, in the region of the SICP trace corresponding to the elution of the internal standard (if the congener possesses an internal standard) or in the region of the SICP where the congener is expected to elute by comparison with the routine calibration data (for those congeners that do not have a ^{13}C-labeled standard), multiplying that noise height by 2.5, and relating the product to an estimated concentration that would produce that product height.

NOTE: The quantitation ions for both the unlabeled PCDDs/PCDFs and their internal standard must be consistently paired (using either both ligher mass ions or both heavier mass ions).

Use the formula:

$$\text{EDL(specific 2,3,7,8-subst.PCDD/PCDF)} = \frac{2.5 \times H_X \times Q_{is}}{H_{is} \times W \times \overline{RRF}(n)}$$

where

EDL = estimated detection limit for homologous 2,3,7,8-substituted PCDDs/PCDFs.

H_X = height of the average noise for one of the quantitation ions (Table 6) for the unlabeled PCDDs/PCDFs.

H_{is} = height of one of the quantitation ions (Table 6) for the labeled internal standards.

W, $\overline{RRF}(n)$, and Q_{is} retain the same meanings as defined in Section 14.1.

14.5.2 Samples characterized by a response above the background level with a S/N of at least 2.5 for at least one of the quantitation ions (Tables 6 and 9).

14.5.2.1 When the response of a signal having the same retention times as a 2,3,7,8-substituted congener has a S/N in excess of 2.5 and does not meet any of the other qualitative identification criteria listed in Section 12.4, calculate the "Estimated Maximum Possible Concentration" (EMPC) according to the expression shown in Section 14.1, except that A^x in Section 14.1 should represent the sum of the area under the smaller peak and of the other peak area calculated using the theoretical chlorine isotope ratio. Alternatively, an EDL can be calculated using the above formula and the height of one of the ions as appropriate.

14.6 The relative percent difference (RPD) is calculated as follows:

$$RPD = \frac{|S_1 - S_2|}{(S_1 + S_2) / 2} \times 100$$

S_1 and S_2 represent sample and duplicate sample results.

14.7 The 2,3,7,8-TCDD toxic equivalents (TE) of PCDDs and PCDFs present in the sample are calculated, only at the data user's request. This method assigns a 2,3,7,8-TCDD toxicity equivalency factor (TEF) to each of the fifteen 2,3,7,8-substituted PCDDs and PCDFs (Table 3 and the non-2,3,7,8-substituted compounds as shown in Table 11). The 2,3,7,8-TCDD equivalent of the PCDDs and PCDFs present in the sample is calculated by summing the TEF times their concentration for each of the compounds or groups of compounds listed in Table 11. The exclusion of other homologous series such as mono-, di-, and tri- and chlorinated dibenzodioxins and dibenzofurans does not mean that they are non-toxic.

Their toxicity, as known at this time, is much less than the toxicity of the compounds listed in Table 11. The above procedure for calculating the 2,3,7,8-TCDD toxic equivalents is not claimed by the CDWG to be based on a thoroughly established scientific foundation. The procedure, rather, represents a "Consensus recommendation on science policy". Since the procedure may be changed in the future, reporting requirements for PCDD and PCDF data would still include the reporting of the analyte concentrations of the PCDD/PCDF congener as calculated in Sections 14.1 and 14.4.

14.7.1 Two-GC Column TEF Determination

Isomer specificity for all 2,3,7,8-substituted PCDDs/PCDFs cannot be achieved on the 60-m DB-5 GC column. In order to determine the proper concentrations of the individual 2,3,7,8-substituted congeners, the sample extract may be reanalyzed on another GC column.

14.7.1.1 The concentration of 2378-TCDD (see note below), is calculated from the analysis of the sample extract on the 60m DB-5 fused silica capillary column. The chromatographic separation of this isomer must be $\leq$ 25% valley.

14.7.1.2 For samples that have a positive result for 2378-TCDF on the DB-5 column, the extract is reanalyzed on a 30m DB-225 fused silica column. The GC/MS conditions are altered so that only the first descriptor (Table 6) is used. The reported concentration for 2378-TCDF is then the result calculated from the DB-225 analysis. The chromtographic separation between 2378-TCDF and any other unlabled TCDF isomers must be $\leq$ 25% valley using the column performance check solution for the DB-225 column. Concentration calculations are performed as in section 14.1 through 14.6 (this exhibit).

14.7.1.3 For samples that have positive results for the 2378-substituted penta and hexa isomers, the extract can be anlyzed on either the 30m DB-225 column or a 60m SP-2330 (or SP-2331) column, if requested by the client. The GC/MS conditions are altered so that only the second and third descriptor (Table 6) are used. Concentration calculations are performed as in section 14.1 through 14.6 (this exhibit).

NOTE: The confirmation and quantitation of 2,3,7,8-TCDD (this exhibit, Section 14.7.1.1) may be accomplished on the SP-2330 GC column instead of the DB-5 column, provided the criteria listed in Section 8.1.2 (this exhibit) are met and the requirements described in Section 13.1 (this exhibit) are followed.

14.7.1.4 For a gas chromatographic peak to be identified as a 2,3,7,8-substituted PCDD/PCDF congener, it must meet the ion abundance (Section 12.4.2, this exhibit) and signal-to-noise ratio criteria. In addition, the retention time identification criterion criterion described in Section 12.4.1.1 (this exhibit) applies here for congeners for which a carbon-labeled analog is available in the sample extract. However, the relative retention time (RRT) of the 2,3,7,8-substituted congeners for which no carbon-labeled analogs are available must fall within 0.006 units of the carbon-labeled standard RRT. Experimentally, this is accomplished by using the attributions described in Table 12 and the results from the routine calibration run on the DB-5 column.

15. REFERENCES

15.1 SW846, Third Edition, Method 8290, USEPA, November, 1990.

15.2 "Protocol for the Analysis of 2,3,7,8-TCDD by HRGC/HRMS". J.S. Stanley and T.M. Sack, EPA 600/4-86-004.

15.3 "Safety in Academic Chemistry Laboratories", American Chemical Society Publication, Committee on Chemical Safety (3rd Edition, 1979.)

15.4 "Carcinogens - Working with Carcinogens". Department of Health, Education, and Welfare, Public Health Service, Center for Disease Control. National Institute for Occupational Safety and Health. Publication No. 77-206, August 1977.

15.5 "OSHA Safety and Health Standards, General Industry", (29 CFR 1910) Occupational Safety and Health Administration, OSHA 2206 (revised January 1976).

APPENDIX

This procedure is designed for the periodic evaluation of potential contamination by 2,3,7,8-substituted PCDD/PCDF congeners of the working areas inside the laboratory.

PERFORMING WIPE TEST

Perform the wipe tests on surface areas of two inches by one foot with laboratory wipers saturated with distilled-in-glass acetone using a pair of clean stainless steel forceps. Use one wiper for each of the designated areas. Combine the wipers to one composite sample in an extraction jar containing 200 mL distilled-in-glass hexane. Place an equal number of unused wipers in 200 mL hexane and use this as a control.

SAMPLE PREPARATION

Close the jar containing the wipers and 200 mL hexane and extract for 20 minutes using a wrist-action shaker. Use an appropriate means to reduce the volume to approximately 1.0 ml. Put through an alumina column to clean up potential interfering compounds. Add appropriate amount of recovery standard.

EXTRACT ANALYSIS

Concentrate the contents of the vial to a final volume of 20 uL (either in a minivial or in a capillary tube). Inject two uL of each extract (wipe and control) onto a capillary column and analyze for 2,3,7,8-substituted PCDDs/PCDFs as specified in the analytical method Section 12 (this exhibit). Perform calculations according to Section 14 (this exhibit).

REPORTING FORMAT

Report the presence of 2,3,7,8-substituted PCDDs and PCDFs as a quantity (pg or ng) per wipe test experiment (WTE). Under the conditions outlined in this analytical protocol, a lower limit of calibration of 25 pg/WTE is expected for 2,3,7,8-TCDD. A positive response for the blank (control) is defined as a signal in the TCDD retention time window at any of the masses monitored which is equivalent to or above 8 pg of 2,3,7,8-TCDD per WTE. For other congeners, use the multiplication factors listed in Table 1, footnote (a) (e.g., for OCDD, the lower MCL is 25 x 5 = 125 pg/WTE and the positive response for the blank would be 8 x 5 = 40 pg). Also, report the recoveries of the internal standards during the simplified cleanup procedure.

FREQUENCY OF WIPE TESTS

At a minimum, wipe tests should be performed when there is evidence of contamination in the method blanks.

CORRECTIVE ACTION

An upper limit of 25 pg per TCDD isomer and per wipe test experiment is allowed. (Use multiplication factors listed in footnote (a) from Table 1 for other congeners.) This value corresponds to the lower calibration limit of the analytical method. Steps to correct the contamination must be taken whenever these levels are exceeded. To that effect, first vacuum the working places (hoods, benches, sink) using a vacuum cleaner equipped with a high-efficiency particulate absorbant (HEPA) filter and then wash with a detergent. A new set of wipes should be analyzed before anyone is allowed to work in the dioxin area of the laboratory.

TABLE 1

Types of Matrices, Sample Sizes and 2,3,7,8-TCDD-Based Method Calibration Limits (Parts per Trillion)

	Water	Soil Sediment Paper Pulp	Fly Ash	Fish Tissue	Human Adipose Tissue	Sludges, Fuel Oil	Still-Bottom
Lower MCL[a]	0.02	2.0	2.0	2.0	2.0	10	20
Upper MCL[a]	4.0	400	400	400	400	2000	4000
Weight (g)	1000	10	10	10	10	2.0	1.0
IS Spiking Levels (ng)	1.0	1.0	1.0	1.0	1.0	1.0	1.0
Final Extr. Vol.(uL)	20	20	20	20	20	20	20

([a]) For other congeners, multipy the values by 1 for TCDF/PeCDD/PeCDF, by 2.5 for HxCDD/HxCDF/HpCDD/HpCDF, and by 5 for OCDD/OCDF.

TABLE 2

COMPOSITION OF THE SAMPLE FORTIFICATION AND RECOVERY STANDARD SOLUTIONS

Analyte	Sample Fortification Solution Concentration (pg/uL; Solvent: Isooctane)	Recovery Standard Solution Concentration (pg/uL; Solvent: Tetradecane)
^{13}C-2,3,7,8-TCDD	10	--
^{13}C-2,3,7,8-TCDF	10	--
^{13}C-1,2,3,4-TCDD	--	100
^{13}C-1,2,3,7,8-PeCDD	10	--
^{13}C-1,2,3,7,8-PeCDF	10	--
^{13}C-1,2,3,6,7,8-HxCDD	25	--
^{13}C-1,2,3,4,7,8-HxCDF	25	--
^{13}C-1,2,3,7,8,9-HxCDD	--	100
^{13}C-1,2,3,4,6,7,8-HpCDD	25	--
^{13}C-1,2,3,4,6,7,8-HpCDF	25	--
^{13}C-OCDD	50	--

TABLE 3

THE FIFTEEN 2,3,7,8-SUBSTITUTED PCDD AND PCDF CONGENERS

PCDD	PCDF
2,3,7,8-TCDD(*)	2,3,7,8-TCDF(*)
1,2,3,7,8-PeCDD(*)	1,2,3,7,8-PeCDD(*)
1,2,3,6,7,8-HxCDD(*)	2,3,4,7,8-PeCDF
1,2,3,4,7,8-HxCDD	1,2,3,6,7,8-HxCDF
1,2,3,7,8,9-HxCDD(+)	1,2,3,7,8,9-HxCDF
1,2,3,4,6,7,8-HpCDD(*)	1,2,3,4,7,8-HxCDF(*)
	2,3,4,6,7,8-HxCDF
	1,2,3,4,6,7,8-HpCDF(*)
	1,2,3,4,7,8,9-HpCDF

(*)The ^{13}C-labeled analog is used as an internal standard.
(+)The ^{13}C-labeled analog is used as a recovery standard.

TABLE 4

ISOMERS OF CHLORINATED DIOXINS AND FURANS AS A FUNCTION OF THE NUMBER OF CHLORINE ATOMS

Number of Chlorine Atoms	Number of Dioxin Isomers	Number of 2,3,7,8 Isomers	Number of Furan Isomers	Number of 2,3,7,8 Isomers
1	2	---	4	---
2	10	---	16	---
3	14	---	28	---
4	22	1	38	1
5	14	1	28	2
6	10	3	16	4
7	2	1	4	2
8	1	1	1	1
Total	75	7	135	10

TABLE 5

HIGH-RESOLUTION CONCENTRATION CALIBRATION SOLUTIONS

Compound	HRGCC	Concentration (pg/uL, in Nonane) 5	4	3	2	1
Unlabeled Analytes						
2,3,7,8-TCDD		200	50	10	2.5	1
2,3,7,8-TCDF		200	50	10	2.5	1
1,2,3,7,8-PeCDD		500	125	25	6.25	2.5
1,2,3,7,8-PeCDF		500	125	25	6.25	2.5
2,3,4,7,8-PeCDF		500	125	25	6.25	2.5
1,2,3,4,7,8-HxCDD		500	125	25	6.25	2.5
1,2,3,6,7,8-HxCDD		500	125	25	6.25	2.5
1,2,3,7,8,9-HxCDD		500	125	25	6.25	2.5
1,2,3,4,7,8-HxCDF		500	125	25	6.25	2.5
1,2,3,6,7,8-HxCDF		500	125	25	6.25	2.5
1,2,3,7,8,9-HxCDF		500	125	25	6.25	2.5
2,3,4,6,7,8-HxCDF		500	125	25	6.25	2.5
1,2,3,4,6,7,8-HpCDD		500	125	25	6.25	2.5
1,2,3,4,6,7,8-HpCDF		500	125	25	6.25	2.5
1,2,3,4,7,8,9-HpCDF		500	125	25	6.25	2.5
OCDD		1000	250	50	12.5	5
OCDF		1000	250	50	12.5	5
Internal Standards						
$^{13}C_{12}$-2,3,7,8-TCDD		50	50	50	50	50
$^{13}C_{12}$-2,3,7,8-TCDF		50	50	50	50	50
$^{13}C_{12}$-1,2,3,7,8-PeCDD		50	50	50	50	50
$^{13}C_{12}$-1,2,3,7,8-PeCDF		50	50	50	50	50
$^{13}C_{12}$-1,2,3,6,7,8-HxCDD		125	125	125	125	125
$^{13}C_{12}$-1,2,3,4,7,8-HxCDF		125	125	125	125	125
$^{13}C_{12}$-1,2,3,4,6,7,8-HpCDD		125	125	125	125	125
$^{13}C_{12}$-1,2,3,4,6,7,8-HpCDF		125	125	125	125	125
$^{13}C_{12}$-OCDD		250	250	250	250	250
Recovery Standards						
$^{13}C_{12}$-1,2,3,4-TCDD(a)		100	100	100	100	100
$^{13}C_{12}$-1,2,3,4,7,8,9-HxCDD(b)		100	100	100	100	100

(a) Used for recovery determination of TCDD, TCDF, PeCDD and PeCDF internal

TABLE 6

IONS MONITORED FOR HRGC/HRMS ANALYSIS OF PCDD/PCDFs
((S) = internal/recovery standard)

Descriptor	Accurate[a] Mass	Ion ID	Elemental Composition	Analyte
1	303.9016	M	$CH^{35}Cl$	TCDF
	305.8987	M+2	$CH^{35}Cl^{37}Cl_0$	TCDF
	315.9419	M	$CH^{35}Cl_0$	TCDF (S)
	317.9389	M+2	$^{13}CH^{35}Cl^{37}Cl_0$	TCDF (S)
	319.8965	M	$CH^{35}Cl_0$	TCDD
	321.8936	M+2	$CH^{35}Cl^{37}Cl_0$	TCDD
	331.9368	M	$^{13}CH^{35}Cl_0$	TCDD (S)
	333.9339	M+2	$^{13}CH^{35}Cl^{37}Cl_0$	TCDD (S)
	339.8597	M+2	$CH^{35}Cl^{37}Cl_0$	PeCDF
	341.8567	M+4	$CH^{35}Cl^{37}Cl_0$	PeCDF
	375.8364	M+2	$CH^{35}Cl_0$	HxCDPE
	316.9824	LOCK	CF	PFK
2	351.9000	M+2	$^{13}CH^{35}Cl^{37}Cl_0$	PeCDF (S)
	353.8970	M+4	$^{13}CH^{35}Cl^{37}Cl_0$	PeCDF (S)
	355.8546	M+2	$CH^{35}Cl^{37}Cl_0$	PeCDD
	357.8516	M+4	$CH^{35}Cl^{37}Cl_0$	PeCDD
	367.8949	M+2	$^{13}CH^{35}Cl^{37}Cl_0$	PeCDD (S)
	369.8919	M+4	$^{13}CH^{35}Cl^{37}Cl_0$	PeCDD (S)

TABLE 6

Continued

Descriptor	Accurate[a] Mass	Ion ID	Elemental Composition	Analyte
	339.8597	M+2	$CH^{35}Cl^{37}Cl_0$	PeCDF
	341.8567	M+4	$CH^{35}Cl^{37}Cl_0$	PeCDF
	409.7974	M+2	$CH^{35}Cl_0$	HpCDPE
	366.9793	LOCK	CF	PFK
3	373.8208	M+2	$CH^{35}Cl^{37}Cl_0$	HxCDF
	375.8178	M+4	$CH^{35}Cl^{37}Cl_0$	HxCDF
	383.8642	M	$^{13}CH^{35}Cl_0$	HxCDF (S)
	385.8610	M+2	$^{13}CH^{35}Cl^{37}Cl_0$	HxCDF (S)
	389.8156	M+2	$CH^{35}Cl^{37}Cl_0$	HxCDD
	391.8127	M+4	$CH^{35}Cl^{37}Cl_0$	HxCDD
	401.8559	M+2	$^{13}CH^{35}Cl^{37}Cl_0$	HxCDD (S)
	403.8529	M+4	$^{13}CH^{35}Cl^{37}Cl_0$	HxCDD (S)
	445.7555	M+4	$CH^{35}Cl^{37}Cl_0$	OCDPE
	380.9760	LOCK	CF	PFK
4	407.7818	M+2	$CH^{35}Cl^{37}Cl_0$	HpCDF
	409.7789	M+4	$CH^{35}Cl^{37}Cl_0$	HpCDF
	417.8253	M	$^{13}CH^{35}Cl_0$	HpCDF (S)

TABLE 6

Continued

Descriptor	Accurate[a] Mass	Ion ID	Elemental Composition	Analyte
	419.8220	M+2	$^{13}CH^{35}Cl^{37}Cl_0$	HpCDF (S)
	423.7766	M+2	$CH^{35}Cl^{37}Cl_0$	HpCDD
	425.7737	M+4	$CH^{35}Cl^{37}Cl_0$	HpCDD
	435.8169	M+2	$^{13}CH^{35}Cl^{37}Cl_0$	HpCDD (S)
	437.8140	M+4	$^{13}CH^{35}Cl^{37}Cl_0$	HpCDD (S)
	479.7165	M+4	$CH^{35}Cl^{37}Cl_0$	NCDPE
	442.9730	LOCK	CF	PFK
5	441.7428	M+2	$C^{35}Cl^{37}Cl_0$	OCDF
	443.7399	M+4	$C^{35}Cl^{37}Cl_0$	OCDF
	457.7377	M+2	$C^{35}Cl^{37}Cl_0$	OCDD
	459.7348	M+4	$C^{35}Cl^{37}Cl_0$	OCDD
	469.7779	M+2	$^{13}C^{35}Cl^{37}Cl_0$	OCDD (S)
	471.7750	M+4	$^{13}C^{35}Cl^{37}Cl_0$	OCDD (S)
	513.6775	M+4	$C^{35}Cl^{37}Cl_0$	DCDPE
	454.9728	LOCK	CF	PFK

(a) The following nuclidic masses were used:

H = 1.007825	O = 15.994915
C = 12.000000	^{35}Cl = 34.968853
^{13}C = 13.003355	^{37}Cl = 36.965903

TABLE 7

RECOMMENDED GC OPERATING CONDITIONS

The GC Operating Conditions (Temperatures (°C), and Times (minutes)) Are as Follows:

Injector Temperature: 280°C

Interface Temperature: 280°C

Initial Temperature and Time: 190°C / 1 Minute

Temperature Program: 190°C, increasing at a rate of 4°C per minute up to 240°C, and maintaining at this temperature until the last of the tetra- group has eluted from the column. (The total time required for this is approximately 25 minutes, depending on the length of the column). The maintained temperature of 240°C is then increased to 320°C at the rate of 20°C per minute and held at this level until the last compound (octa-group) has eluted from the column.

TABLE 8
PCDD AND PCDF CONGENERS PRESENT IN THE GC PERFORMANCE EVALUATION SOLUTION AND USED FOR DEFINING THE HOMOLOGOUS GC RETENTION TIME WINDOWS ON A 60-M DB-5 COLUMN

No. of Chlorine Atoms	PCDD-Positional Isomer		PCDF-Positional Isomer	
	Early Eluter	Late Eluter	Early Eluter	Late Eluter
4[a]	1,3,6,8	1,2,8,9	1,3,6,8	1,2,8,9
5	1,2,4,6,8/ 1,2,4,7,9	1,2,3,8,9	1,3,4,6,8	1,2,3,8,9
6	1,2,3,4,6,8	1,2,3,4,6,7	1,2,3,4,6,8	1,2,3,4,8,9
7	1,2,3,4,6,7,8	1,2,3,4,6,7,9	1,2,3,4,6,7,8	1,2,3,4,6,7,9
8		1,2,3,4,6,7,8,9		1,2,3,4,6,7,8,9

(a) In addition to these two PCDD isomers, the 1,2,3,4-, 1,2,3,7-, 1,2,3,8-, 2,3,7,8-, $^{13}C_{12}$-2,3,7,8-, and 1,2,3,9-TCDD isomers must also be present.

TABLE 9
THEORETICAL ION ABUNDANCE RATIOS AND THEIR CONTROL LIMITS
FOR PCDDs AND PCDFs

Number of Chlorine Atoms	Ion Type	Theoretical Ratio	Control Limits lower	Control Limits upper
4	M / M+2	0.77	0.65	0.89
5	M+2 / M+4	1.55	1.32	1.78
6	M+2 / M+4	1.24	1.05	1.43
6[a]	M / M+2	0.51	0.43	0.59
7[b]	M / M+2	0.44	0.37	0.51
7	M+2 / M+4	1.04	0.88	1.20
8	M+2 / M+4	0.89	0.76	1.02

[a] Used only for ^{13}C-HxCDF (IS).

[b] Used only for ^{13}C-HpCDF (IS).

TABLE 10
RELATIVE RESPONSE FACTOR [RRF (number)] ATTRIBUTES

Number	Specific Congener Name
1	2,3,7,8-TCDD (and total TCDDs)
2	2,3,7,8-TCDF (and total TCDFs)
3	1,2,3,7,8-PeCDD (and total PeCDDs)
4	1,2,3,7,8-PeCDF
5	2,3,4,7,8-PeCDF
6	1,2,3,4,7,8-HxCDD
7	1,2,3,6,7,8-HxCDD
8	1,2,3,7,8,9-HxCDD
9	1,2,3,4,7,8-HxCDF
10	1,2,3,6,7,8-HxCDF
11	1,2,3,7,8,9-HxCDF
12	2,3,4,6,7,8-HxCDF
13	1,2,3,4,6,7,8-HpCDD (and total HpCDDs)
14	1,2,3,4,6,7,8-HpCDF
15	1,2,3,4,7,8,9-HpCDF
16	OCDD
17	OCDF
18	$^{13}C_{12}$-2,3,7,8-TCDD
19	$^{13}C_{12}$-2,3,7,8-TCDF
20	$^{13}C_{12}$-1,2,3,7,8-PeCDD
21	$^{13}C_{12}$-1,2,3,7,8-PeCDF
22	$^{13}C_{12}$-1,2,3,6,7,8-HxCDD
23	$^{13}C_{12}$-1,2,3,4,7,8-HxCDF
24	$^{13}C_{12}$-1,2,3,4,6,7,8-HpCDD
25	$^{13}C_{12}$-1,2,3,4,6,7,8-HpCDF
26	$^{13}C_{12}$-OCDD
27	Total PeCDFs
28	Total HxCDFs
29	Total HxCDDs
30	Total HpCDFs

TABLE 11
2,3,7,8-TCDD EQUIVALENT FACTORS (TEFs) FOR THE
POLYCHLORINATED DIBENZODIOXINS AND DIBENZOFURANS

Number	Compound(s)	TEF
1	2,3,7,8-TCDD	1.00
2	1,2,3,7,8-PeCDD	0.50
3	1,2,3,6,7,8-HxCDD	0.10
4	1,2,3,7,8,9-HxCDD	0.10
5	1,2,3,4,7,8-HxCDD	0.10
6	1,2,3,4,6,7,8-HpCDD	0.01
7	1,2,3,4,6,7,8,9-OCDD	0.001
8	2,3,7,8-TCDF	0.1
9	1,2,3,7,8-PeCDF	0.05
10	2,3,4,7,8-PeCDF	0.5
11	1,2,3,6,7,8-HxCDF	0.1
12	1,2,3,7,8,9-HxCDF	0.1
13	1,2,3,4,7,8-HxCDF	0.1
14	2,3,4,6,7,8-HxCDF	0.1
15	1,2,3,4,6,7,8-HpCDF	0.01
16	1,2,3,4,7,8,9-HpCDF	0.01
17	1,2,3,4,6,7,8,9-OCDF	0.001

TABLE 12
TOXICITY EQUIVALENCY FACTOR: ANALYTE RELATIVE RETENTION TIME REFERENCE ATTRIBUTES

Analyte	Analyte RRT Reference(a)
1,2,3,4,7,8-HxCDD	$^{13}C_{12}$-1,2,3,6,7,8-HxCDD
1,2,3,6,7,8-HxCDF	$^{13}C_{12}$-1,2,3,4,7,8-HxCDF
1,2,3,7,8,9-HxCDF	$^{13}C_{12}$-1,2,3,4,7,8-HxCDF
2,3,4,6,7,8-HxCDF	$^{13}C_{12}$-1,2,3,4,7,8-HxCDF

(a) The retention time of 2,3,4,7,8-PeCDF on the DB-5 column is measured relative to $^{13}C_{12}$-1,3,7,8-PeCDF and the retention time of 1,2,3,4,7,8,9-HpCDF relative to $^{13}C_{12}$-1,2,3,4,6,7,8-HpCDF.

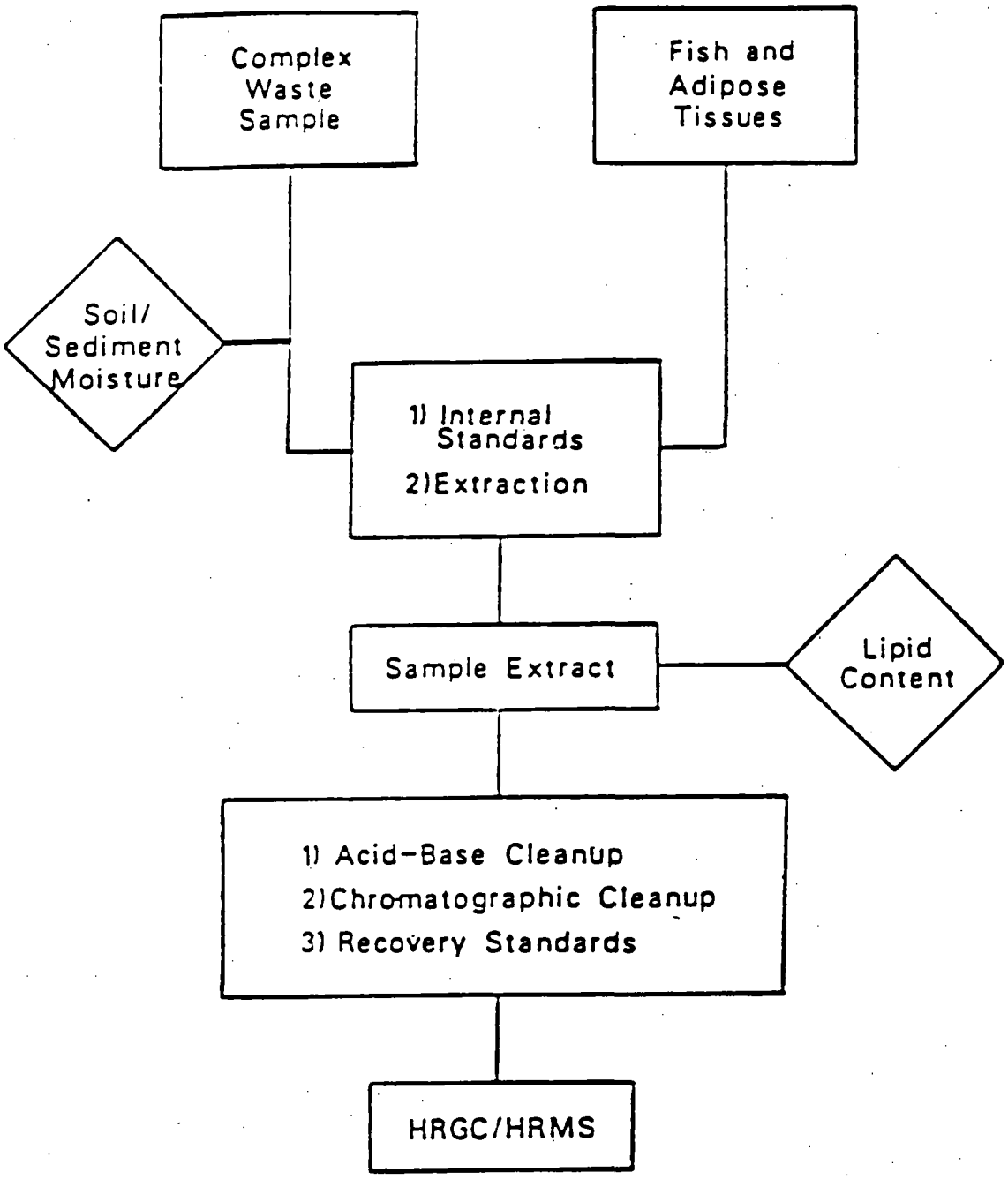
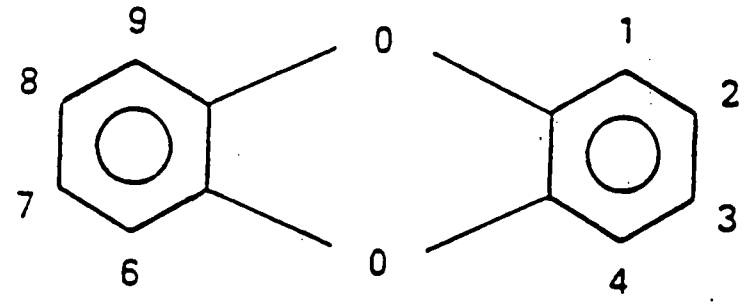
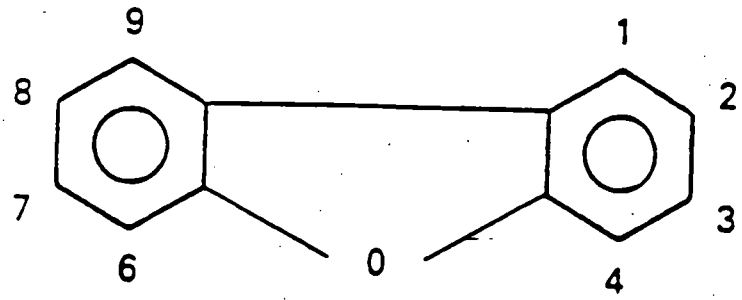

Complex Waste Sample
Fish and Adipose Tissues
Soil/ Sediment Moisture
1) Internal Standards
2)Extraction
Sample Extract
Lipid Content
1) Acid-Base Cleanup
2)Chromatographic Cleanup
3) Recovery Standards
HRGC/HRMS

Figure 1



Dibenzodioxin



Dibenzofuran

Figure 2

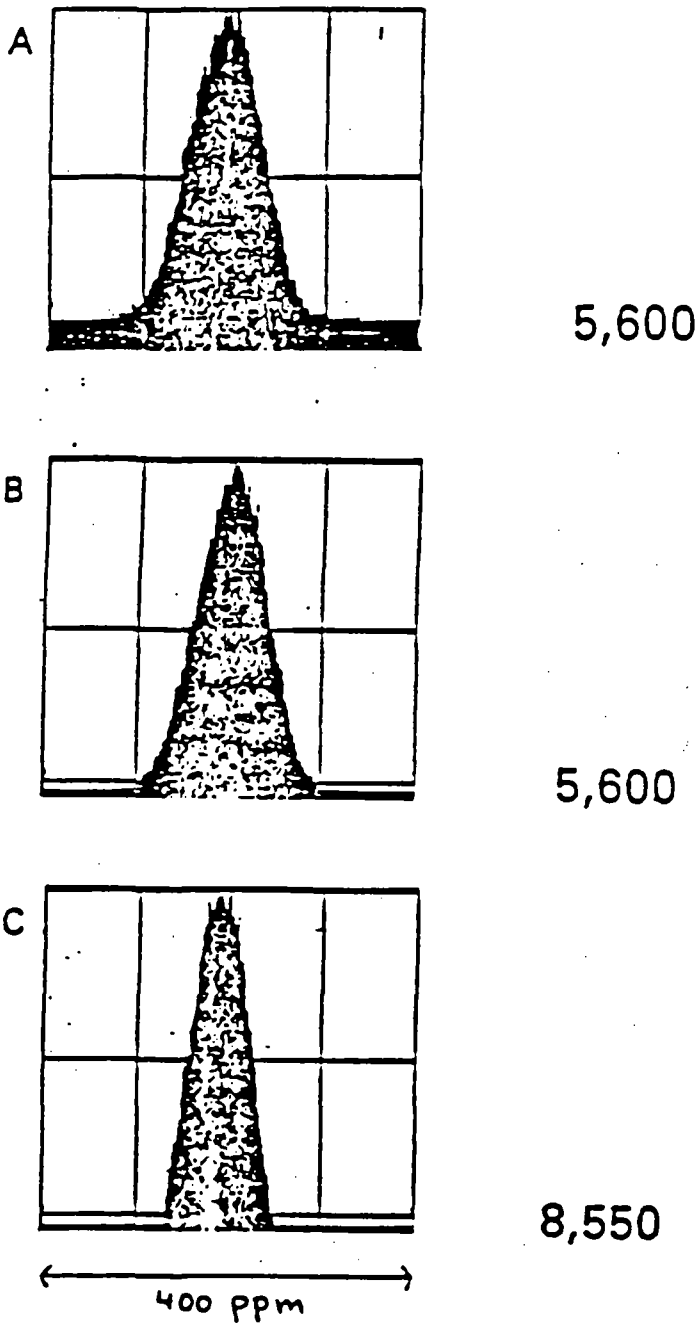


Figure 3

Analytical Procedure

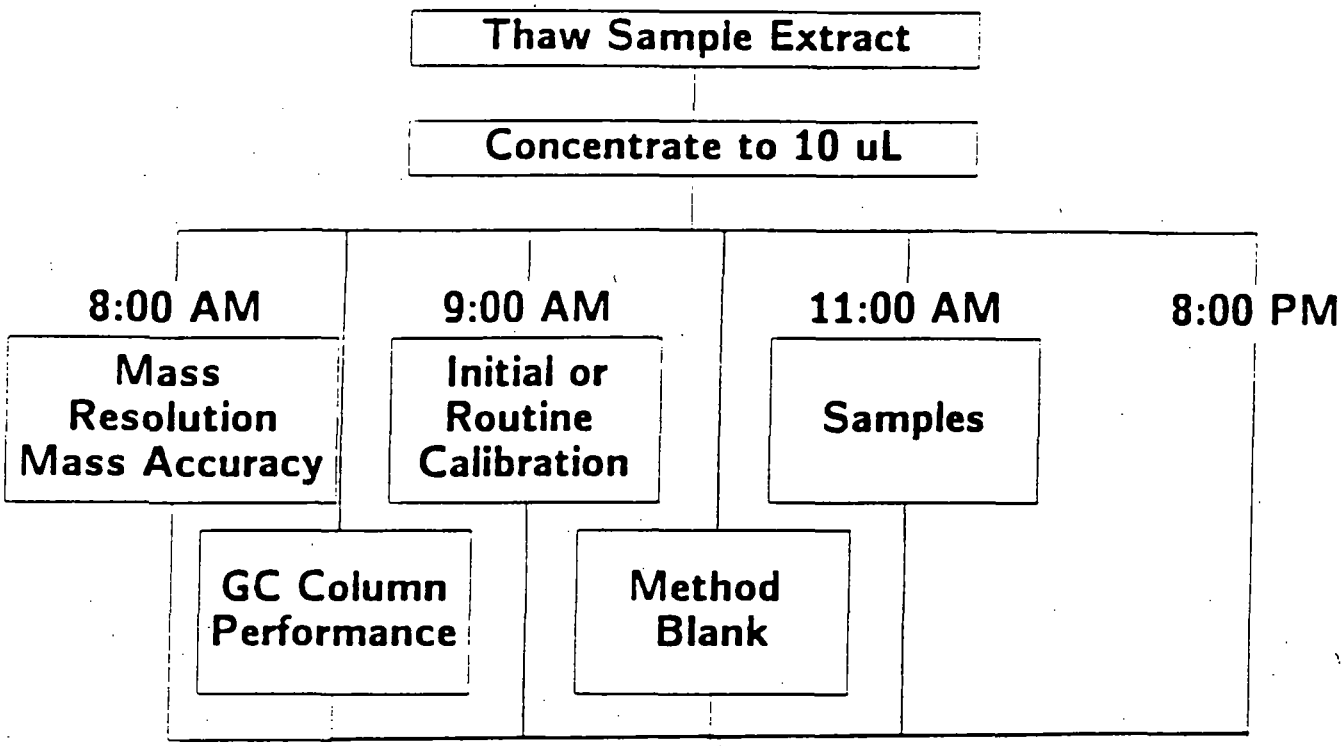
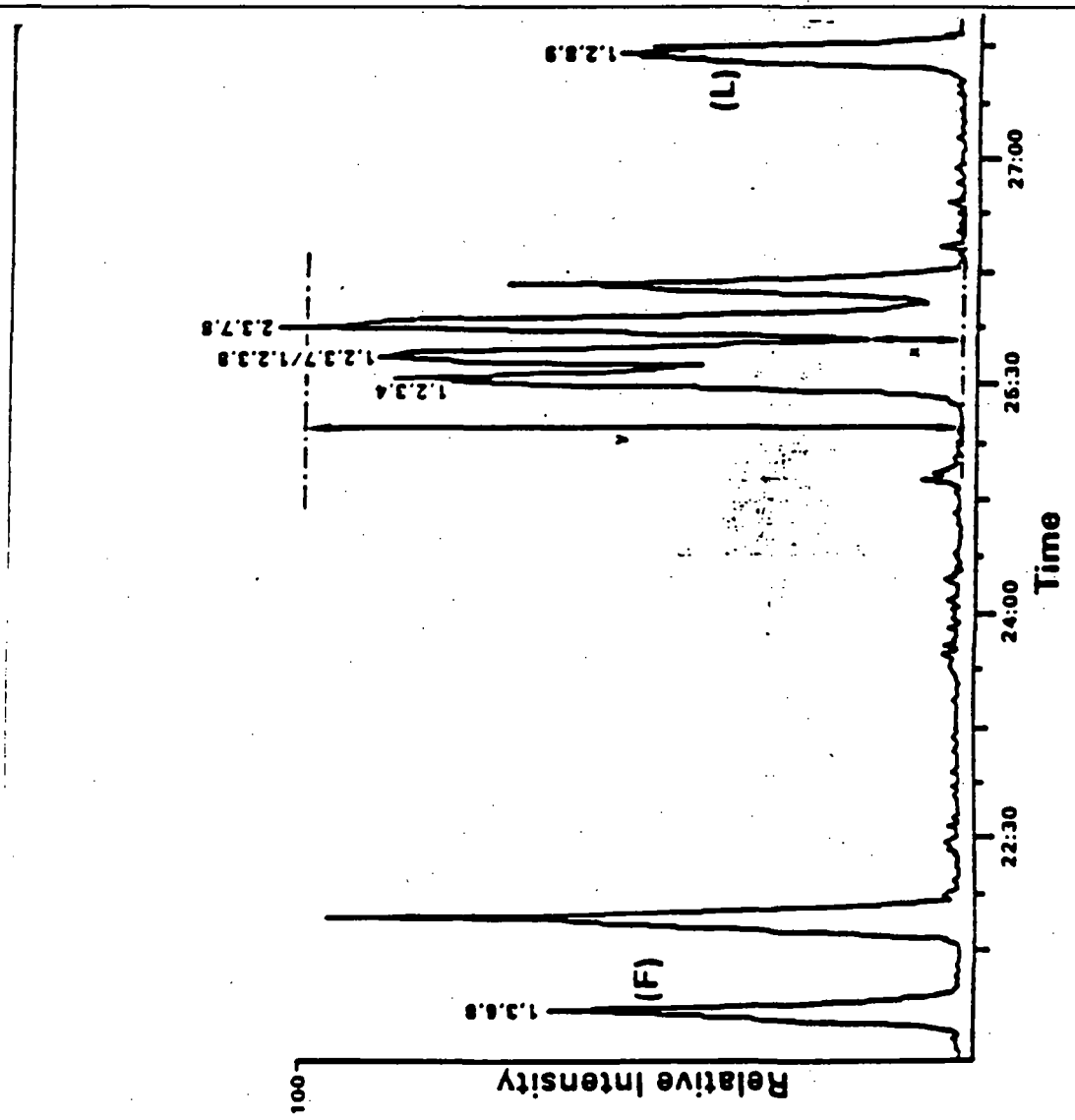
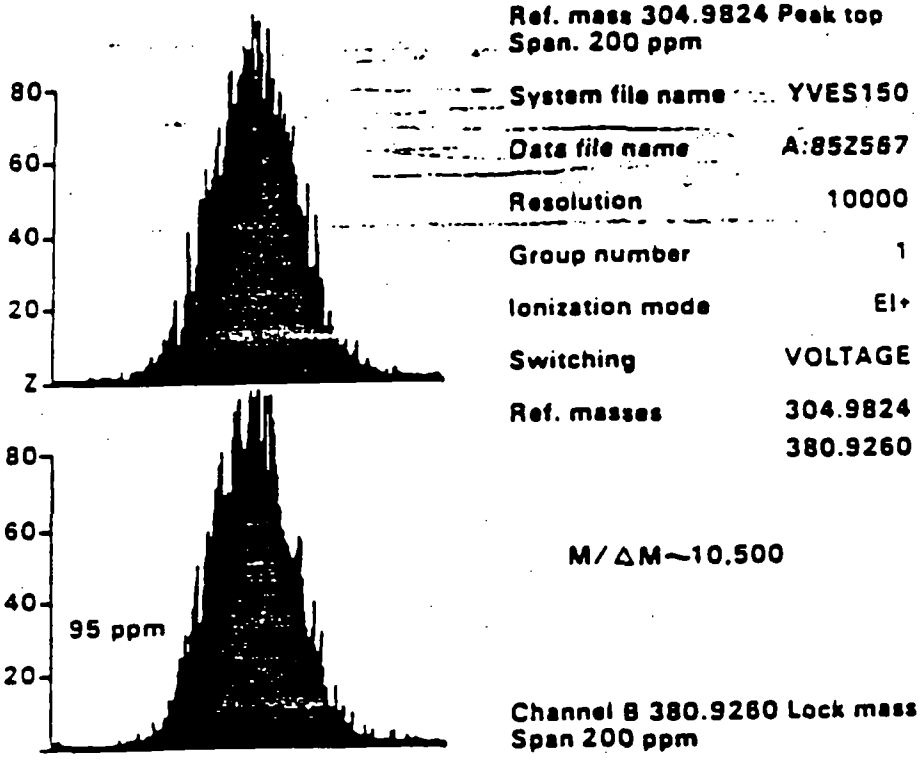


Figure 4



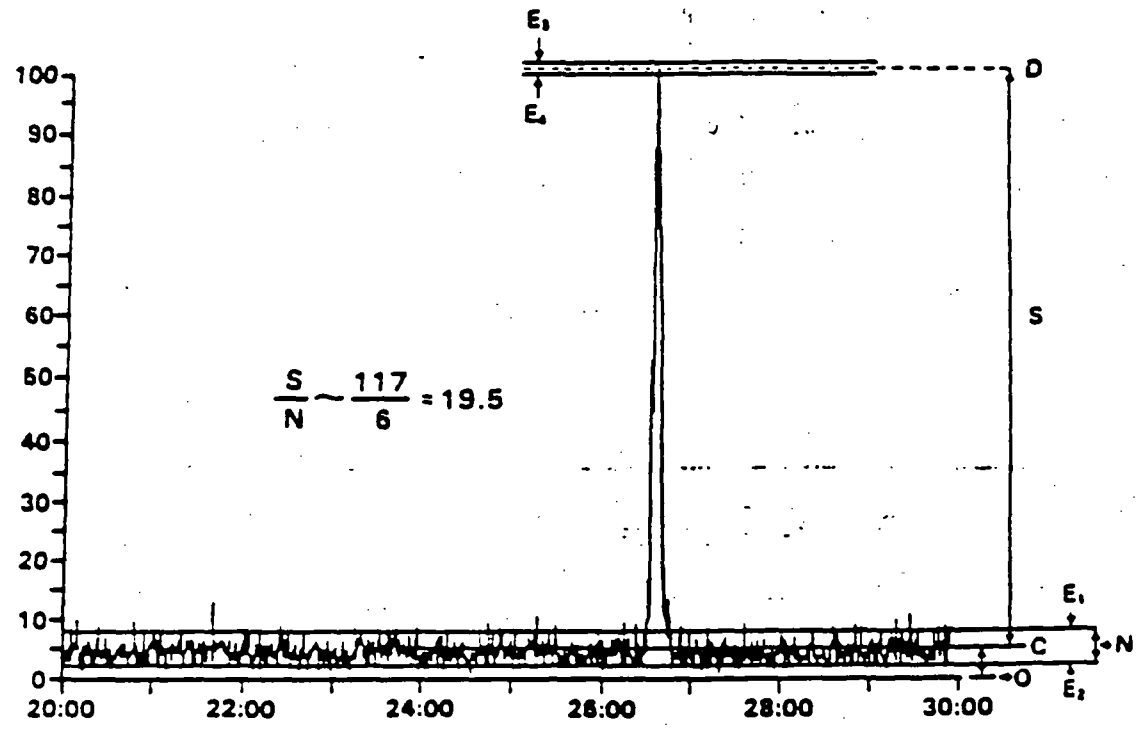
Selected ion current profile for m/z 322 (TCDDs) produced by MS analysis of the GC performance check solution on a 60 m DB-5 fused silica capillary column under the conditions listed in Section 7.6.

Figure 5



Peak profiles representing two PFK reference ions at m/z 305 and 381. The resolution of the high-mass signal is 95 ppm at 5 percent of the peak height; this corresponds to a resolving power M/ΔM of 10,500 (10 percent valley definition).

Figure 6



Manual determination of S/N.

The peak height (S) is measured between the mean noise (lines C and D). These mean signal values are obtained by tracing the line between the baseline average noise extremes, E1 and E2, and between the apex average noise extremes, E3 and E4, at the apex of the signal.

NOTE: It is imperative that the instrument interface amplifier electronic zero offset be set high enough so that negative going baseline noise is recorded.

Figure 7

18. IEA SOP FOR SW-846 7471 MERCURY ANALYSIS IN SOIL

1.0 APPROVALS

The signatures of the following individuals indicate that this SOP is complete and meets the requirements specified in corporate document # QAS00200.NET. In addition, it signifies that the content meets the specifications of the referenced "Test Code".

Laboratory Director

Quality Assurance Manager

2.0 SCOPE AND APPLICATION

2.1 The purpose of this SOP is to define the analysis of samples by cold-vapor atomic absorption for samples of a solid matrix. Samples are digested and then analyzed for mercury by reduction with stannous chloride.

2.2 The element determined is defined as "Total Metals" for a digested sample.

2.3 The IEA test code for this analysis is HG_54.

3.0 SUMMARY OF METHOD

3.1 This method is used to analyze samples that have been digested for mercury analysis. Samples are introduced into a flow cell with a nitrogen carrier gas where they absorb light at a characteristic wavelength. As the intensity of light absorbed is proportional to the concentration of mercury in the sample, these values are compared to a standard curve and the concentration of mercury determined.

3.2 This method is based on EPA method 7471.

3.3 This method deviates from the EPA method 7471 in that calibration curves are not constructed hourly. Instead, CCVs must pass or the run is terminated.

4.0 INTERFERENCES

4.1 Potassium permanganate is added to eliminate possible interference from sulfide. Concentrations as high as 20 mg/L of sulfide do not interfere with the recovery of mercury.

4.2 Copper has been reported to interfere; however, copper concentrations as high as 10 mg/L have no effect on mercury recovery.

4.3 High levels of free chlorine interfere with the analysis as chlorine absorbs at a wavelength of 253.7 nm also. Potassium permanganate oxidizes chlorides to chlorine. Therefore, additional potassium permanganate may be needed for seawaters, brines, etc. where salt content of the samples may be high. Hydroxylamine sulfate is added to the sample and the cell is purged with nitrogen before stannous chloride is added to the sample to remove any chlorine interferences.

4.4 Certain organic compounds interfere with mercury analysis. When organic interference is suspected, the sample should be analyzed without reagents to check for this interference.

5.0 SAFETY

5.1 Care should be used when working with concentrated acids and all protective gear worn. Safety glasses, lab coat and latex gloves are recommended to protect against glass breakage and sample spillage.

5.2 **Mercury is very toxic !** Care should be taken when handling mercury because elemental mercury is absorbed through the skin. Mercury is very volatile and is toxic if inhaled. All work should be vented to a hood.

6.0 SAMPLE CONTAINERS, COLLECTION AND PRESERVATION

6.1 SAMPLE CONTAINERS: Sample containers may be either plastic or glass.

6.2 SAMPLE COLLECTION: Samples are collected in plastic or glass bottles.

6.3 SAMPLE PRESERVATION: None.

6.4 HOLDING TIMES: Samples are analyzed within 13 days after collection when collected in plastic containers and within 35 days after collection when collected in glass containers.

7.0 APPARATUS AND MATERIALS

7.1 Instrumentation - either of the following instruments may be used:

7.1.1 Perkin Elmer 5000 Spectrophotometer.

7.1.2 Mercury Monitor LDC 3200 with autosampler.

7.2 High purity nitrogen.

7.3 Calculator with linear regression function.

7.4 5 and 6 mL pipets and pipet bulb.

7.5 Peristaltic pump and tubing.

7.6 Tungsten Lamp.

7.7 Analytical balance capable of weighing 0.20 grams, 0.01 g sensitivity.

7.8 Spatulas.

7.9 Plastic digestate storage bottle, with lid.

8.0 REAGENTS AND STANDARD PREPARATION

8.1 Reagent water - 16.6 megohm-cm, or higher, deionized.

8.2 Aqua regia, prepared immediately before use, three parts HCl and one part HNO_3

8.3 Sulfuric Acid, 0.5N, dilute 14.0 mL of concentrated sulfuric acid to 1 liter.

8.4 Stannous Chloride, 25 g to 250 mL in 0.5N H_2SO_4 this mixture should be stirred continuously as it is a suspension.

8.5 Sodium chloride-Hydroxylamine sulfate solution, dissolve 12 g of each in reagent water and dilute to 100 mL.

8.6 Potassium permanganate. 5% solution dissolve 5 g of potassium permanganate in 100 mL reagent water.

8.7 Potassium persulfate, 5% solution. Dissolve 5 g of potassium persulfate in reagent water and dilute to 100 mL.

8.8 Mercury Calibration Standard Preparation

8.8.1 Stock Mercury Standard is purchased at 1000 ppm from Ricca.

8.8.2 Dilute 1.0 mL of Stock Mercury Standard (Section 8.8.1) to 100 mL in a volumetric flask to prepare a 10 ppm standard solution.

8.8.3 Dilute 1.0 mL of the 10 ppm standard solution (Section 8.8.2) to 100 mL to prepare the 0.1 ppm intermediate standard.

8.8.4 Prepare a working curve from the 0.1 ppm intermediate standard as follows:

0.5 mL to the BOD bottle for the 0.5 ppb standard
1.0 mL to the BOD bottle for the 1.0 ppb standard
3.0 mL to the BOD bottle for the 3.0 ppb standard
5.0 mL to the BOD bottle for the 5.0 ppb standard
10.0 mL to the BOD bottle for the 10.0 ppb standard

8.8.5 For each of the standards prepared in Section 8.8.5, add enough reagent water to make the final volume 10 mL. Add 5 mL of aqua regia. Heat in a hot water bath for 2 minutes at 95°C. Cool the sample and add 50 mL of reagent water and 15 mL of $KMnO_4$. Heat the sample in the bath for 30 minutes. Add 6 mL of sodium chloride-hydroxylamine sulfate to each sample and allow the sample to aerate for thirty seconds.

8.8.6 Transfer the contents of the BOD bottle to a graduated cylinder and dilute to 150 mL with reagent water.

8.8.7 Transfer the contents of the graduated cylinder to a labelled digestate storage bottle.

8.9 Sample spikes receive 1.0 mL of the 10 ppb standard prepared in Sections 8.8.4 - 8.8.7 for every 100 mL of sample digested.

8.10 Initial Calibration Verification (ICV) Preparation

8.10.1 Stock Mercury Standard at 1000 ppm is purchased from Mallinckrodt.

8.10.2 The ICV is at 4 ppm and is prepared by adding 4.0 mL of Stock Mercury Standard (Section 8.10.1) to a BOD bottle and processing the aliquot as in Sections 8.8.5 - 8.8.7.

8.11 Mercury Certified Reference Material is purchased from Environmental Resource Associated to be used in preparation of the Laboratory Control Sample (LCSS).

8.12 All reagents and chemicals are documented in the Chemical Receipt Logbook (Attachment I). Chemical solutions for use in preparation and analysis for mercury are recorded in the Mercury Solutions Logbook (Attachment II).

9.0 CALIBRATION

9.1 Standards from Section 8.8 are to be analyzed in ascending order per the procedure described in Sections 11.2 and 11.3. After all standards are analyzed, the concentration and absorbance is entered into the linear regression function of the calculator. A correlation coefficient of $\geq$ 0.995 is an acceptable linear regression for the curve. Initial calibration acceptance criteria is $\pm$ 10% of the true value.

10.0 QUALITY CONTROL

10.1 Instrument detection limits are calculated based on the EPA SOW 3/90. A minimum of seven replicate standards are analyzed at three to five times the expected IDL on three nonconsecutive days and the average standard deviation multiplied by three to determine the IDL.

10.2 The Practical quantitation limit for this method is 0.10 ug/kg.

10.3 MDLs are determined in accordance with the SOP for Conducting MDL studies, IEA Doc# QAS02000.NET.

10.4 The Initial Calibration Blank (ICB), Continuing Calibration Blank (CCB) and Preparation Blank consist of reagent water that is prepared as in Sections 8.8.5 - 8.8.7.

10.5 The LCSS is prepared and analyzed as in Section 11.0 using 0.20 - 0.25 g of Mercury Certified Reference Material (Section 8.11). The LCSS is not prepared in triplicate.

10.6 The Initial Calibration Verification (ICV) is prepared as in Section 8.10.

10.7 A Low Detection Limit Standard or Contract Required Standard (CRA) is prepared at 0.2 ppb by dilution and preparation of 0.2 mL of the 0.1 ppm standard (Section 8.8.3) as in Sections 8.8.5 - 8.8.7.

10.8 A sample duplicate is prepared as in Section 11.1 and is analyzed every 10 samples.

10.9 The 5 ppb standard prepared in Sections 8.8.4 - 8.8.7 is used as the Continuing Calibration Verification (CCV) and is analyzed every ten samples.

10.10 Sample spikes and sample spike duplicates are prepared as in Section 8.9.

10.11 The control limits, frequencies, and corrective actions for Quality Control Samples such as Sample Spikes, Duplicates, etc. are described in Table One.

11.0 SAMPLE PREPARATION AND INSTRUMENTAL PROCEDURES

11.1 Sample Preparation

11.1.1 Weigh a 0.2 g aliquot of sample in triplicate into clean 300 mL BOD bottles. Record the weight in the Mercury Digestion Logbook (Attachment III).

11.1.2 Add 5 mL of reagent water and 5 mL of aqua regia and heat in a hot water bath for 2 minutes at 95°C.

11.1.3 Cool the sample and add 50 mL of reagent water and 15 mL of $KMnO_4$. Heat the sample in the bath for 30 minutes.

11.1.4 Add 6 mL of sodium chloride-hydroxylamine sulfate to each sample and allow the sample to aerate for thirty seconds.

11.1.5 Transfer the contents of the BOD bottle to a graduated cylinder and dilute to 150 mL with reagent water.

11.1.6 Transfer the contents of the graduated cylinder to a labelled digestate storage bottle.

11.1.7 Sample spikes receive 1.0 mL of the 10 ppb standard prepared in Section 8.8.4 - 8.8.7 for every 100 mL of sample digested.

11.2 PE 5000 Analysis of Mercury

11.2.1 Place a cart in front of the PE 5000 and set up the stirrer and the peristaltic pump.

11.2.2 Attach a short length of Tygon tubing to the pump and attach the bubbler to the other end of the tubing. Run another length of tubing from the outlet on the bubbler to the inlet on the cell.

11.2.3 Make sure that all connections are air tight.

11.2.4 Place the bubbler in a digestate storage bottle containing about 150 mL of reagent water. Turn the pump on and set the air flow to deliver one cubic liter of air per minute.

11.2.5 Place a 1000 mL plastic bottle containing the stannous chloride on the stirrer with a stir bar in the bottle and turn the stirrer on. The stirrer should remain on at all times.

11.2.6 Turn the unit ON and turn the standby switch to RUN. Wait for the keyboard indicator to light up.

11.2.7 Plug in the mercury lamp and place it in the turret. Type the corresponding slot # for the lamp on the keyboard and press LAMP #. Type in the operating current on the mercury lamp and press LAMP MA.

11.2.8 To set the wavelength, type 253.7 and press PEAK. To set the slit width, type .7 and press SLIT HIGH.

11.2.9 To maximize the lamp, press SETUP and adjust the lamp until the reading on the instrument peaks. Then press SETUP again to exit. Press .5, t, CONT, REC ABS, ABS, AA.

11.2.10 Physically adjust the cell using the three silver knobs until the lowest reading is obtained. Press AZ to auto zero the instrument. Allow the instrument to stabilize for at least 10 minutes. The energy output should show at least 59.

11.2.11 Place the digestate storage bottle containing the sample to be run next to the bubbler on the cart. Pipet 5 mL of stannous chloride solution into the sample and **immediately** insert the bubbler into the sample. Do not disturb the sample during the read cycle.

11.2.12 Record the highest absorbance reading from the instrument readout in the Mercury Analysis Runlog (Attachment IV).

11.2.13 Leave the bubbler in the sample until all the mercury has been evolved and then place the bubbler back in the digestate storage bottle containing reagent water.

11.2.14 Repeat this process for each sample.

11.2.15 Calculate the concentration of mercury in each sample and record in the Mercury Analysis Runlog.

11.2.16 Upon completion of the run type 0 and press LAMP MA. Turn off the PE 5000 by switching the instrument to STANDBY and OFF. Clean up the area and remove pump tubing.

11.3 Mercury Monitor LDC 3200 Analysis of Mercury

NOTE: The operator of this instrument should familiarize himself/herself with WINDOWS software and with HGTALK software before operating this instrument.

11.3.1 Remove the Grey dryer tube (located on the front of the Mercury Monitor LDC 3200). Take off the bottom end of the tube and remove all the hardened magnesium perchlorate. Refill tube and reconnect to pneumatic tubes.

11.3.2 Turn on the computer. When "C:>" appears type in "Win" and hit "Return".

11.3.3 Power up the system components. The power switches for the Mercury Module and the Mercury Monitor LDC 3200 are located on the fronts of the components in the bottom right corner. The power switch for the Autosampler 2000 is located on the rear panel. Allow these to warm up for at least thirty minutes.

11.3.4 The flow meter is scaled from 0 to 10 and if nitrogen, argon or compressed air are used as the carrier gas, this should be set at three. This may be adjusted by rotating the "gas flow regulator". The reagent pressure gauge displays the reagent bottle pressure. This should be set from 4-6 psi. This is adjusted using the reagent regulator located on the rear panel of the unit.

11.3.5 A window directory will be on the screen. Place the arrow on "Cancel" and click once. Open a sequence file by placing the arrow on "Edit" at the top of the screen and clicking once. Place the arrow on "Edit Sequence" and click once.

11.3.6 A directory window containing sequences will be displayed. Choose AQUES.HSQ by clicking on it and then clicking once on "OK". A header will be displayed. Place the arrow on "Data File". Enter the new data file and click once on "OK".

11.3.7 The operator should enter his/her name and the date the samples were digested. Click once on "OK". Enter the samples in the correct vial positions. Move the arrow to "File",located in the top left corner of the screen and click once. Place the arrow on "Save" and click once. Open the "File" menu again and click on "Exit".

11.3.8 Move the arrow to "File" and click once. A window will appear that the file is about to be overwritten, press "OK". Another window will appear saying the results file will be overwritten. Click on "No". Enter a new results file name (use the same file name as the data file) and click once on "OK".

11.3.9 Load the standards and samples into their corresponding vial positions. Press F3 on the keyboard. Advance the autosampler to the correct position by pressing the "Advance Button", located on the rear of the autosampler, until the autosampler syringe is above the first standard. The system is now running.

11.3.10 The analysis must be documented in the Mercury Runlog (Attachment V). The LDC printout will be attached to a copy of the runlog and the analytical data filed by batch. The internal digestate COC is completed when digestates are removed for analysis and upon return to storage for all Level III analyses or samples designated for internal COCs.

12.0 CALCULATIONS

12.1 Sample Spike Recovery: $\%\ R = [(SSR - SR)/SA] \times 100$

SSR is Sample spike recovery in ug/L
SR is Sample result in ug/L
SA is Spike added in ug/L

12.2 $\%\ RPD = \left| (| A - B |)/ [(A + B)/2] \right| \times 100$

A is the sample result in ug/L
B is the duplicate result in ug/L

13.0 ACCEPTANCE CRITERIA

13.1 Initial Calibration	$\geq$ 0.995
Initial Calibration Verification	$\pm$ 10%
Continuing Calibration Verification	$\pm$ 20% of the true value
Initial Calibration Blank	< PQL
Continuing Calibration Blank	< PQL
Method Blank	< Reporting Limit (0.10 ug/kg)
Duplicates	$\pm$ 20% RPD
	$\pm$ Reporting Limit if sample concentration < 5 x Reporting Limit
Laboratory Control Sample	95% Confidence Window

14.0 REPORTING OF RESULTS

14.1 Practical Quantitation Limit:	0.10 ug/kg
Units of measure:	ug/kg
Significant Figures:	5 figures
Data Reporting:	Below quantitation limit: BQL
QC Information included in report:	Level I QC: Blank values, sampling COCs Level II QC: Blank values, duplicate data (%RPD), spike data (% Recovery), sampling COCs Level III QC: Blank values, duplicate data (%RPD), spike data (%Recovery), ICV/CCV, LCSS, narrative, sampling and internal COCs

15.0 SUPPLEMENTAL DOCUMENTS

15.1 SOP for Conducting MDL Studies, IEA Doc# QAS02000.NET.

16.0 REFERENCES

16.1 "Test Methods for Evaluating Solid Waste", SW-846 Third Edition, September 1986, USEPA, Method 7471.

17.0 SUBSTANTIVE REVISIONS

17.1 Test codes and spike levels were revised in this SOP. Also triplicate preps were added to match the method.

17.2 Section 8.1 amended from 18.0 to 16.6 mega-ohm d.i. reagent water. Addendum to 8.8.1 to include concentration of intermediate standard. Amendments to 8.8.1 to correct concentrations of standards. Addendum to Section 9.1 to include initial calibration acceptance criteria. Section 12.2 amended to more clearly express absolute value. Addendum to Section 13.0 to include initial acceptance criteria. Addendum to Table #1 to include initial calibration control limit.

17.3 Section 6.4 revised: Holding time changed from 28 days after collection to 13 days after collection for plastic bottles and 35 days after collection for glass. Addenda to Section 7.0: Section 7.1.1 added to include the Mercury Monitor 3200. Section 8.1 revised: megaohm changed to megohm-cm, or higher. Addendum to Section 8.0: Section 8.11 added to reference the Chemical Receipt Logbook and the Mercury Solutions Logbook. Addendum to Section 9.1: Correlation coefficient criteria defined. Initial calibration acceptance criteria clarified. Sections 10.2, 13.1, 14.1 and Table One revised to correct the PQL. Addenda to Section 10.0: Section 10.4 added to reference the SOP for Conducting MDL Studies. Addendum to Section 11.0: Section 11.2.12 added to reference the Mercury Runlog. Section 11.3 added to discuss mercury analysis by the Mercury Module 3200. Addendum to Section 13.1: Acceptance criteria for LCS, CCB and CCV defined. Addenda to Section 14.1: Level III QC added and sampling COCs added to Level I and Level II reporting. Addenda to Section 15.0: Section 15.1 added to reference the SOP for Conducting MDL Studies. Table #1 titled. Addendum to Table One: Sample Spike Duplicate control limit and corrective action listed. The frequency of QC samples added to Table #1. (05/10/96)

Quality Control Sample Control Limits and Corrective Action

Table #1

Quality Control Sample	Control Limit	Frequency	Corrective Action
IC	$\geq$ 0.995 correlation coefficient	Onset of Analysis	Recalibrate
ICV	$\pm$ 10%	Onset of Analysis	Recalibrate
ICB	$\pm$ 0.10 ug/kg	Onset of Analysis	Recalibrate
CRA	$\pm$ 20%	Onset of Analysis	None
CCV	$\pm$ 20%	1/10 samples	Rerun Samples
CCB	$\pm$ 0.10 ug/kg	1/10 samples	Rerun Samples
Duplicate	$\pm$ 20% RPD	1/10 samples	Flag Sample
Sample Spike	$\pm$ 25% Recovery	1/20 samples	Flag Sample
Sample Spike Duplicate	$\pm$ 20% RPD	1/20 samples	Flag Sample
Prep Blank	$\pm$ 0.10 ug/kg	1/20 samples	Reprep Samples
Lab Control Sample	95% Confidence Window	1/20 samples	Reprep Samples

Attachment I

Metals Lab Chemical Receipt Log

DATE	LOT #	VENDOR	REFERENCE #	GRADE	QTY	VOLUME/MASS	ANALYST

Secondary Review:_____ By:_____

IEA, Inc. Form# MEL01600.NC

Attachment II

Mercury Reagent Logbook
Metals Laboratory

Date	Analyst	Solution ID.	Description of Preparation	Expiration Date

Secondary Review By:_____ Date:_____

IEA, Inc. Form# MEF01100.NC

IEA Logbook# ME15*

Attachment III

Mercury Analysis Runlog
Instrument-*

Instrumental Analyst		SDG Number		Computer File Name	
Instrumental Analysis Date		SOP #		Correlation	

Sample ID#	Batch	Dilution Factor	Recovery	Comments

Secondary Review By:_____ Date:_____

IEA, Inc. Form# MEL00502.NC

IEA Logbook# ME16*

19. IEA SOP FOR SW-846 7470 MERCURY ANALYSIS IN WATER

1.0 APPROVALS

The signatures of the following individuals indicate that this SOP is complete and meets the requirements specified in corporate document # QAS00200.NET. In addition, it signifies that the content meets the specifications of the referenced "Test Code".

Laboratory Director

Quality Assurance Manager

2.0 SCOPE AND APPLICATION

2.1 This SOP defines the analysis of samples of by cold-vapor atomic absorption for samples of an aqueous matrix. Samples are digested and then analyzed for mercury by reduction with stannous chloride.

2.2 The element determined is defined as "Total Metals" for a digested sample and as "Dissolved Metals" for a filtered and then digested sample.

2.3 The IEA test codes for this analysis are HG_15, HG_17, HG_45, HG_47, HG_51, HG_52, HG_53.

3.0 SUMMARY OF METHOD

3.1 This method is used to analyze samples that have been digested for mercury analysis. Samples are introduced into a flow cell with a nitrogen carrier gas where they absorb light at a characteristic wavelength. As the intensity of light absorbed is proportional to the concentration of mercury in the sample, these values are compared to a standard curve and the concentration of mercury determined.

3.2 This method is based on EPA method 7470.

3.3 This method does not deviate from the EPA method 7470.

4.0 INTERFERENCES

4.1 Potassium permanganate is added to eliminate possible interference from sulfide. Concentrations as high as 20 mg/L of sulfide do not interfere with the recovery of added mercury.

4.2 Copper has been reported to interfere; however, copper concentrations as high as 10 mg/L have no effect on mercury recovery.

4.3 High levels of free chlorine interfere with the analysis as chlorine absorbs at a wavelength of 253.7 nm also. Potassium permanganate oxidizes chlorides to chlorine. Therefore, additional potassium permanganate may be needed for seawaters, brines, etc. where salt content of the samples may be high. Hydroxylamine sulfate is added to the sample and the cell is purged with nitrogen before stannous chloride is added to the sample to remove any chlorine interferences.

4.4 Certain organic compounds interfere with mercury analysis. When organic interference is suspected, the sample should be run without reagents to check for this interference.

5.0 SAFETY

5.1 Care should be used when working with concentrated acids and all protective gear worn. Safety glasses, lab coat and latex gloves are recommended to protect against glass breakage and sample spillage.

5.2 **Mercury is very toxic !** Care should be taken in handling mercury as elemental mercury is absorbed through the skin. Mercury is very volatile and is toxic if inhaled. All work should be vented to a hood.

6.0 SAMPLE CONTAINERS, COLLECTION AND PRESERVATION

6.1 SAMPLE CONTAINERS: Sample containers may be either plastic or glass, 500 mL capacity or more and thoroughly rinsed and dried before sampling. Sample bottles are not to be reused.

6.2 SAMPLE COLLECTION: Samples are collected in plastic or glass bottles with nitric acid as a preservative to a pH of less than two.

6.3 SAMPLE PRESERVATION: Sample preservation is with nitric acid as noted above for this method.

6.4 HOLDING TIMES: Samples are analyzed within 13 days after collection for samples collected in plastic containers and 35 days after collection for samples collected in glass containers.

7.0 APPARATUS AND MATERIALS

7.1 Perkin Elmer 5000 Spectrophotmer

7.2 High purity nitrogen

7.3 Calculator with linear regression function

7.4 5 and 6 mL pipets and pipet bulb

7.5 Peristaltic pump and tubing

7.6 Tungsten Lamp

8.0 REAGENTS AND STANDARD PREPARATION

8.1 Reagent water - 16.6 megohm-cm, or higher, deionized

8.2 Concentrated Sulfuric Acid, Reagent Grade

8.3 Concentrated Nitric Acid, Trace Grade

8.4 Stannous Chloride, 25 g to 250 mL in 0.5N H_2SO_4 this mixture should be stirred continuously as it is a suspension

8.5 Sodium chloride-Hydroxylamine sulfate solution, dissolve 12 g of each in DI water and dilute to 100 mL

8.6 Potassium permanganate, 5% solution dissolve 5 g of potassium permanganate in 100 mL DI water

8.7 Potassium persulfate, %5 solution dissolve 5 g of potassium persulfate in DI water to 100 mL

8.8 1000 ppm Stock Mercury Standard

8.8.1 Dilute 1.0 mL of Stock Mercury Standard (Section 8.8) to 100 mL in a volumetric flask and then 1.0 mL of this solution to 100 mL to make the 0.1 ppm intermediate standard. From this standard, prepare a working curve as follows:

0.5 mL to the BOD bottle for the 0.5 ppb standard

1.0 mL to the BOD bottle for the 1.0 ppb standard
3.0 mL to the BOD bottle for the 3.0 ppb standard
5.0 mL to the BOD bottle for the 5.0 ppb standard
10.0 mL to the BOD bottle for the 10.0 ppb standard

8.9 For each of the standards prepared in Section 8.8.1, add enough DI water to make the final volume 100 mL. Add 5 mL of concentrated H_2SO_4 and 2.5 mL of concentrated HNO_3 to each standard, mixing after each addition. Next add 15 mL of potassium permanganate solution to each standard and allow to stand for 15 minutes. Add 8 mL of potassium persulfate to each sample and heat for two hours in the hot water bath at 95°C. Allow the sample to cool. Add 6 mL of sodium chloride-hydroxylamine sulfate solution to the sample and allow it to aerate for thirty seconds.

8.10 Sample spikes receive 1.0 mL of the final solution prepared in Section 8.8.1 for every 100 mL of sample digested.

8.11 All reagents and chemicals are documented in the Chemical Receipt Logbook (Attachment I). Chemical solutions for use in preparation and analysis for mercury are recorded in the Mercury Solutions Logbook (Attachment II).

9.0 CALIBRATION

9.1 Standards are to be run in ascending order per the procedure described in Section 11.2. After all standards are run the concentration and absorbance is entered into the linear regression function of the calculator. Initial calibration acceptance criteria is $\geq$ 0.995 correlation coefficient.

10.0 QUALITY CONTROL

10.1 Instrument detection limits are calculated based on the EPA SOW 3/90. A minimum of seven replicate standards are analyzed at three to five time the expected IDL on three nonconsecutive days and the average standard deviation multiplied by three to get the IDL.

10.2 Practical quantitation limits for this method is 0.20 ug/L.

10.3 Sample Spikes, Duplicates, etc are described in Table One.

10.4 MDLs are determined in accordance with the SOP for Conducting MDL studies, IEA Doc# QAS02000.NET.

11.0 SAMPLE PREPARATION AND INSTRUMENTAL PROCEDURES

11.1 Transfer 100 mL aliquot of sample to a clean 300 mL BOD bottle. Add 5 mL of concentrated H_2SO_4 and 2.5 mL of concentrated HNO_3 to the sample, mixing after each addition. Next add 15 mL of potassium permanganate solution to the sample. Some samples may require additional potassium permanganate if the sample losses it's purple color after the permanganate is added. Potassium permanganate should be added until the purple color persists for a least 15 minutes. Next add 8 mL of potassium persulfate to each sample and heat for two hours in the hot water bath at 95°C. Allow the sample to cool. Add 6 mL of sodium chloride-hydroxylamine sulfate solution to the sample and allow it to aerate for thirty seconds.

11.2 Place a cart in front of the PE 5000. Set up the stirrer and the peristaltic pump. Attach a short length of tygon tubing to the pump and attach the bubbler to the other end of the tubing. Next run another length of tubing from the outlet on the bubbler to the inlet on the cell. Make sure all connections are air tight. Put the bubbler in a BOD bottle containing about 150 mL of DI water. Turn the pump on and set the air flow to deliver one cubic liter of air per minute. Place a 1000 mL plastic bottle containing the stannous chloride on the stirrer with a stir bar in the bottle and turn the stirrer on. The stirrer remains on at all times.

11.3 Operation of the PE 5000 is as follows. Turn the unit ON and the standby switch to RUN. Wait for the keyboard indicator to light up. Plug in the mercury lamp and place in the turret. Type the corresponding slot # for the lamp on the keyboard and press LAMP #. Type in the operating current on the mercury lamp and press LAMP MA. To set the wavelength type 253.7 and press PEAK. To set the slit width type .7 and press SLIT HIGH. To maximize the lamp press SETUP and adjust the lamp until the reading on the instrument peaks. Then press SETUP again to exit. Next press .5, t, CONT, REC ABS, ABS, AA. Physically adjust the cell using the three silver knobs until the lowest reading is obtained. Next press AZ to auto zero the instrument. Allow the instrument to stabilize for at least 10 minutes. The energy output should show at least 59.

11.4 To analyze the standards and the samples place the BOD bottle containing the sample to be run next to the bubbler on the cart. Pipet 5 mL of stannous chloride solution into the BOD and **immediately** insert the bubbler into the sample BOD. Do not disturb the sample BOD during the read cycle. Record the highest absorbance reading from the instrument readout in the mercury note book. Leave the bubbler in the BOD until all the mercury has been evolved. Then place the bubbler back in the BOD with the DI water. Repeat this process for each sample. Use the calculator to calculate the concentration on each sample and record in the notebook. If the sample is a leachate then an analytical spike must also be run and a single point standard addition calculated from these two

runs.

11.5 Upon completion of the run type 0 and press LAMP MA. Turn off the PE 5000 by switching the instrument to STANDBY and OFF. Clean up the area and remove pump tubing.

11.6 The analysis must be documented in the Mercury Runlog (Attachment III). The LDC printout will be attached to a copy of he runlog and the analytical data will be filed by batch.

12.0 CALCULATIONS

12.1 Sample Spike Recovery: $\% R = [(SSR - SR)/SA] \times 100$

SSR is Sample spike recovery in ug/L
SR is Sample result in ug/L
SA is Spike added in ug/L

12.2 $\% RPD = \left| (| A - B |)/ [(A + B)/2] \right| \times 100$

A is the sample result in ug/L
B is the duplicate result in ug/L

13.0 ACCEPTANCE CRITERIA

13.1 Initial Calibration	$\geq$ 0.995
Initial Calibration Verification	$\pm$ 10%
Method Blank	Reporting Limit (0.20 ug/L)
Duplicates	$\pm$ 20% or $\pm$ Reporting Limit if sample concentration < 5 x Reporting Limit

14.0 REPORTING OF RESULTS

Practical Quantitation Limits:	0.20 ug/L.
Units of measure:	ug/L
Significant Figures:	5 figures
Data Reporting:	Below quantitation limit: BQL

QC Information included in report: Level I QC: Blank values, sampling COCs
Level II QC: Blank values, duplicate data (%RPD), spike data (% Recovery), sampling COCs
Level III QC: Blank values, duplicate data (%RPD), spike data (%Recovery), ICV/CCV, LCSW, narrative, sampling and internal COCs

15.0 SUPPLEMENTAL DOCUMENTS

15.1 SOP for Conducting MDL Studies, IEA Doc# QAS02000.NET.

16.0 REFERENCES

16.1 "Test Methods for Evaluating Solid Waste", SW-846 Third Edition, September 1986, USEPA, Method 7470.

17.0 SUBSTANTIVE REVISIONS

17.1 Test codes and spike levels were revised in this SOP.

17.2 Section 8.1 amended from 18.8 to 16.0 mega-ohm d.i. reagent water. Addendum to Section 8.8.1 to include the concentration of the intermediate standard. Amendment to Section 8.8.1 to correct standard concentrations. Addendum to Section 9.1 to include initial calibration acceptance criteria. Section 12.2 amended to more clearly express absolute value. Addendum to Section 13.0 to include initial calibration acceptance criteria. Addendum to Table #1 to include initial calibration control limit.

17.3 Section 6.4 revised: Holding time changed from 28 days after collection to 13 days after collection for plastic bottles and 35 days after collection for glass. Section 8.1 revised: megaohm changed to megohm-cm or higher. Addendum to Section 8.0: Section 8.11 added to reference the Chemical Receipt Logbook and the Mercury Solutions Logbook. Addenda to Section 10.0: Section 10.4 added to reference the SOP or Conducting MDL Studies. Addendum to Section 11.0: Section 11.6 added to reference the Mercury Runlog. Addenda to Section 14.1: Level III QC added and sampling COCs added to Level I and Level II reporting. Addenda to Section 15.0: Section 15.1 added to reference the SOP for Conducting MDL Studies. Addendum to Table One: Sample Spike Duplicate control limit and failure action listed.

Table #1

Quality Control Sample	Control Limit	Failure Action
IC	0.995	Recalibrate
ICV	± 10%	Recalibrate
ICB	± 0.20 ug/L	Recalibrate
CRA	± 20%	None
CCV	± 20%	Rerun Samples
CCB	± 0.20 ug/L	Rerun Samples
Duplicate	± 20% RPD	Flag Sample
Sample Spike	± 25% Recovery	Flag Sample
Sample Spike Duplicate	± 20% RPD	Flag Sample
Prep Blank	± 0.20 ug/L	Reprep Samples
Lab Control Sample	± 20%, 95% Confid Win	Reprep Samples

Attachment I

Metals Lab Chemical Receipt Log

DATE	LOT #	VENDOR	REFERENCE #	GRADE	QTY	VOLUME/MASS	ANALYST

Secondary Review:_____ By:_____

Attachment II

Mercury Reagent Logbook
Metals Laboratory

Date	Analyst	Solution ID.	Description of Preparation	Expiration Date

Secondary Review By:_____ Date:_____

Attachment III

Mercury Analysis Runlog
Instrument-*

Instrumental Analyst		SDG Number		Computer File Name	
Instrumental Analysis Date		SOP #		Correlation	

Sample ID#	Batch	Dilution Factor	Recovery	Comments

Secondary Review By:_____ Date:_____

IEA, Inc. Form# MEL00502.NC

20. IEA SOP FOR TJA 61E TRACE METALS SW-846 6010A

1.0 APPROVALS

The signatures of the following individuals indicate that this SOP is complete and meets the requirements specified in corporate document # QAS00200.NET. In addition, it signifies that the content meets the specifications of the referenced "Test Code".

Laboratory Director

Quality Assurance Manager

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PROPRIETARY DOCUMENT

2.0 SCOPE AND APPLICATION

2.1 This SOP defines the analysis of samples by inductively coupled argon plasma for samples of both solid, aqueous matrix, and TCLP extracts. Sample are introduced into a plasma where they emit light which is measured and the concentrations of those samples determined.

2.2 The elements determined are defined as "Total Metals" for a digested sample and as "Dissolved Metals" for a filtered and undigested sample.

2.3 The IEA test codes for this analysis are all test codes with the format EE_YX where EE is the element symbol from the periodic table and Y is 5 (5=SW846) and X is either 1, 2, 3, or 4 (1=Total Waters, 2=Dissolved lab filtered, 3=Dissolved field filtered, and 4=Soil samples). Also all EE_15 and EE_45 (15=NC TCLP and 45=Federal TCLP).

3.0 SUMMARY OF METHOD

3.1 Samples that have been previously digested for ICAP analysis are analyzed using a TJA Trace ICAP with an axial torch. Samples are nebulized with a meinhard nebulizer and a cyclonic spray chamber into an axial mounted plasma. Element-specific atomic-line emission spectra are dispersed by a grating polychrometer and the intensities of each line is measured by an array of photomultiplier tubes. Background correction is performed off peak center or by "Crawford/Kunselman" background correction technique.

3.2 This SOP is based on method SW-846 6010A.

3.3 This SOP deviates from method 6010A in respect to the following. RPD acceptance criteria for samples is five times the reporting limit instead of ten times the instrument detection limit.

4.0 INTERFERENCES

4.1 ICAP is subject to spectral interferences from overlap of the analytical line by other element emission lines, background continuum or recombination phenomena, overlap of molecular band spectra, and stray light from the line emission of high concentration elements. Background and stray light effects are corrected for by background correction as mentioned in section 3.1. Molecular band overlap is minimized by careful selection of emission lines and plasma operating conditions. Spectral overlap is corrected by Interelement Correction Factors (IECs), which are updated annually or as needed. Interelement Correction is performed automatically by the software before the raw data is generated.

4.2 Physical interferences occur due to problems with sample transport and nebulization due to viscosity and surface tension changes. These effects are overcome by use of a mass flow controller on the sample gas flow. Internal standards are also used to correct for physical interferences. Scandium and Yttrium are used as internal standards.

4.3 Chemical interferences are not normally present to much extent in ICAP analysis due to the high temperature of the argon plasma. Chemical interferences that may be present can be further minimized by careful selection of operating parameters and matrix matching.

5.0 SAFETY

5.1 Care should be used when working with concentrated acids and all protective gear worn. Safety glasses, lab coat and latex gloves are recommended to protect against glass breakage and sample spillage.

5.2 The torch produces intense ultraviolet light and should not be viewed without protection. Typical plasma temperature is between 6000 and 10,000 degrees Kelvin. Care should be used to insure that all parts are cool before attempting to service the instrument.

5.3 The instrument utilizes high voltages and service work should be restricted to qualified service personnel.

6.0 SAMPLE CONTAINERS, COLLECTION AND PRESERVATION

6.1 Sample Containers: Sample containers may be either plastic or glass, 500 mL capacity or more and thoroughly rinsed and dried before sampling. Sample bottles are not to be reused.

6.2 Sample Collection: Samples are collected in plastic or glass bottles with nitric acid as a preservative to a pH of less than two.

6.3 Sample Preservation: Samples are preserved with 3 mL of 1:1 HNO_3 acid to a pH of less than two. Additional acid may be necessary for some samples.

6.4 Holding Times: 180 days from the date of collection.

7.0 APPARATUS AND MATERIALS

7.1 Thermo Jarrell Ash Trace 61E Vacuum ICAP.

7.2 High purity argon, welders grade or better

7.3 13 x 100 mm plastic test tubes

7.4 100 uL pipet

7.5 5.0 or 10.0 mL pipet

8.0 Reagents and Standard Preparation

8.1 Reagent water - 16.6 megohm or higher, deionized

8.2 1% HCl and 1% HNO_3 rinse, To a twenty liter carboy containing approximately five liters of 8.1 add 200 ml concentrated HCl and 200 mL concentrated HNO_3. Fill the remainder of the carboy with 8.1.

8.3 1000 mg/L Yttrium.

8.4 1000 mg/L Scandium.

8.5 CAL4 Stock, 1000 mg/L of Aluminum, Arsenic, Barium, Beryllium, Cadmium, Chromium, Cobalt, Copper, Iron, Lead, Lithium, Manganese, Nickel, Selenium, Strontium, Thallium, Vanadium, and Zinc and 100 mg/L of Silver.

8.6 CAL3 Stock, 10000 mg/L of Calcium, Sodium, Potassium, and Magnesium.

8.7 CAL2 Stock, 1000 mg/L of Tin, Boron, Titanium, and Molybdenum.

8.8 CAL1 Stock, 1000 mg/L of Antimony.

8.9 10000 mg/L Calcium.

8.10 10000 mg/L Magnesium.

8.11 10000 mg/L Iron.

8.12 10000 mg/L Aluminum.

8.13 Concentrated HCl Acid.

8.14 Concentrated HNO_3 Acid.

8.15 Internal Standard Stock, To a 100.0 mL volumetric flask add 20 mL of 8.1 and 1.0 mL of 8.14. Pipet 50.0 mL of 8.3 and 10.0 mL of 8.4 into the flask. Dilute to volume with 8.1.

8.16 Working Standards are prepared as follows:

8.16.1 Blank, To a 200.0 mL volumetric flask containing 50 mL of 8.1 add 10.0 mL of 8.13, 2.0 mL of 8.14 and 2.0 mL of 8.15. Dilute to the mark with 8.1.

8.16.2 CAL4, To a 200.0 mL volumetric flask containing 100 mL of 8.1 add 2.0 mL of 8.14. Next add 2.0 mL of 8.5 and swirl. Add 10.0 mL of 8.13 and swirl again. Dilute to the mark with 8.1.

8.16.3 CAL4D, To a 200.0 mL volumetric flask containing 50 mL of 8.1 add 10.0 mL of 8.13, 2.0 mL of 8.14 and 2.0 mL of 8.15. Dilute to the mark with 8.1.

8.16.4 CAL2D, To a 200.0 mL volumetric flask containing 50 mL of 8.1 add 10.0 mL of 8.13, 2.0 mL of 8.14 and 2.0 mL of 8.7. Dilute to the mark with 8.1. To a second 200.0 mL volumetric flask containing 50 mL of 8.1 add 10.0 mL of 8.13, 2.0 mL of 8.14, 2.0 mL of 8.15 and 20.0 mL of the solution from the first flask. Dilute to the mark with 8.1. This second flask contains CAL2D.

8.16.5 CAL1, To a 200.0 mL volumetric flask containing 50 mL of 8.1 add 10.0 mL of 8.13, 2.0 mL of 8.14 and 2.0 mL of 8.8. Dilute to the mark with 8.1.

8.16.6 CAL1D, To a 200.0 mL volumetric flask containing 50 mL of 8.1 add 10.0 mL of 8.13, 2.0 mL of 8.14 and 2.0 mL of 8.16.5. Dilute to the mark with 8.1.

8.16.7 CAL3, To a 200.0 mL volumetric flask containing 50 mL of 8.1 add 10.0 mL of 8.13, 2.0 mL of 8.14, 2.0 mL of 8.15 and 2.0 mL of 8.6. Dilute to the mark with 8.1.

8.16.8 CAL5, To a 100.0 mL volumetric flask containing 50 mL of 8.1 add 5.0 mL of 8.13, 1.0 mL of 8.14, 1.0 mL of 8.15, 2.0 mL of 8.9, 2.0 mL of 8.10, 2.0 mL of 8.12 and 1.0 mL of 8.11. Dilute to the mark with 8.1.

9.0 CALIBRATION

9.1 Calibration standards are as follows, BLANK, CAL4D, CAL4, CAL2D, CAL1D, CAL1, CAL3, CAL5. Standards are analyzed in that order.

9.2 Working ranges and specific elements for each standard can be found in table two.

9.3 From the Analysis Menu press F3. The standards will be displayed on the screen. Place the cursor over the standard to be run and press F1 to run the standard after placing the probe in the standard. When the standard is complete press F9 to save the standard. Continue this process until all standards are run. After all standards are run press F9 to print the calibration. After the standards are printed press F9 to save the calibration. The standards are to be run in the order stated in 9.1. Internal standard must be added to CAL4 and CAL1 before these standards are run. All other standards have the internal already added.

9.4 1% Internal Standard (8.15) is added to all samples, QC, and standards.

9.5 Calibration correlation coefficient should be 0.995 or greater for all elements.

10.0 QUALITY CONTROL

10.1 MDLs are performed and calculated in accordance with DOC # QAS02000.NET.

10.2 Practical Quantitation Limits (PQL's) can be found in table two.

10.3 Daily Performance Checks are as follows.

10.3.1 Profile of the spectrum shifter using a five mg/L arsenic standard. Spectrum shifter position should be zero (0) before beginning the run.

10.3.2 Cadmium Background Ratio (BR) should be checked to insure proper instrument sensitivity. Cadmium BR should be 22.000. to 30.000.

10.4 Matrix Sample spikes (MS) are to be performed one for every twenty samples digested per matrix or one per batch, whichever is more frequent.

10.5 Matrix Sample Duplicates (MSD) spikes are to be performed one for every twenty samples digested per matrix or one per batch, whichever is more frequent.

10.6 Continuing Calibration Verification (CCV) and Continuing Calibration Blanks (CCB) are to be performed every ten measurements and CCV and CCB are to be run at the end of the analytical run.

10.7 Duplicates are to be performed one for every twenty samples digested per matrix or one per batch, whichever is more frequent.

10.8 Serial dilutions are to be performed one for every twenty samples digested per matrix or one per batch, whichever is more frequent at a five fold dilution. Serial dilution results should be within $\pm$ 10% RPD.

10.9 Post spikes are to be performed one for every twenty samples digested per matrix or one per batch, whichever is more frequent. Post spikes should recover with 75 to 125 % of the known value.

10.10 Lab Contol Samples are to be performed one for every twenty samples digested per matrix or one per batch, whichever is more frequent.

10.11 QC activities are as follows:

ICV	Beginning of the run.
High Std Read-Back	Beginning of the run.
Interference Check	Beginning and end of run and every 8 hrs
Calibration Check Std. (CCV)	Every 10 samples and at the end of the run.
CCB	Every 10 samples and at the end of the run.
Method Blanks	One per Batch, per day, or every twenty samples
Matrix Spike	One per Batch, per day, or every twenty samples
Matrix Spike Duplicate	One per Batch, per day, or every twenty samples
Serial Dilution	One per Batch, per day, or every twenty samples
Post Spike	One per Batch, per day, or every twenty samples
Lab Control Sample	One per Batch, per day, or every twenty samples

11.0 SAMPLE PREPARATION AND INSTRUMENTAL PROCEDURES

11.1 Autosampler tables are built by pressing the "Ctrl" key and the F3 key at the same time. The instrument will prompt for a macro name. Type AUTONAME and press return. After the screen stops press Ctrl F3 again and this time type AUTOMAKE and press return. The instrument will build a autosampler table with all required QC, except the four required mixed standards. Return to the table using the appropriate keys and place the samples IDs in the sample slots. Insert the four mixed standards CAL4D, CAL1D, CAL2D, and CAL3 by pressing F1 and typing these names. Save these changes by pressing F9 until the table is saved.

11.2 To setup data storage perform the following steps. Press Ctrl F9, D, M, you will be prompted for a method name. Enter the method you are using. Press Enter followed by F3. Arrow down to analysis data file and enter the month, day, year, T, and the letter of the run (i.e. A for the first run of the day, B for the second, etc.). An example would be 82194TA for the first run on August 21, 1994. Press F9 until you exit the method. Press Ctrl F9, C, I followed by F9 until you reach the remote data screen. Arrow down to the remote data file and enter the same file name here. Press F9 until the main screen returns. Leave the software by pressing X, Q. Type CD\STATION\DATA\ANALYTIC and return. Rename the SAMPLES.DBF file to XXXXXXX.SAM using the command REN SAMPLES.DBF XXXXXXXX.SAM, where XXXXXXXXX is some user chosen name. Return to the software by typing WIN followed by return.

11.3 The analysis menu may be reached by pressing Ctrl F9, O, A and entering the method to be run followed by the return key.

11.4 To profile the instrument press the F5 key followed by the F3 key while aspirating a five mg/L solution of arsenic. Next press F1 to start the profile. If the profile is acceptable (between - 0.10 and 0.10) press F9 twice to save the profile. If the profile is not acceptable press F1 to calculate the required SS position and turn the dial on the instrument to this position. Repeat the profile procedure.

11.5 Calibrate as described in section 9.3.

11.6 To load the autosampler tray pour the solution into the QC cups (the L shaped rack). These solutions already have the internal standards present. To load the samples pipet 100 uL of internal standard into the 15 mL test tube and 10 mL of sample. Mix. Place in the correct rack position.

11.7 To run samples press F9 from the analysis menu followed by F1 to start the autosampler tray. Analysis will proceed automatically.

12.0 CALCULATIONS

12.1 $$RPD_{duplicate} = \left| \frac{S - D}{(S+D)/2} \right| \times 100$$

12.2 $$RPD_{MS/MSD} = \left| \frac{MS - MSD}{(MS+MSD)/2} \right| \times 100$$

12.3 $$\text{Recovery} = [(PS - S)/SA] \times 100$$

12.4 $$RPD_{SERIALDILUTION} = \left| \frac{S - SD}{S} \right| \times 100$$

where:

S = Sample result
SD = Serial Dilution result
MS = Matrix Spike result
MSD = Matrix Spike Duplicate result
SA = Spike Added
PS = Post Spike result
D = Duplicate Result

13.0 ACCEPTANCE OF DATA

13.1 Duplicate control limit is as follows:

13.1.1 ± 20% RPD for samples greater than or equal to five times the reporting limit.

13.1.2 ± the reporting limit for samples less than five times the reporting limit.

13.2 Post Spike control limit is 75 to 125% of known value.

13.3 Serial Dilution control limit is ± 10% RPD.

13.4 Control limit for MS and MSD is ± 20% RPD.

13.5 Matrix Spike control limit is 80 to 120 % recovery.

13.6 Method Blanks must be within ± PQL.

13.7 ICV, CCV must be within ± 10% of known value.

13.8 High standard read back must be within ± 5% of known values.

14.0 REPORTING OF RESULTS

14.1 Results are reported as follows:

Level I:	Sample Results, Method Blank Results
Level II:	Level I plus sample spike recovery and duplicate RPD summaries
Level III:	Level II plus ICV and CCV recovery and internal COCs

14.2 Results are reported to three significant figures.

15.0 SUPPLEMENTAL DOCUMENTS

15.1 SOP for Conducting MDL Studies, IEA Doc# QAS02000.NET.

16.0 REFERENCES

16.1 EPA SW-846 Method 6010A.

17.0 SUBSTANTIVE REVISIONS

17.1 Original Issue.

TABLES

Table One

Std. Name	Elements Used from This Standard	Concentration (ug/L)
BLANK	All Elements	0.0
CAL4D	Al, As, Ba, Be, Cd, Cr, Co, Cu, Fe, Mn, Ni, Ag, Tl, V, Zn, Li, Sr, Pb, Se	1000, except Ag (100)
CAL4	Al, As, Ba, Cr, Co, Cu, Fe, Mn, Ni, Ag, Tl, V, Pb, Se	10000, except Ag (1000)
CAL2D	Mo, Sn, Ti, B	1000
CAL1D	Sb	1000
CAL1	Sb	10000
CAL3	Na, K, Ca, Mg	100000
CAL5	Ca, Mg, Al, Fe	100000 Fe,200000 Al,Ca,Mg

Table Two

Element(s)	PQL (ug/L)
Ca, Mg, K, Na	5000
Se, Cd, Be	5.0
As, Tl, Ag, Cr	10.0
Cu	25.0
Co, Mo, V, Ti, Sn, B, Sr, Li	50.0
Zn	20.0
Ni	40.0
Sb	6.0
Pb	3.0
Al, Ba	200.0
Fe	100.0
Mn	15.0

21. IEA SOP FOR METALS DIGESTION METHOD SW-846 3050

1.0 APPROVALS

The signatures of the following individuals indicate that this SOP is complete and meets the requirements specified in corporate document # QAS00200.NET. In addition, it signifies that the content meets the specifications of the referenced "Test Code".

UNCONTROLLED COPY
PROPRIETARY DOCUMENT

Laboratory Director

Quality Assurance Manager

2.0 SCOPE AND APPLICATION

2.1 This document is used to specifically describe the digestion of soils, waters and sludge samples for graphite furnace, ICAP, and flame AA analysis. Samples prepared by these methods may be analyzed for the listed metals.

2.2

ICAP	FLAA		GFAA
All Metals	Aluminum	Magnesium	Arsenic
	Barium	Manganese	Beryllium
	Beryllium	Molybdenum	Cadmium
	Cadmium	Nickel	Chromium
	Calcium	Potassium	Cobalt
	Chromium	Sodium	Iron
	Cobalt	Thallium	Molybdenum
	Copper	Vanadium	Selenium
	Iron	Zinc	Thallium
	Lead		Vanadium

2.3 The test codes for this document are METP54 and METP58.

3.0 SUMMARY OF METHOD

3.1 A representative 1 gm to 2 gm (wet weight) sample is digested in nitric acid and hydrogen peroxide. The digestate is then refluxed with either nitric acid or hydrochloric acid. Hydrochloric acid is used as the final reflux acid for the furnace AA analysis of Sb, the flame AA or ICP analysis of Al, Sb, Ba, Be, Ca, Cd, Cr, Co, Cu, Fe, Pb, Mg, Mn, Ni, K, Ag, Na, Tl, V and Zn. Nitric acid is employed as the final reflux for the furnace AA analysis of As, Be, Cd, Cr, Co, Pb, Mo, Se, Tl, and V.

3.2 This method is based on EPA method 3050.

3.3 This method does not deviate from method 3050.

4.0 INTERFERENCES

4.1 Samples of varying matrices provide different interferences. A list of quality controls are performed to evaluate these and are described in section 10.0.

4.2 Contamination can be a problem in sample preparation. Trace grade acids are used to reduce this problem. These chemicals are described in section 8.0. In addition, a clean work area is very important.

5.0 SAFETY

5.1 This method uses concentrated acids and hydrogen peroxide. These chemicals can cause severe burns and eye damage. Always wear gloves when handling samples or reagents. Wear face shield, lab coat or apron to help protect against spills and splashes. Wear respirator to protect against acid fumes and fumes from samples. Keep hood closed while samples are digesting.

6.0 SAMPLE CONTAINERS, COLLECTION AND PRESERVATION

6.1 Final digestates are to be placed in new 250 mL plastic bottles. Sample bottles are not to be reused.

6.2 Samples should be received at pH <2.

6.3 Samples are stable when digestion is complete and need no preservation.

6.4 Holding time is 180 days from sampling.

7.0 APPARATUS AND MATERIALS

7.1 Equipment

7.1.1 Ventilation Hood

7.1.2 Hotplates

7.1.3 150 mL beakers

7.1.4 Watchglasses

7.1.5 Spatulas

7.1.6 Weighboats

7.1.7 Calibration Weights

7.1.8 Balance

7.1.9 Mortar and pestle

7.1.10 Whatman number 42 filter paper

7.1.11 Funnels

7.1.12 100 mL volumetrics

7.1.13 Plastic bottles

8.0 REAGENTS AND STANDARD PREPARATION

8.1 Barnstead deionized water, 16.6 megohm or higher

8.2 Concentrated Nitric Acid, Trace Grade

8.3 1:1 Nitric Acid, Prepared by mixing one part 8.1 with one part 8.2

8.4 Concentrated Hydrochloric Acid, Trace grade Hydrochloric acid

8.5 30% Hydrogen Peroxide

8.6 For preparation code METP54 the sample spike receives 1.00 mL of IEA-SPK-2 and 1.00 mL of IEA-SPK-1. Samples requiring B, Mo, Si, Sn, or Ti also receive 1.00 mL of IEA-ICV-2.

8.7 For preparation code METP58 the sample spike receives 1.00 mL of FURNACE SPIKE/LCSW.

9.0 CALIBRATION

9.1 Calibration Standards are not prepared as this is a sample preparation SOP only.

9.2 The balance should be calibrated daily prior to use according to Corporate SOP for Calibrating Lab Balances, Doc# QAS01002.NET.

10.0 QUALITY CONTROL

10.1 Preparation blanks are performed to check for contamination. If a preparation blank is above the reporting limit and the samples are not a least ten times the preparation blank value then the samples in the batch are reprepped. The exception to this is if none of the samples are above the reporting limit then the batch is not reprepped.

10.2 A lab control sample is prepared to check that no analyte is lost or gained during the batch preparation. If the lab control sample does not meet the 95 % confidence window or internally established control limits, whichever is more stringent, then the entire batch is reprepped.

10.3 A duplicate sample is prepared and the RPD is calculated from the original sample. If the RPD is greater than 20 % and the sample is greater than five times the reporting limit the samples in that batch are flagged. If the sample is less than five times the reporting limit then the control limit is $\pm$ the reporting limit.

10.4 A sample spike is also performed for each batch. This must agree within $\pm$ 25 % of the spiked value unless the original sample is greater than four times the spike level.

10.5 Control Charts are used to track the results of the lab control sample over time. Trends are noted to evaluate possible future problems such as analytes falling out of solutions and to problem solve, i.e. replacing the solution or standard.

10.6 A sample spike duplicate is also performed for each batch of ICAP samples prepared. This must agree within $\pm$ 25 % of the spiked value unless the original sample is greater than four times the spike level.

10.7 QC related Activities

Prep. Blank	Every Batch not to exceed 20 samples
Lab Control Sample	Every Batch not to exceed 20 samples
Sample Spike	Every Batch not to exceed 20 samples
Duplicate	Every 10 samples
Sample Spike Duplicate	Every Batch not to exceed 20 samples

11.0 SAMPLE PREPARATION AND INSTRUMENTAL PROCEDURES

11.1 Mix the sample thoroughly to achieve homogeneity. For each digestion procedure, weigh (to the nearest 0.01 g) a 1 to 2 g portion of sample and transfer to a breaker.

11.2 Add 10 mL of 1:1 nitric acid (HNO_3), mix the slurry, and cover with a watchglass. Heat the sample to 95° C and reflux for 10 to 15 minutes without boiling. Allow the sample to cool, add 5 mL of concentrated HNO_3, replace the watchglass, and reflux for 30 minutes. Repeat this last step to ensure complete oxidation. Using a ribbed watch glass, allow the solution to evaporate to 5 mL without boiling, while maintaining a covering of solution over the bottom of the beaker.

11.3 After step 11.2 has been completed and the sample has cooled, add 2 mL of deionized water and 3 mL of 30% hydrogen peroxide (H_2O_2). Return the beaker to the hot plate for warming to start the peroxide reaction. Care must be taken to ensure that losses do not occur due to excessively vigorous effervescence. Heat until effervescence subsides.

11.4 Continue to add 30% H_2O_2 in 1 mL aliquots with warming until the effervescence is minimal or until the general sample appearance is unchanged. (NOTE: Do not add more than a total of 10 mL 30% H_2O_2.)

11.5 If the sample is being prepared for the furnace AA analysis of Sb, the flame AA or ICP analysis of Al, Sb, Ba, Be, Ca, Cd, Cr, Co, Cu, Fe, Pb, Mg, Mn, Ni, K, Ag, Na, Tl, V, and Zn, then add 5 mL of concentrated HCl and 10 mL of deionized water, return the covered beaker to the hotplate, and heat for an additional 15 minutes. Cool samples. Filter sample through Whatman No. 42 filter paper into a 100 mL volumetric. Dilute the sample with DI water to volume. Transfer to a new plastic bottle. The sample is now ready for analysis.

11.6 If the sample is being prepared for the furnace analysis of As, Be, Cd, Cr, Co, Pb, Mo, Se, Tl, and V, continue heating the acid-peroxide digestate until the volume has been reduced to approximately 5 mL. Cool the samples. Filter through Whatman No. 42 filter paper into 100 mL volumetric. Dilute the sample with deionized water to volume. Transfer to a new plastic bottle. The sample is now ready for analysis.

12.0 CALCULATIONS

12.1 Results are reported on the basis of the actual dry weight of the sample.

13.0 ACCEPTANCE OF DATA

13.1 Acceptance of the data is described in section 10.0

14.0 REPORTING OF RESULTS

14.1 Each log book page should have a date, analyst's name and batch number.

14.2 A batch system is used. Only 20 samples are allowed in a batch. Each batch should have a duplicate sample, a spiked sample, a blank sample and a laboratory control sample. If the batch is for ICAP, then a sample spike duplicate must also be performed. All of these samples should be indicated in the logbook.

14.3 Record weight, matrix, initial volume and final volume for each sample.

14.4 Record lot number of all solutions and reagents added.

14.5 Record in comment column if sample is diluted, concentrated, limited, preserved, reprepped or other changes.

15.0 SUPPLEMENTAL DOCUMENTS

15.1 None

16.0 REFERENCES

16.1 Method 3050A, SW-846, 2nd ed., Test Methods for Evaluating Solid Waste, EPA Office of Solid Waste and Emergency Response, July 1992 Revision.

16.2 USEPA Contract Laboratory Statement of Work for Inorganic Analysis, Exhibit D, Section 3, Part B.

16.3 Employee Chemical Safety Handbook, Industrial and Environmental Analysts, Inc., 1990.

17.0 SUBSTANTIVE REVISIONS

17.1 Test codes and sample spike spiking levels were updated in this revision.

17.2 Addendum to Section 6.3 to clarify holding time. Section 8.1 amended from 18.0 to 16.6 mega-ohn d.i. reagent water.

17.3 The MSD was added to sections 10.6, 10.7, and 14.2 for ICAP analysis. Addendum to Section 6.0: Section 6.2 added to detail preservation. Section 9.2 revised to comply with corporate policy on balance calibration. Addendum to Section 10.2: Internal limits criteria established. Revision to Section 10.7: Duplicate changed from every 20 to every 10 samples per NC. Section 12.0 revised: Reporting restricted to dry weight only.

22. IEA SOP FOR NON-VOC AND INORGANIC TCLP PREPARATION

1.0 APPROVALS

The signatures of the following individuals indicate that this SOP is complete and meets the requirements specified in corporate document # QAS00200.NET. In addition, it signifies that the content meets the specifications of the referenced "Test Code".

Laboratory Director

Quality Assurance Manager

2.0 SCOPE AND APPLICATION

2.1 The purpose of this Standard Operating Procedure is to outline the specific methods to prepare TCLP samples for non-volatile organic and inorganic metals analysis.

2.2 The following test codes are used to define analyses conducted in accordance with all of the conditions set forth by this SOP:

LCH_10, LCH_11, LCH_12, LCH_13

3.0 SUMMARY OF METHOD

3.1 A known amount of sample is leached using the TCLP extraction in order to analyze the leaching capacity of the sample.

3.2 This SOP deviates from SW-846 Method 1311 in that cellulose filters are substituted for glass fiber filters when filtering samples for metals analyses to prevent metals contamination due to the glass fiber filters.

4.0 INTERFERENCES

4.1 Interferences may occur through contamination of vessels or glassware. These are kept to a minimum by cleaning all reusable parts prior to use.

5.0 SAFETY

5.1 Chemical resistant polyvinyl gloves should be worn at all times when working with solvents.

5.2 Each analyst has access to a personal lab coat and it should be worn at all times in the sample preparation laboratory area.

5.3 MSD sheets are available in the lab area for immediate reference in the case of an emergency.

5.4 Vapor monitors are used quarterly to determine exposure of employees to solvents.

6.0 SAMPLE CONTAINERS, COLLECTION AND PRESERVATION

6.1 Sample containers should be glass walled with Teflon sealed lids to prevent exposure for organic analysis. Should samples require inorganic parameters only, either glass or plastic containers may be used.

6.2 Samples are not preserved prior to sample preparation.

6.3 Holding Times

6.3.1 Samples for organic analyses must be extracted within 14 days of sample collection.

6.3.2 Samples for inorganic metals analysis must be extracted within 28 days for mercury and 180 days for all other metals.

7.0 APPARATUS AND MATERIALS

7.1 Analytical balance - Toploading, accurate to +/- 0.01 g.

7.2 Hazardous Waste Filtration Unit - Millipore Catalog #YT30142HW, or equivalent.

7.3 Nitrogen hose.

7.4 15.0 cm Glass fiber filter (0.6 to 0.8 μ) - Whatman GF/F, Baxter Catalog #F2834-150, or equivalent

7.5 Pipets - 1, 5, 10, and 25 mL, Class A Volumetric.

7.6 500 mL glass TCLP bottles (LCH_11, LCH_12, LCH_13) - Associated Design and Manufacturing Catalog #3740-WGB, or equivalent.

7.7 500 mL plastic storage bottles (LCH_10), Baxter catalog #B7357-11.

7.8 Rotator - Capable of rotating end-over-end fashion at 30 +/-2 rpm.

7.8.1 The rotator rpm must be documented at initial use in the maintenance logbook.

7.9 Stir plate.

7.10 Hot plate.

7.11 250 mL glass beaker.

7.12 Metal spatula.

7.13 100 mL graduated cylinder.

7.14 1 liter graduated cylinder.

7.15 Pipet bulb.

7.16 Drying oven (100 $\pm$ 20°C).

7.17 5.0 mL pipetman.

7.18 1 mL repipetter.

7.19 Cellulose Nitrate Filters (0.45 um).

7.20 pH meter.

7.21 Large glass funnel.

7.22 2 liter plastic bottle, purchased from QEC.

7.23 2 liter glass bottle, purchased from Associated Designs.

7.24 Aluminum foil prepared pans.

7.25 9.5 mm Standard Sieve.

8.0 REAGENTS AND STANDARD PREPARATION

8.1 Reagents

8.1.1 Acetic Acid: 99.7%, ACS reagent, Aldrich Catalog #32,009-9, or equivalent.

8.1.2 Sodium Hydroxide, ACS Grade, Fisher Catalog #5320-500, or equivalent.

8.1.2.1 1N Sodium Hydroxide - Add 40 g Sodium Hydroxide to 1 liter of de-ionized water. Mix well.

8.1.3 De-ionized Water, Reagent Grade ASTM, Type II.

8.1.4 1N HCL - Certified, Fisher Catalog 3 SA48 - 1, or equivalent.

8.1.5 Nitric Acid (Conc.), Reagent ACS grade, Fisher Catalog #A2005-212, or equivalent.

8.1.5.1 10% Nitric Acid - Add 100 mLs concentrated nitric acid to 1000 mLs de-ionized water. Mix well.

8.1.6 pH Buffer 4.0.

8.1.7 pH Buffer 7.0.

8.1.8 pH Buffer 10.0.

8.1.9 Nitrogen - Ultrapure gas.

8.1.10 Preparation of Extraction Fluid #1

8.1.10.1 Add 5.7 mL of Acetic Acid to 500 mL of reagent water and add 64.3 mLs of 1N Sodium Hydroxide. Dilute to 1 liter in de-ionized water and mix well.

8.1.10.2 Confirm the pH of the extraction fluid is 4.93 +/- 0.05 prior to use. If the pH does not meet these requirements, the extraction fluid should be discarded and new fluid should be prepared.

8.1.11 Preparation of Extraction Fluid #2

8.1.11.1 Dilute 5.7 mL of Acetic Acid to 1 liter in de-ionized water and mix well.

8.1.11.2 Confirm the pH of the extraction fluid is 2.88 +/- 0.05 prior to use. If the pH does not meet these requirements, the extraction fluid should be discarded and new fluid should be prepared.

8.1.11.3 Record the date of extraction fluid preparation and the pH on the date of use in the TCLP Extraction Logbook (Attachment I).

8.1.12 Reagent receipt is documented in the Chemical/Standard Receipt Logbook (Attachment V).

8.2 Standards

8.2.1 Stock Mercury Solution is purchased from a commercial vendor and contains 1000 ug/mL of Mercury.

8.2.2 Federal Spike A is a custom standard purchased from High Purity Standards, containing Arsenic, Chromium, Lead at 50 ug/mL; Cadmium, Selenium, Silver at 100 ug/mL; Barium at 10,000 ug/mL.

8.2.3 Federal Spike B is prepared in the metals department by the dilution of 4.0 mL of Stock Mercury Solution (Section 8.2.1) to 200 mL in 1% HNO_3.

8.2.4 North Carolina TCLP Spike A is purchased from High Purity Standards and contains Arsenic, Chromium, Lead, Silver at 25 ug/mL; Cadmium, Selenium at 5 ug/mL; Barium at 500 ug/mL.

8.2.5 North Carolina Spike B is prepared in the metals department by the dilution of 0.4 mL of Stock Mercury Solution to 200 mL in 1% HNO_3.

8.2.6 The receipt of Federal Spike A and North Carolina TCLP Spike A is documented in the Chemical/Standard Receipt Logbook (Attachment V).

8.2.7 Mercury stock standard receipt and the preparation of Federal Spike B and NC Spike B are documented in the metals department.

9.0 CALIBRATION

9.1 Analytical balances are calibrated daily to +/- 0.01 g in accordance with the SOP for Balance Calibration, IEA Doc# QAS01002.NET.

9.2 pH meters are calibrated daily according to the following procedure:

9.2.1 Pour approximately 20 mL of each buffer, pH 4.0, 7.0 and 10.0 into 50 mL beakers.

9.2.2 Place the electrode in the pH 4.0 buffer solution and push the two-point cal. button. When the LED reading stabilizes, key in 25.00 and press the enter button. Rinse the electrode in de-ionized water and blot dry with laboratory wipe.

9.2.3 Place the electrode in the pH 10.0 buffer solution and wait for the LED reading to stabilize. Key in 25.00 and press the enter button.

9.2.4 Press the EFF button. The correlation coefficient should read 0.995 or greater. If the correlation coefficient is not 0.995 or better, recalibrate with fresh buffer solutions.

9.2.5 The electrode should be rinsed with DI water and left in the pH 7.0 buffer solution at all times. If the pH drifts out of the 6.9 - 7.1 range, the meter should be recalibrated.

10.0 QUALITY CONTROL

10.1 One leachate blank is prepared with each batch of samples. A batch consists of 10 samples not including the blank. The leachate blank consists of HPLC grade water and is treated identically to other samples being leached.

10.2 A matrix spike and matrix spike duplicate (MS/MSD) are prepared for every batch of 10 samples from one waste stream for inorganic leachates only.

10.3 A sufficient volume of sample is leached so that a matrix spike and a matrix spike duplicate can be prepared every 10 samples during preparation of the leachate for the appropriate organic analysis.

11.0 SAMPLE PREPARATION AND INSTRUMENTAL PROCEDURES

11.1 Preparation of the Millipore Filtration Unit

11.1.1 Disassemble the filtration unit into 3 pieces (cover, canister and base).

11.1.2 Rinse each piece with 10% Nitric Acid and rinse with de-ionized water.

11.1.3 Rinse the filter paper first with 10% Nitric Acid and then using de-ionized water. This must occur for metals leachates and is acceptable for organics leachates, but not required.

11.1.4 Place the filter paper onto the base of the filter unit and center it over the metal screen. Use glass filters for organic leachates and membrane filters for inorganic leachates.

11.1.5 Place the canister evenly over the filter paper.

11.1.6 Using 3 Teflon bolts, secure the cover/canister onto the base of the unit.

11.2 Preliminary Evaluations

11.2.1 If the sample is entirely solid with no appearance of liquidity evaluate the solid particle size. Particle size reduction is necessary if the sample is not capable of passing through a 9.5 mm standard sieve. Reduce the particle size, if necessary, by crushing or cutting the sample until it will pass through a 9.5 mm standard sieve. Proceed to Section 11.4.1.

11.2.2 If the sample is a liquid, sludge or has a muddy consistency, determine the percent solids by using the following method:

11.2.2.1 Shake the sample container or mix the sample mechanically as in the SOP for Soil Homogenization and Percent Solids, IEA Doc# QAS01400.NC to assure homogenization of the sample.

11.2.2.2 Weigh a minimum of 100 grams of the sample into a clean beaker. Record the weight in the wet weight column in the TCLP Extraction Logbook (Attachment I).

11.2.2.3 Allow the slurries to stand to permit the solid phase to settle.

11.2.2.4 Prepare an aluminum foil pan that is large enough to hold a filter (glass fiber for organic parameters and cellulose for metals). Weigh the pan and a filter. Record the weight in the TCLP Extraction Logbook.

11.2.2.5 Quantitatively transfer the sample from the beaker in Section 11.2.2.2 (liquid and solid phases) to the filtration apparatus assembled in Section 11.1.

11.2.2.6 Attach the nitrogen line to the unit and turn on the nitrogen flow. Allow the sample to filter. Note: If necessary, slowly increase pressure to 50 psi in 10 psi increments to complete filtration. Filtration is complete when all the liquid is removed or when the filtration process has completely stopped.

11.2.2.7 Dispose of the liquid filtrate.

11.2.2.8 Remove the filter containing the solid sample and place it on the foil pan. Weigh the pan, the filter and the sample. Determine the solid phase weight of the sample by subtracting the filter weight and the pan weight. Record the solid phase weight in the TCLP Extraction Logbook.

11.2.2.9 Determine the % solid as in Section 12.1 and record the % solid in the Dry Weight Determination Logbook. Reference the Dry Weight Determination Logbook in the TCLP Extraction Logbook.

11.2.2.10 If the % solids is equal to or greater than 0.5% complete the particle size reduction, as in Section 11.2.1, if necessary and determine the % dry solids as follows:

11.2.2.10.1 Transfer the pan + filter weight and the wet weight (determined in Sections 11.2.2.2 and 11.2.2.4) recorded in the TCLP Extraction Logbook to the appropriate data columns in the Dry Weight Determination Logbook (Attachment II).

11.2.2.10.2 Dry the sample and the filter in the pan (from Section 11.2.2.8) in an oven at 100 +/- 20°C overnight. Weigh the pan and its contents. Continue to dry and weigh the pan and its contents until 2 successive weighings yield the same value $\pm$ 1%. Record the final weighing in the Dry Weight Determination Logbook.

11.2.2.10.3 Calculate the % dry solids as in Section 12.2. Record this information in the TCLP Extraction Logbook.

11.3 Determination of the Appropriate Extraction Fluid

11.3.1 Weigh a 5 gram sample into a clean beaker. Add 96.5 mLs de-ionized water to the sample.

11.3.2 Place the beaker on a stir plate. Add a stir bar and slurry the sample for 5 minutes.

11.3.3 Check the pH level of the sample using a precalibrated pH meter. Record the pH under the initial pH column of the Extraction Fluid Section in the TCLP Extraction Logbook.

11.3.4 If the pH is < 5 use extraction fluid #1.

11.3.5 If the pH is ≥ 5, re-mix the sample to obtain optimum level by performing the following:

11.3.5.1 Add 3.5 mLs of 1N HCL to the sample. Cover the beaker with a watchglass and heat to 50°C for 10 minutes while stirring on a hot plate. Remove the sample from the hot plate and allow it to cool to room temperature. Check the pH. If the pH is <5 use extraction fluid #1. If pH is equal to or >5 use extraction fluid #2.

11.3.6 Record this final pH under the final pH of the Extraction Fluid Section in the TCLP Extraction Logbook.

11.4 Leaching the Sample

11.4.1 Solid Samples

11.4.1.1 If the waste is 100% solid, weigh at least 100 g into a 2 liter glass TCLP bottle (LCH_11, LCH_12, LCH_13) or a 2 liter plastic bottle (LCH_10). Record the sample weight in TCLP Extraction Logbook in the sample weight column (Record of Extraction section) in the TCLP Extraction Logbook.

11.4.1.2 If the sample will pass through the sieve as received, continue to Section 11.4.1.4.

11.4.1.3 If required, completed the particle size reduction (Section 11.2.1; crush, cut or grind the sample until it will pass through a 9.5 mm sieve).

11.4.1.4 Determine the volume of the appropriate extraction fluid (Section 11.3) by using the calculation in Section 12.3 and add this volume to the sample. Record the volume under No.1 or No.2 ,as appropriate, in the Extraction Fluid Section in the TCLP Extraction Logbook. Tighten the cap with the Teflon lining facing down towards the sample and place on a rotator for 18 $\pm$ 2 hours. Record the extraction start time and the room temperature in the TCLP Extraction Logbook. The room temperature should be 23°C +/- 2°C. If not, adjust the temperature of room to this setting.

11.4.1.5 After the sample has rotated for 18 $\pm$ 2 hours, record the extraction end time in the TCLP Extraction Logbook and pour the extracted sample from the TCLP bottle through a hot acid washed large funnel containing a new glass fiber filter and into the canister of a filtration unit which has been prepared as in Section 11.1. Filter as in Section 11.2.2.6. Collect the leachate in a 500 mL glass bottle (LCH_11, LCH_12, LCH_13) or a 500 mL plastic bottle (LCH_10). Record the filtration time and the extract (leachate) volume in the TCLP Extraction Logbook.

11.4.1.6 See Section 11.5 and/or Section 11.6 for preservation and storage instructions.

11.4.1.7 Cap the bottles with Teflon-lined lids if the leachate is to be analyzed for organic parameters. If inorganic parameters are to be determined proceed to Section 11.6.

11.4.1.8 Label each bottle with the sample identification number and extraction date; deliver bottles, along with the original extract chain-of-custody form, and a copy of the TCLP Extraction Log to the sample management department. Deliver copies of the extract COC to the various departments analyzing the samples. Keep one copy of the extract COC in a binder in the sample preparation department.

11.4.2 Liquid Samples with < 0.5% Solids

11.4.2.1 Assemble a filtration apparatus as in Section 11.1.

11.4.2.2 Weigh fresh another minimum of 100 g portion of sample and quantitatively transfer the sample to the canister of the filtration apparatus. Record the sample weight in the TCLP Extraction Logbook under sample weight in the Record of Extraction Section.

11.4.2.3 Attach the nitrogen line to the unit and turn on the nitrogen flow. Allow the sample to filter. Note: If necessary, slowly increase pressure to 50 psi in 10 psi increments to complete filtration.

11.4.2.4 Repeat steps 11.4.2.1 through 11.4.2.3 until the appropriate number of 500 mL bottles are filled or filtration is complete. [NOTE: The leachate volume (number of bottles) is dependant upon the number of fractions that will be analyzed from the sample. Generally, 1500 mL is needed for metals, 1000 mL for BNAs, 500 mL for herbicides and 500 mL for pesticides.] This filtrate is the sample leachate.

11.4.2.6 Cap the bottles with Teflon-lined lids if the leachate is to be analyzed for organic parameters. If inorganic parameters are to be determined proceed to Section 11.6.

11.4.2.7 Record the filtration time and the extract (leachate) volume in the TCLP Extraction Logbook.

11.4.2.8 See Section 11.5 and/or Section 11.6 for preservation and storage instructions.

11.4.2.9 Label each bottle with the sample identification number and extraction date; deliver bottles, along with the original extract chain-of-custody form, and a copy of the TCLP Extraction Log to the sample management department. Also deliver copies of the extract COC to the various departments analyzing the samples. Keep one copy of the extract COC in a binder in the sample preparation department.

11.4.3 Sludge or Multiphasic Samples (Samples with $\geq$ 0.5% Solids)

11.4.3.1 Assemble a filtration apparatus as in Section 11.1.

11.4.3.2 Use the percent solids information to determine the optimum sample size for filtration to achieve a minimum of 100 g sample dry weight (Section 12.4).

11.4.3.2.1 Weigh fresh another portion of sample to achieve the 100 g minimum weight as determined in Section 11.4.3.2. Record the sample weight in the TCLP Extraction Logbook (Record of Extraction section).

11.4.3.3 Allow slurries to stand to permit phase separation.

11.4.3.4 Quantitatively transfer the sample (liquid and solid phases) to the filter holder. Spread the waste evenly over the filter.

11.4.3.5 Attach the nitrogen line to the unit and turn on the nitrogen flow. Allow the sample to filter. Note: If necessary, slowly increase pressure to 50 psi in 10 psi increments to complete filtration.

11.4.3.6 Collect the pre-filtrate in a glass amber bottle. The material in the filter holder is the solid phase of the waste and the liquid phase is the pre-filtrate. Label the pre-filtrate bottle and store at 4°C.

11.4.3.7 Quantitatively transfer the solid phase of the sample and the filter to a 2 liter extractor bottle (glass for organics and plastic for inorganics).

11.4.3.8 Determine the volume of the appropriate extraction fluid (Section 11.3) based on the calculation in Section 12.3 and add this volume to the sample in the extractor bottle. Record the extraction fluid volume under No.1 or No.2, as appropriate, in the Extraction Fluid Section of the TCLP Extraction Logbook. Tighten the cap with the Teflon lining facing down towards sample and place on a rotator for 18 $\pm$ 2 hours. Record the extraction start time and the room temperature in the TCLP Extraction Logbook. The room temperature should be 23°C +/- 2°C. If not, adjust the temperature of room to this setting.

11.4.3.9 Record the extraction end time and separate the solid and the liquid phases of the sample by filtration as in Sections 11.1 and 11.4.3.4-11.4.3.5. Collect the liquid leachate in a 500 mL glass bottle if analysis is for organic parameters or a 500 mL plastic bottle for metals analysis. Record the time of filtration and the extract (leachate) volume in the TCLP Extraction Logbook.

11.4.3.10 If the pre-filtrate obtained from Section 11.4.3.6 and the leachate obtained in Section 11.4.3.9 are compatible or miscible, combine the pre-filtrate and the leachate and mix until well blended.

11.4.3.11 If the pre-filtrate and the leachate are heterogeneous **DO NOT MIX** them. Consider the two phases as two samples for login at sample management. File a PSR (Attachment III) with the appropriate project manager to notify the client and confirm the analysis requirement of multiple phases.

11.4.3.12 If the leachate is multi-phasic, record the appropriate data in the Multi-phase Logbook (Attachment IV) and reference this documentation in the TCLP Extraction Logbook. The top layer is appropriately identified and labeled as aqueous or organic. The bottom layer is identified similarly and documented. If additional layers are determined, see the laboratory supervisor for correct documentation and labeling.

11.4.3.13 See Section 11.5 and/or Section 11.6 for preservation and storage instructions.

11.4.3.14 Cap the bottles with Teflon-lined lids if the leachate is to be analyzed for organic parameters. If inorganic parameters are to be determined proceed to Section 11.6.

11.4.3.15 Label each bottle with the sample identification number and extraction date; deliver bottles, along with the original extract chain-of-custody form, and a copy of the TCLP Extraction Logbook to the sample management department. Also deliver copies of the extract COC to the various departments analyzing the samples. Keep one copy of the extract COC in a binder in sample preparation department.

11.5 Store all resultant leachates at 4°C. Preserve only those leachates that are for metals analysis.

11.6 Spiking and Preserving Metals Leachates

11.6.1 One sample per waste stream will be chosen for the matrix spike and matrix spike duplicate. Prepare the MS/MSD in the same manner as the other samples.

11.6.2 Use of the either the Federal spike or the NC Spike will be based on client request by test code.

11.6.3 If the Federal spike is necessary, add 5 mL of Federal spike A and 5.0 mL of Federal spike B to both the MS and the MSD. If the NC spike is necessary, add 10 mL of NC spike A and 5.0 mL of NC spike B to both the MS and the MSD. A witness will observe the addition of the appropriate spike(s) and will sign and date the TCLP Extraction Logbook.

11.6.3.1 Spike volumes are based on 500 mL sample volumes. If the volume extracted is less than 500 mL, adjust the spike volumes proportionately.

11.6.4 Check the extract (leachate) pH and record the pH. Preserve if the pH is greater than 2.

11.6.5 Preserve the samples by adding 4.0 mL - 5.0 mL of Nitric acid to the bottles after spiking. Check the pH with wide range pH paper. The pH should be less than 2. Record the pHs (both initial and final) on the TCLP Extraction Logbook (Record of Extraction section).

11.6.6 Cap the bottles. Label each bottle with the sample identification number and extraction date. Deliver the bottles, the original extract chain-of-custody and a copy of the TCLP Extraction Log to the sample management department. Make one copy each of the extract COC for the lab notebook and the metals department. Forward a copy of the extraction page to the metals department.

12.0 CALCULATIONS

12.1 Determination of Percent Solids

$$\% \text{ Solids} = \frac{\text{Weight of solid (solid phase weight, 11.2.2.8)}}{\text{Total weight of waste (wet weight, 11.2.2.2)}} \times 100$$

12.2 Determination of Percent Dry Solids

$$\% \text{ Dry Solids} = \frac{(\text{Wt. of dry waste (11.2.2.10.2)} + \text{filter wt.} + \text{pan wt.}) - (\text{tared filter wt.})}{\text{Initial wt. of waste (wet wt., 11.2.2.2)}} \times 100$$

12.3 Determination of Extraction Fluid Weight

$$\text{Wt. of extraction fluid} = \frac{20 \times \%\ \text{solid} \times \text{wt. of waste filtered (wet wt., 11.2.2.2 or wt. 100\% dry sample used)}}{100}$$

12.4 Determination of Dry Weight

Dry weight = % solid X wet weight (11.2.2.2)

13.0 ACCEPTANCE OF DATA

13.1 Extraction leachate blanks must not contain target analytes exceeding the PQL for that specified analyte.

14.0 REPORTING OF RESULTS

N/A

15.0 SUPPLEMENTAL DOCUMENTS

15.1 SOP for Soil Homogenization and Percent Solids, IEA Doc# QAS01400.NC

15.2 SOP for Balance Calibration, IEA Doc# QAS01002.NET.

16.0 REFERENCES

16.1 Test Methods for Evaluating Solid Waste, SW-846 Third Edition, September 1994 Revision, USEPA, Method 1311.

17.0 SUBSTANTIVE REVISIONS

17.1 Original issue (01/13/94)

17.2 Section 2.2: Test codes for screening omitted. Section 3.2 added. Section 5.4: Quarterly monitoring revised to monthly. Section 7.18: L corrected to mL. Sections 8.0 and 11.0 revised for clarification. Sections 9.2.1 - 9.2.5 and 10.2 added. Section 11.1.2.13.1 drying time revised to overnight. Section 11.3.3.3: pH corrected to > 5. Sections 11.6.2 and 11.6.4: room temperature revised to 23 °C. Section 16.1 revised to reference the current method. (06/21/96)

Attachment I

TCLP Extraction Log

IEA Sample #	Client Sample #	Percent Solid Phase				Extraction Fluid			Record of Extraction						
		Filter Wt. & Pan Wt. (g)	Wet Wt. (g)	Solid Phase Wt. (g)	% Dry Solid	Initial/ Final pH	No.1 Volume (mLs)	No.2 Volume (mLs)	Sample Wt. (g)	Ext. Start Time	Ext. Stop Time	Time of Filtration	Initial pH	Final pH	Extract Vol (mLs)

Batch #		Extraction Date		pH of Ext. Fluid No.1		Spike A Witness/Date		Comments:
Method	SW-846 Method 1311	Ext. Fluid No.1 Date		pH of Ext. Fluid No.2		Spike B Witness/Date		
Analyst		Ext. Fluid No.2 Date		Acid Lot #		Dry Weight Logbook #/Page		
Applicable SOP	SPS02001.NC	Room Temp.(°C)		Multiphasic Log Page #				

Secondary Review By:_____ **Date:**_____

Attachment II

IEA Corporation **Dry Weight Determination**

Analyst: _____
Date: _____

Sample ID	EPA ID	Pan Wt. + Filter (g)	Wet Wt.(g)	Pan + Dry Wt. + Filter (g)	% Solids

Secondary Review By: _____ **Date:** _____

Attachment III

PROJECT STATUS REPORT

Date_____

Project ID_____ Client_____

Lab Due Date_____ Project Due Date_____

Initiated By_____ Department_____

Parameter_____ Sample_____

☐ Late Notification

Status_____

Reason_____

Expected Completion_____

☐ Matrix Discrepancy ☐ Method Discrepancy

Problem_____

Resolution_____

☐ Protocol Exceeded

Date Sampled_____ Date Protocol Expired_____

Date Analyzed_____ Reason_____

CLIENT NOTIFICATION

_____

_____

_____

_____

_____

_____

Attachment IV

Multiphasic Sample Log

IEA Sample #	Client Sample #	# of Phases	Vol. Aqueous Phase		Vol. Organic Phase	
			Initial	Final	Initial	Final

Batch #		Extraction Date:	
Method		TCLP Log Page #:	
Analyst		Comments:	
Applicable SOP			

Secondary Review By:____________________ Date:______________

Attachment V

Sample Preparation Lab Chemical/Standard Receipt Log

DATE	CHEMICAL/STANDARD NAME	LOT #	VENDOR	QTY	VOLUME/MASS	EXPIRATION DATE	ANALYST

Secondary Review:_____ By:_____

23. IEA SOP FOR HEXAVALENT CHROMIUM IN SOIL

1.0 APPROVALS

The signature of the following individuals indicates that this Standard Operation Procedure (SOP) is complete and meets the requirements specified in Corporate Document # QAS00200.NET. In addition, it also signifies that the content meets the specifications of the referenced "Test Code".

Laboratory Director

Quality Assurance Manager

UNCONTROLLED COPY
PROPRIETARY DOCUMENT

2.0 SCOPE AND APPLICATION

2.1 This method is applicable to the measurement of hexavalent chromium in solid wastes.

2.2 This method is applicable to Test Code CR6_02 for hexavalent chromium (Chromium VI) analysis in alkaline digestates of solid wastes.

3.0 SUMMARY OF METHOD

3.1 This method uses an alkaline digestion of waste samples to solubilize both water-insoluble and water-soluble hexavalent chromium compounds. The resulting dissolved hexavalent chromium reacts with diphenylcarbazide at a pH of 0.7 to 1.3 to form a red-violet, water soluble compound which absorbs light at 540 nm.

3.2 The analytical methodology is based on EPA SW-846 Method 7196A, while the digestion procedure is based on SW-846 Method 3060A.

3.3 This method is based on EPA SW-846 Method 7196A chemistry, modified for analysis using a Lachat QuickChem Flow Injection Analyzer. The digestion procedure deviates from SW-846 Method 3060A in the following ways:

3.3.1 The spiking solution is the same as the stock hexavalent chromium (Section 8.2.1.1, 50 mg/L) instead of 1000 mg/L.

3.3.2 The spiking level is fixed at 10 mg/kg instead of twice the concentration of hexavalent chromium found in the unspiked aliquot of sample since the concentration is unknown at the time of sample preparation.

3.3.3 Preparation Blanks must be less than the Practical Quantitation Limit (PQL) instead of less than the MDL or 1/10 of the lowest sample concentration.

3.3.4 The temperature during sample preparation is monitored by immersing the hotplate temperature probe in a sample, not a temperature blank.

4.0 INTERFERENCES

4.1 The chromium reaction with diphenylcarbazide is usually free from interferences, however, certain substances may interfere if the chromium concentration is relatively low. Hexavalent molybdenum and mercury salts also react to form color with the reagent; however, the red-violet intensities produced are much lower than those for chromium at the specified pH. Concentrations of up to 200 mg/L of molybdenum and mercury can be tolerated. Vanadium interferes strongly, but concentrations up to 10 times that of chromium will not cause trouble.

5.0 SAFETY

5.1 Diphenylcarbazide has no known acute toxicity, although irritation may result from exposure to the skin, respiratory tract and mucous membranes. Consult Material Safety Data Sheets (MSDS) for precautions and proper handling.

5.2 Hexavalent Chromium is a known carcinogen. Avoid direct contact with both the solid powder and solutions of potassium dichromate ($K_2Cr_2O_7$).

6.0 SAMPLE CONTAINERS, COLLECTION AND PRESERVATION

6.1 Sample Containers: Containers may be either glass or plastic, thoroughly rinsed and dried before use and are not to be reused.

6.2 Sample Collection: Samples are collected into plastic or glass containers. Samples are cooled and maintained at 4° C and shipped to the laboratory for analysis.

6.3 Sample Preservation: Sample preservation is not necessary. Samples should be refrigerated at 4° C until analysis.

6.4 Holding Times: The holding time for hexavalent chromium is 24 hours from sampling and is applied to the soil matrix as well as the water matrix. If the holding time is exceeded, a Project Status Report (PSR) is completed; informing the project manager that the holding time has been exceeded and that a resampling should be scheduled.

7.0 APPARATUS AND MATERIALS

7.1 Lachat QuikChem Flow Injection Analyzer.

7.2 Programmable Hotplate, with temperature probe.

7.3 250 mL beakers.

7.4 100 mL, 500, 1000 volumetric flasks.

7.5 Short stem funnels.

7.6 pH meter.

7.7 50 mL graduated cylinder.

7.8 Analytical Balance, 0.1 mg sensitivity.

7.9 Watch glasses.

7.10 Filtration apparatus.

7.11 0.45 μ membrane filters.

8.0 REAGENTS AND STANDARD PREPARATION

8.1 Reagents

8.1.1 Reagent water, 16.6 megohm-cm or greater, deionized.

8.1.2 Diphenylcarbizide Color Reagent: In a 1.0 liter volumetric flask, dissolve 0.40 grams s-diphenylcarbazide ($C_{13}H_{14}N_4O$) in 200 mL 99% isopropanol. Add 720 mL reagent water, then 80 mL concentrated sulfuric acid and dilute to volume.

8.1.3 Digestion solution: Dissolve 20.0 grams NaOH and 30.0 grams sodium carbonate (Na_2CO_3) in reagent water and dilute to 1000 mL in a volumetric flask. Store the solution in a tightly capped polyethylene bottle and prepare fresh monthly. The pH of the solution must be checked before usage. The pH must be $\geq$ 11.5 or the solution should be discarded.

8.1.4 Diphenylcarbizide-free Color Reagent: In a 1.0 liter volumetric flask, add 200 mL 99% isopropanol. Add 720 mL reagent water, followed by 80 mL concentrated sulfuric acid and dilute to volume.

8.1.5 Magnesium Chloride: $MgCl_2$ (anhydrous), analytical reagent grade.

8.1.6 1.0 M Phosphate Buffer at pH 7 Preparation:

8.1.6.1 K_2HPO_4, analytical reagent grade.

8.1.6.2 KH_2PO_4, analytical reagent grade.

8.1.6.3 Dissolve 87.09 g K_2HPO_4 and 68.04 g KH_2PO_4 into 700 mL of reagent water. Transfer the solution to a 1 liter volumetric flask and dilute to volume.

8.1.7 Lead Chromate, analytical reagent grade.

8.1.8 Reagent receipt and preparation is documented in the Reagent Preparation and Receiving Logbook (Attachment I).

8.2 Standard Preparation

8.2.1 Calibration Standard Preparation

8.2.1.1 Stock Potassium Dichromate solution (50 mg/L as Cr^{6+}): Dissolve 141.4 mg dried potassium dichromate ($K_2Cr_2O_7$) in deionized water and dilute to 1.0 L.

8.2.1.2 Intermediate Potassium Dichromate solution (4.0 mg/L as Cr^{6+}): Dilute 8.0 mL of the stock solution (Section 8.2.1.1) to 100 mL.

8.2.1.3 Prepare the calibration standards by serial dilution using the following table as a reference.

Standard Concentration	Dilution Std., Volume	Final Volume (mL)
0.40 mg/L	4.0 mg/L, 10.0 mL	100
0.20 mg/L	4.0 mg/L, 5.0 mL	100
0.10 mg/L	0.40 mg/L, 25.0 mL	100
0.04 mg/L	0.40 mg/L, 10.0 mL	100
0.02 mg/L	0.40 mg/L, 5.0 mL	100
0.01 mg/L	0.10 mg/L, 10.0 mL	100

8.2.2 Initial and Continuing Calibration Verification Standard Preparation

8.2.2.1 Initial and Continuing Calibration Verification Standard (ICV, CCV, respectively), 0.25 mg/L as Cr^{6+}: Dilute 10.0 mL of 12.5 mg/L Hach Standard (Purchased from Hach Co., Loveland Co. Catalog # 14256-10) to 500 mL.

8.2.3 All standard receipt and preparation is documented in the Standards Receipt and Preparation Logbook (Attachment II).

9.0 CALIBRATION

9.1 The working range of the calibration is 0.01 to 0.40 mg/L as Cr^{6+}.

9.2 Instrument Startup:

9.2.1 Switch the power strip located behind the system computer to the "on" position. When the computer has completed the self-diagnostics, the CPU monitor will display the main menu with the METHODS category illuminated.

9.2.2 Install the Hexavalent chromium chemistry manifold onto the Lachat module, making proper connections to reagent lines as shown in the diagram in the Lachat method manual.

9.2.3 Place all reagent lines into proper reagent containers and adjust the tension levers on the peristaltic pump until the first "click" is heard. While the system is pumping reagents through the manifold, proceed with METHOD DOWNLOADING.

9.3 Method Downloading

9.3.1 To download the chemistry program to the system computer, observe the "METHODS" category illuminated at the CPU monitor, press [ENTER].

9.3.2 The monitor will display "Download Method" and "Method Definition". Using the arrow keys, highlight "Download Method" and press [ENTER].

9.3.3 A menu will appear listing pre-programmed methods in the system. Move the cursor until "HEXAVALENT CHROMIUM" is highlighted, then press ENTER.

9.3.4 The method will now start downloading the chemistry method to the computer.

9.4 Calibration

9.4.1 To set up the standard curve on the autosampler, obtain a vial for each standard beginning with the 0.40 mg/L standard. Fill the vial and place it in the standards rack in the front left spot. Continue with the other standards, in order of decreasing concentration.

9.4.2 The computer monitor will display "Edit". Using the arrow keys, highlight "Submit" and "Calibrate Now" then hit [ENTER]. The screen will give the prompt "Should a calibration be started now?", press "Y" for yes.

9.4.3 The Lachat will begin calibrating. When the calibration is complete the system will indicate whether the pass/fail criteria has been met by displaying "calibration failed!" or "calibration approved".

Note: Analysis cannot proceed until the calibration is approved.

9.5 Acceptance criteria for the calibration requires the linear regression correlation coefficient of the calibration standards to be greater than 0.9975.

9.6 Calculations: Analyte concentrations in samples are calculated by Lachat software which is based on linear regression.

10.0 QUALITY CONTROL

10.1 Method detection limits are performed in accordance with the SOP for Conducting MDL Studies, IEA Doc# QAS02000.NET which is based on the 40 CFR, Part 136, Appendix B definition of method detection limit.

10.2 The practical quantitation limit for this method is 0.5 mg/kg.

10.3 The Lachat software performs a diagnostics check on the autosampler, switching valves and the detector after the initial powering-up of the system computer.

10.4 Soluble Pre-Digestion Matrix Spikes: Pipet 2.0 mL stock potassium dichromate solution (Section 8.2.1.1, 50 mg/L) to 2.5 grams of sample, and prepare as in Section 11.2. This comprises a matrix spike of 40 mg/kg at analysis. Matrix spikes are analyzed with every 20 samples or less, per batch.

10.5 Insoluble Pre-Digestion Matrix Spikes: Add 10 - 20 mg of lead chromate (Section 8.1.7) to 2.5 grams of sample and prepare as in Section 11.2.

10.6 Post-Digestion Matrix Spike: Pipet 2.0 mL stock potassium dichromate solution (Section 8.2.1.1, 50 mg/L) to a previously digested sample. This comprises a matrix spike of 40 mg/kg at analysis. Matrix spikes are analyzed with every 20 samples or less, per batch.

10.7 Initial and Continuing Calibration Verification standards (ICV and CCV, respectively), (Section 8.2.2) represent QC check samples for this method. ICVs are analyzed at the onset of analysis and CCVs are analyzed every 10 samples.

10.8 Initial and Continuing Calibration Blanks (ICB and CCB, respectively) consist of digestion solution (Section 8.1.3). ICBs are analyzed at the onset of analysis and CCBs are analyzed every 10 samples.

10.9 Preparation Blanks consist of digestion solution (Section 8.1.3) that is prepared as in Section 11.2. Preparation Blanks are analyzed with every 20 samples or less, per batch.

10.10 Duplicate samples are analyzed with every 20 samples or less, per batch.

10.11 Surrogates are not applicable to this method.

10.12 Internal standards are not applicable to this method.

10.13 The Laboratory Control Sample (LCS) is prepared by spiking 50 mL of the digestion solution (Section 8.1.3) with 0.4 mL of ICV Calibration Standard (Section 8.2.2.1). LCSs are analyzed with every 20 samples or less, per batch.

10.14 QC SCHEDULES:

ACTIVITY:	FREQUENCY:
ICV/ICB	Onset of every analysis
Preparation Blank	Every 20 samples, per batch
Duplicate sample	Every 20 samples, per batch
Soluble Pre-Digestion Matrix Spike	Every 20 samples, per batch
Insoluble Pre-Digestion Matrix Spike	Every 20 samples, per batch
Post Digestion Matrix Spike	Every 20 samples, per batch
QC Check Samples (CCV/CCB)	Every 10 samples, per analysis
Laboratory Control Sample	Every 20 samples, per batch

DEFINITIONS:
Batch: Every 20 samples or less leached, digested, or distilled, per day.
Analysis: Each calibration, each day.

11.0 SAMPLE PREPARATION AND INSTRUMENTAL PROCEDURES

11.1 Instrumental Conditions

11.1.1 See Section 9.0 for specific procedures for instrument startup and calibration.

11.2 Sample Preparation

11.2.1 The methodology for the preparation of soils for hexavalent chromium analysis is based on an Alkaline Digestion method, SW-846 Method 3060A. This procedure is reduced by a factor of 10 for use with the Lachat Flow Injection Analyzer.

11.2.2 Homogenize the sample in accordance with the SOP for Homogenization and Percent Solids, IEA Doc# QAS01400.NC.

11.2.3 Place 2.5 grams of sample into a 250 mL beaker and record the weight in the Lachat Runlog (Attachment III).

11.2.4 Add 50 mL of digestion solution (Section 8.1.3) to each sample. Add 392.18 mg of $MgCl_2$ (Section 8.1.5) and 0.5 mL of 1.0 M phosphate buffer (Section 8.1.6). Stir with a stir bar for at least 5 minutes unheated. Cover the beaker with a watch glass and continue to stir until the 60 minute digestion is complete (Section 11.2.9).

11.2.5 Press the [ON/OFF] key on the programmable hotplate to turn the power on and set the stirrer speed to 250 rpm by pressing [ENT], [3], [2][5][0], [ENT].

11.2.6 Place the temperature probe in a sample beaker as prepared in Section 11.2.4 making sure that the tip of the probe is completely submersed.

11.2.7 Set the target probe temperature to 95 degrees C by pressing [ENT], [2], [9][5], [ENT]. At this point the display will flash the current temperature and the target temperature repeatedly.

11.2.8 When the current temperature reaches 92 degrees C set the timer to one hour. Do not allow the sample to go to dryness as hexavalent chromium may be lost.

11.2.9 When one hour has expired, stop the heater by pressing [ENT], [2], [2][0], [ENT].

11.2.10 When the current temperature reaches 50 degrees C, turn the power to the hotplate off.

11.2.11 Up to nine sample may be digested on the programmable hotplate at one time, using one sample with the temperature probe.

11.2.12 Gradually cool each solution to room temperature and transfer it quantitatively to the filtration apparatus with reagent water rinses and filter through a 0.45 μ membrane filter.

11.2.13 Rinse the inside of the filter flask and filter pad with reagent water and transfer the filtrate and the rinses to a clean 250 mL beaker.

11.2.14 Place a stir bar in the filtrate beaker and, with constant stirring, slowly add nitric acid dropwise until the pH is 7.5 $\pm$ 0.5. Monitor the pH with a pH meter. If the pH drops below 7.0 discard the solution and digest again.

11.2.15 Remove and rinse the stir bar. Collect the rinsate in the filtrate beaker and transfer the contents of the beaker to a 100 mL volumetric flask and dilute to volume in reagent water. The sample is now ready for analysis.

11.2.16 Repeat Sections 11.2.1 - 11.2.15 until all samples including QC samples have been prepared.

11.3 Sample Analysis

11.3.1 All samples are analyzed initially at a 5x dilution.

11.3.2 After obtaining an approved calibration (Section 9.0), press the [Esc] key until the "Sample Tray" program menu appears. Use the arrow keys to highlight "Edit" then press [ENTER].

11.3.3 A sub menu will appear with "Identification" highlighted. Press [ENTER].

11.3.4 Proceed to enter the sample ID's in the exact order that they are arranged in the sample tray. After the last sample has been entered, type two periods " .. " as the next sample entry; this indicates the end of the tray.

11.3.5 Press the [ESC] key to back out to the "Sample tray" menu once again. Using the arrow keys, highlight "SUBMIT" and then press [ENTER].

11.3.6 The display will prompt the operator to submit the tray for analysis.

Note: Real time display of sample results may be accessed by pressing the [CTRL], [SHIFT], and [1] keys simultaneously. To return to the program menu, simply press [CTRL], [SHIFT], and [2] simultaneously. DO NOT use the arrow keys to scroll the screen in this mode; it will cause the computer to lock up. This will require the system to be shut down and the method downloading and calibration to be executed again.

11.4 After all analyses are complete, switch the color reagent line to the diphenylcarbizide free reagent (Section 8.1.4) and rinse for 5 minutes.

11.5 The analysis run is then resubmitted exactly as it was performed previously. The absorbance from this run is subtracted from the color reagent run to correct for nonspecific absorbance for all data points. This should be noted on the final report to the client. Both corrected and uncorrected data should be maintained on file.

11.6 All analyses including calibration, samples, and QC Samples are documented in the Lachat Runlog (Attachment III).

12.0 CALCULATIONS

12.1 ICV,and CCV Recovery:

$$\frac{\text{Actual mg/L}}{\text{Theor. mg/L}} \times 100 = \%\ \text{Recovery}$$

12.2 Spike Recovery:

$$\frac{(\text{Spiked sample} + \text{sample conc., mg/kg}) - (\text{sample conc., mg/kg})}{\text{Spike Added (mg/kg)}} \times 100 = \%\ \text{Recovery}$$

12.3 Relative Percent Difference:

$$\left[\frac{(\text{Sample 1} - \text{Sample 2})}{\text{avg., sample 1 \& 2}}\right] \times 100 = \%\ \text{RPD}$$

13.0 ACCEPTANCE CRITERIA

13.1	Blanks	< 0.5 mg/kg (PQL)
	ICV, CCV	85% - 115% Recovery
	Pre-digestion MSs	75% - 125% Recovery
	Post-Digestion MS	85% - 115% Recovery
	Duplicates	$\pm$ 20% RPD or $\pm$ MDL if sample conc. < 5 x MDL
	LCS	85% - 115% Recovery

14.0 REPORTING OF RESULTS

14.1 Practical Quantitation Limits: 0.5 mg/kg as Cr^{6+}
Units of Measure: mg/kg
Significant Figures: 2 figures
Data Reporting:
Below quantitation limit: BQL

QC information included in report:
Level 1 QC: Blank values, sampling COC.
Level 2 QC: Blank values, Duplicate data (%RPD), sampling COC.
Level 3 QC: Blank values, Duplicate data (%RPD), sampling and internal COC, matrix spike recoveries, specific to client's samples.

15.0 SUPPLEMENTAL DOCUMENTS

15.1 Corporate SOP for Conducting MDL Studies, IEA Doc# QAS02000.NET.

15.2 SOP for Soil Homogenization and Percent Solids, IEA Doc#QAS01400.NC.

16.0 REFERENCES

16.1 Test Methods for Evaluating Solid Wastes, 3rd Edition, Method 7196A, July 1992 Revision.

16.2 Test Methods for Evaluating Solid Wastes, 3rd Edition, Method 3060A, January 1995 Revision.

16.3 Lachat QuikChem Flow Injection Analyzer Method #10-124-13-1-A.

17.0 SUBSTANTIVE REVISIONS

17.1 Section 8.1.1 revised for correct units. Sections 8.1.7 and 11.4 added. COCs added to Level 2 and Level 3 report QC. Sections 8.2 and 11.5 added. Logbook reference added to Sections 9.1 and 11.2.1. SOP reference added in Section 10.1.

17.2 Sections 3.3.3, 7.8-7.12 and 11.3.1 added. Sections 3.2, 3.3 and 16.1 revised to the current reference method.Section 8.0 revised for clarification including the addition of sections 8.1.5, 8.1.6 and 8.1.7. Section 10.0 revised for clarification including the addition of Section 10.4-10.6 and 10.13-10.14. Section 11.0 revised for clarification including the addition of Sections 11.2.2 and 11.2.5-11.2.16. Section 11.2.4 revised to 50 mL of digestion solution, the addition of magnesium chloride and the addition of phosphate buffer for method compliance. Sections 13.1 and 14.1 revised to reflect the correct PQL. Pre-digestion spike, post-digestion spike and LCS acceptance criteria added in Section 13.1. (05/10/96)

24. IEA SOP FOR HEXAVALENT CHROMIUM IN WATER

1.0 APPROVALS

The signatures of the following individuals indicate that this SOP is complete and meets the requirements specified in corporate document # QAS00200.NET. In addition, it signifies that the content meets the specifications of the referenced "Test Code".

Laboratory Director

Quality Assurance Manager

UNCONTROLLED COPY
PROPRIETARY DOCUMENT

2.0 SCOPE AND APPLICATION

2.1 This method is applicable to the measurement of hexavalent chromium in EPTOX, ground water, and some domestic and industrial wastes.

2.2 This method is applicable to Test Code CR6_01 for hexavalent chromium (Chromium VI) analysis in water.

3.0 SUMMARY OF METHOD

3.1 Dissolved hexavalent chromium reacts with diphenylcarbazide at a pH of 0.7 to 1.3 to form a red-violet, water soluble compound which absorbs light at 540 nm.

3.2 This method is based on EPA SW-846 Method 7196.

3.3 This method is based on EPA SW-846 Method 7196 chemistry, modified for analysis using a Lachat QuickChem Flow Injection Analyzer.

4.0 INTERFERENCES

4.1 The chromium reaction with diphenylcarbazide is usually free from interferences. However, certain substances may interfere if the chromium concentration is relatively low. Hexavalent molybdenum and mercury salts also react to form color with the reagent; however, the red-violet intensities produced are much lower than those for chromium at the specified pH. Concentrations of up to 200 mg/L of molybdenum and mercury can be tolerated. Vanadium interferes strongly, but concentrations up to 10 times that of chromium will not cause trouble.

4.2 Samples with significant background absorbance due to sample color can cause false positives in the sample. This interference is corrected by background absorbance subtraction.

5.0 SAFETY

5.1 Diphenylcarbazide has no known acute toxicity, although irritation may result from exposure to the skin, respiratory tract and mucous membranes. Consult Material Safety Data Sheets (MSDS) for precautions and proper handling.

5.2 Hexavalent Chromium is a known carcinogen. Avoid direct contact with both the solid powder and solutions of potassium dichromate ($K_2Cr_2O_7$).

6.0 SAMPLE CONTAINERS, COLLECTION AND PRESERVATION

6.1 Sample Containers: Containers may be of glass or plastic, thoroughly rinsed and dried before use and are not to be reused.

6.2 Sample Collection: Samples are collected into plastic or glass containers. Samples are cooled and maintained at 4°C and shipped to the laboratory for analysis.

6.3 Sample Preservation: Sample preservation is not necessary. Samples should be refrigerated at 4°C until analysis.

6.4 Holding Times: The holding time for hexavalent chromium in water is 24 hours from sampling. If the holding time is exceeded, a Project Status Report (PSR) is completed; informing the project manager that the holding time has been exceeded and that a resampling should be scheduled.

7.0 APPARATUS AND MATERIALS

7.1 APPARATUS:

7.1.1 Lachat QuikChem Flow Injection Analyzer.

7.2 pH paper - narrow range strips

8.0 REAGENTS AND STANDARD PREPARATION

8.1 Reagents:

8.1.1 Reagent water, 16.6 megohm-cm, deionized

8.1.2 Stock Potassium dichromate solution: Dissolve 141.4 mg dried potassium dichromate ($K_2Cr_2O_7$) in deionized water and dilute to 1.0 L. (=50 mg/L as Cr^{6+})

8.1.3 Intermediate Potassium dichromate solution: Dilute 8.0 mL of the stock solution (8.1.2) to 100 mL (=4.0 mg/L as Cr^{6+}).

8.1.4 Initial and Continuing Calibration Verification Standard (ICV, CCV, respectively): Dilute 12.5 mg/L Hach Standard (Purchased from Hach Co., Loveland CO. Catalog # 14256-10), 10.0 mL to 500 mL (=0.25 mg/Las Cr^{6+}).

8.1.5 Diphenylcarbizide Color Reagent: In a 1.0 liter volumetric flask, dissolve 0.40 grams s-diphenylcarbazide ($C_{13}H_{14}N_4O$) in 200 mL 99% isopropanol. Add 720 mL water, then 80 mL concentrated sulfuric acid and dilute to the mark.

8.1.6 Color reagent without Diphenylcarbizide: In a 1.0 liter volumetric flask, add 200 mL 99% isopropanol. Add 720 mL water, followed by 80 mL concentrated sulfuric acid and dilute to the mark.

8.2 Reagent receipt and preparation is documented in the Reagent Preparation and Receiving Logbook (Attachment I).

9.0 CALIBRATION

9.1 Calibration Standards: Prepare a series of standards by serial dilution using the following table as a reference. All standard receipt and preparation is documented in the Standards Receipt and Preparation Logbook (Attachment II).

Standard Concentration	Dilution Std., Volume	Final Volume (mL)
0.40 mg/L	4.0 mg/L, 10.0 mL	100
0.20 mg/L	4.0 mg/L, 5.0 mL	100
0.10 mg/L	0.40 mg/L, 25.0 mL	100
0.04 mg/L	0.40 mg/L, 10.0 mL	100
0.02 mg/L	0.40 mg/L, 5.0 mL	100
0.01 mg/L	0.10 mg/L, 10.0 mL	100

9.2 The working range of the calibration is 0.01 to 0.40 mg/L as Cr^{6+}.

9.3 Instrument Calibration:

9.3.1 Instrument startup: Switch the power strip located behind the system computer to the "on" position. When the computer has completed the self-diagnostics, the CPU monitor will display the main menu with the METHODS category illuminated. Install the Hexavalent chromium chemistry manifold onto the Lachat module, making proper connections to reagent lines as shown in the diagram in the Lachat method manual. Place all reagent lines into proper reagent containers and adjust the tension levers on the peristaltic pump until the first "click" is heard. While the system is pumping reagents through the manifold, proceed with METHOD DOWNLOADING.

9.3.2 Method Downloading: To download the chemistry program to the system computer, observe the "METHODS" category illuminated at the CPU monitor, press [ENTER]. The monitor will display "Download Method" and "Method Definition", using the arrow keys, highlight "Download Method" and hit [ENTER]. A menu will appear listing pre-programmed methods in the system. Move the cursor until "HEXAVALENT CHROMIUM" is highlighted, then hit ENTER. The method will now start downloading the chemistry method to the computer.

9.3.3 Calibration: To set up the standard Curve on the autosampler, obtain a vial for each Std. starting with the 0.40 mg/L Standard. Fill the vial and place it in the standards rack in the front left spot. Continue with the other standards, in order of decreasing concentration. The computer monitor will display "Edit". Using the arrow keys, highlight "Submit" and "Calibrate Now" then hit [ENTER]. The screen will give the prompt "Should a calibration be started now?", hit "Y" for yes. The Lachat will begin calibrating. When the calibration is complete the system will indicate whether the pass/fail criteria has been met by displaying "calibration failed!" or "calibration approved". Note: Analysis cannot proceed until the calibration is approved.

9.4 Internal standards are not applicable to this method.

9.5 Acceptance criteria for the calibration requires the linear least-squares regression coefficient of the calibration standards to be greater 0.9975.

9.6 Calculations: Analyte concentrations in samples are calculated by Lachat software and are based on the linear least squares fit of the calibration curve.

10.0 QUALITY CONTROL

10.1 Method detection limits are performed in accordance with the SOP for Conducting MDL Studies, IEA Doc# QAS02000.NET which is based on the 40 CFR, Part 136, Appendix B definition of method detection limit. A minimum of seven replicate standards prepared by referenced methods including all processing steps including distillation and digestion, where appropriate. Method detection limits are calculated by multiplying the standard deviation of all replicate results by student t values as listed in 40 CFR.

10.2 Practical quantitation limits for this method are the same as the method detection limit.

10.3 The Lachat software performs a diagnostics check on the autosampler, switching valves and the detector after the initial powering-up of the system computer.

10.4 Matrix spikes consist of spiking 0.050 mL of the 50 mg/L stock solution (sec 8.1.2) into 8.0 mL of sample and mixing. This comprises a matrix spike of 0.31 mg/L.

10.5 QC Check Samples consist of Initial and Continuing Calibration Verification standards (ICV and CCV, respectively), (Sec 8.1.4.)

10.6 Surrogates are not applicable to this method.

10.7 QC SCHEDULES:

ACTIVITY:	FREQUENCY:
Initial Calibration Verification	Onset of every analysis
Initial Calibration Blank	Onset of every analysis
Duplicate sample	Every 20 samples, per batch
Matrix Spike	Every 20 samples, per batch
Continuing Calibration Verification	Every 10 samples, per analysis
Continuing Calibration Blank	Every 10 samples, per analysis

DEFINITIONS:
Batch: Every 20 samples leached, digested, or distilled, per day.
Analysis: Each calibration, each day.

11.0 SAMPLE PREPARATION AND INSTRUMENTAL PROCEDURES

11.1 Instrumental Conditions: See section 9.3 for specific procedures for instrument startup and calibration.

11.2 Sample Preparation: Sample preparation is minimal since distillation or digestion is not required. Highly turbid samples, or samples with suspended matter may interfere and should be centrifuged or filtered with 0.45 micron filter paper prior to analysis.

11.3 Sample analysis: After obtaining an approved calibration (sec. 9.3), press the [Esc] key until the "Sample Tray" program menu appears. Use the arrow keys to highlight "Edit" then hit [ENTER]. A sub menu will appear with "Identification" highlighted. Hit [ENTER]. Proceed to enter the sample ID's in the exact order that they are arranged in the sample tray. After the last sample has been entered, type two periods " .. " as the next sample entry; this indicates the end of the tray. Press the [ESC] key to back out to the "Sample tray" menu once again. Using the arrow keys, highlight "SUBMIT" and then hit [ENTER]. The display will prompt the operator to submit the tray for analysis. Prior to loading samples, check that the pH is 7 with narrow range pH paper. Use dilute H_2SO_4 or NaOH, as necessary, to adjust the pH to 7.

Note: Real time display of sample results may be accessed by pressing the [CTRL], [SHIFT], and [1] keys simultaneously. To return to the program menu, simply press [CTRL], [SHIFT], and [2] simultaneously. DO NOT use the arrow keys to scroll the screen in this mode; it will cause the computer to lock up. This will require the system to be shut down and the method downloading and calibration to be executed again.

11.4 After all analyses are complete, the color reagent line is switched to the Diphenylcarbizide free reagent (Section 8.1.6) and allowed to rinse for 5 minutes. The analysis run is then resubmitted exactly as it was performed previously. The absorbance from this run is subtracted from the color reagent run to correct for nonspecific absorbance for all data points. This should be noted on the final report to the client. Both corrected and uncorrected data should be maintained on file.

11.5 All analyses including calibration, samples, and QC Samples are documented in the Lachat Runlog (Attachment III).

12.0 CALCULATIONS

12.1 ICV,and CCV Recovery:

$$\frac{\text{Actual mg/L}}{\text{Theor. mg/L}} \times 100 = \%\ \text{Recovery}$$

12.2 Spike Recovery:

$$\frac{(\text{Spiked sample} + \text{sample conc., mg/L}) - (\text{sample conc., mg/L})}{\text{Spike Added (mg/L)}} \times 100 = \%\ \text{Recovery}$$

12.3 Method blank, Initial Calibration, and Continuing Calibration blanks: Not to exceed 0.01 mg/L (MDL).

12.4 Relative Percent Difference:

$$\left[\frac{(\text{Sample 1} - \text{Sample 2})}{\text{avg., sample 1 \& 2}}\right] \times 100 = \%\ \text{RPD}$$

13.0 ACCEPTANCE CRITERIA

ICB, CCB	< 0.01 mg/L (MDL)
ICV, CCV	85% - 115% Recovery
Matrix spike	75% - 125% Recovery
Duplicates	$\pm$ 20 % or $\pm$ 0.01 mg/L (MDL),if sample conc. < 5 x MDL

14.0 REPORTING OF RESULTS

Practical Quantitation Limits: Same as MDL.
Units of Measure: mg/L
Significant Figures: 2 figures
Data Reporting: Below quantitation limit: BQL
QC information included in report:

Level 1 QC: Blank values.
Level 2 QC: Blank values, Duplicate data (%RPD), sampling COC.
Level 3 QC: Blank values, Duplicate data (%RPD), Matrix spike recoveries, specific to client's samples, sampling and internal COC.

15.0 SUPPLEMENTAL DOCUMENTS

15.1 Corporate SOP for Conducting MDL Studies, IEA Doc# QAS02000.NET.

16.0 REFERENCES

16.1 Test Methods for Evaluating Solid Wastes, 3rd Edition, Method 7196, July 1992.

16.2 Lachat Method #10-124-13-1-A.

17.0 SUBSTANTIVE REVISIONS

17.1 Section 8.1.1 corrected to units. Section 8.1.6 added. pH check added to section 11.3. Section 11.4 revised to reference reagent 8.1.6. Level 2 and 3 QC revised to include COCs. Sections 8.2 and 11.5 added. Logbook reference added in Section 9.1. SOP reference added in Sections 10.1 and 15.1. Method reference revised to appropriate format in Section 16.1.

25. IEA SOP FOR TOC IN SOIL METHOD 415.1

1.0 APPROVALS

The signature of the following individuals indicates that this SOP is complete and meets the requirements specified in corporate Document # QAS00200.NET. In addition, it also signifies that the content meets the specifications of the referenced "Test Code".

Laboratory Director

Quality Assurance Manager

UNCONTROLLED COPY
PROPRIETARY DOCUMENT

2.0 SCOPE AND APPLICATION

2.1 Organic carbon is oxidized to carbon dioxide, CO_2, by persulfate in the presence of ultraviolet light and measured directly by a nondispersive infrared detector. The instrument used is a Dohrmann DC - 180 analyzer or equivalent.

2.2 This method is applicable to water samples only.

2.3 This method is applicable to Test Code TOC_01.

3.0 SUMMARY OF METHOD

3.1 Water samples are acidified with phosphoric acid to remove inorganic and purgeable carbon. The remaining organic carbon is then oxidized to carbon dioxide, CO_2, by persulfate in the presence of ultraviolet light. The CO_2 is measured directly by a nondispersive infrared detector. This number is then converted to carbon and the result is displayed as ppm C.

3.2 This SOP is based upon EPA Method 415.1.

3.3 This SOP deviates from EPA Method 415.1 in the following way:

3.3.1 Samples are preserved with either sulfuric or phosphoric acid.

4.0 INTERFERENCES

4.1 Substantial solid organic matter is not efficiently oxidized, therefore, this method measures principally dissolved organic carbon in water. Persulfate oxidation of organic molecules is slowed in samples containing significant concentrations of chloride by the preferential oxidation of chloride; at a concentration of 0.1%, chloride oxidation of organic matter may be inhibited completely. Excessive acidification of sample, producing a reduction in pH of the persulfate solution to 1 or less, can result in sluggish and incomplete oxidation of organic carbon. Removal of carbonate and bicarbonate by acidification and purging with purified gas results in the loss of volatile organic substances. The intensity of the ultraviolet light reaching the sample matrix may be reduced by highly turbid samples or by aging of the ultraviolet source,

resulting in sluggish or incomplete oxidation. Large organic particles or very large or complex organic molecules such as tannins, lignins, and humic acid may be oxidized slowly because persulfate oxidation is rate-limited.

5.0 SAFETY

5.1 Potassium persulfate is a strong oxidizer. Safety glasses, lab coat, and gloves are necessary to prevent contact with solid or solution made from solid.

5.2 Phosphoric acid is corrosive. Safety glasses, lab coat, and gloves are also necessary to prevent contact.

6.0 SAMPLE CONTAINERS, COLLECTION AND PRESERVATION

6.1 Sample containers may be of plastic or glass, thoroughly rinsed and dried before use and are not to be reused. Typically I-Chem brand series 200 40 mL glass vials are used.

6.2 Aqueous samples are collected into bottles containing 3 - 5 drops of concentrated sulfuric or phosphoric acid. Sample vials should be filled completely, eliminating any headspace. Samples should be packaged and delivered to the laboratory at 4 $\pm$ 2° C, where they are maintained until analysis.

6.3 Samples must be preserved with sulfuric or phosphoric acid to a pH of 2. NEVER PRESERVE SAMPLES WITH HYDROCHLORIC ACID.

6.4 The holding time for TOC analysis is 28 days from sampling.

7.0 APPARATUS AND MATERIALS

7.1 Dohrmann DC - 180 Carbon Analyzer or equivalent.

8.0 REAGENTS AND STANDARD PREPARATION

8.1 Reagents:

8.1.1 Reagent Water: 16.6 megohm, deionized.

8.1.2 Potassium Persulfate Solution (3%): Prepare by dissolving 60 grams of reagent grade Potassium Persulfate ($K_2S_2O_8$) in 2 liters of reagent grade water. Add 2 mLs of concentrated phosphoric acid and mix well.

8.2 Standards Preparation:

8.2.1 Organic Carbon Stock Standard (2000 mg/L): Weigh 0.425 grams of reagent grade KHP ($C_8H_5O_4K$), Potassium Hydrogen Phthalate, (dried to a constant weight at 105° C) and transfer quantitatively with 50 - 75 mLs of reagent grade water to a 100 mL

volumetric flask. Add 0.1 mL reagent grade concentrated phosphoric acid, and dilute to volume with reagent grade water. Refrigerate solution and replace monthly.

8.2.2 Organic carbon standard (10 mg/L): Dilute 5.0 mLs of 2000 mg/L organic carbon standard into a 1000 mL volumetric flask and dilute to volume with the highest grade, volatile-free reagent water available. Calibrate the instrument to 10.00 mg/L. Refrigerate solution in a dark glass container and replace monthly.

8.2.3 Initial and Continuing Calibration Verification Standard (12 mg/L): Purchased from Hach Co. (Loveland, CO.) Cat # 12186_11, 120 mg/L TOC. Dilute 10.0 mL volumetrically to 100 mL to make a 12 mg/L calibration verification standard.

8.2.4 Inorganic Carbon Standard (400 mg/L): Dissolve 0.3532 grams of reagent grade anhydrous Sodium Carbonate, Na_2CO_3 (dried to a constant weight) in a 100 mL volumetric flask with deionized water. Store this solution in dark glass and refrigerate. Replace monthly.

8.2.5 Inorganic Carbon Standard (10 mg/L): Dilute 25 mLs of 400 mg/L IC standard to 1 liter with deionized water.

9.0 CALIBRATION

9.1 Dohrmann DC - 180 Start-up:

Turn on the main power by pressing the square white button beside the display screen. The main menu is displayed. From the "System On/Off" menu ([+/-] [1]), select "O2/UV/Pump On". Observe that the UV lamp is on and that gas is bubbling in both the UV reactor vessel and the G/L separator, and that the peristaltic pump is on. From "Main Menu", select "monitor Baseline". Wait for a stable baseline ($\pm$ 5 mg/L C) before starting an analysis. Check "Sequence times" ([+/-] [2] [1]), to ensure correspondence with loop volumes. Refer to Table 1 for pick up loop criteria and Table 2 for injection loop criteria.

9.2 Calibration:

From "Calibration Modes" menu ([+/-] [2] [4]), select the mode of interest. The instrument should be calibrated on both the "NPOC with injection loop" mode; using a 10 mg/L organic carbon standard, and the "IC and POC" mode; using the 10 mg/L Inorganic standard (If POC values are not desired, disregard the "IC and POC" calibration). When [1], [2], or [3] is selected, the appropriate loop volume is displayed with a blinking cursor at the bottom of the screen. If this loop size is correct, press [ENTER]; otherwise use [CLEAR] to erase the line and then enter the correct volume in mLs and then press [ENTER]. Submerge sample pick-up line into container that has the standard solution. Press [CAL], enter the mg/L concentration of the standard being used and then press [ENTER]. After "Run Conditions" are printed, if ready, press [YES]. [NO] will exit from CAL mode and return to the main menu. The CAL factor for the mode selected will be automatically adjusted at

the end of the analysis. Compare the results obtained with Table 3. If your value falls outside the expected range, double check gas flow rate (200 $\pm$ 10% mL/min.) and pump tubes. If OK, the baseline may not have been stable. Calibrate again. When satisfactory calibration factors have been obtained, press [9] on the "Calibration Modes" menu to print them. Determine system blank values (see 9.3) for both NPOC and POC (if POC values are desired). Repeat calibration procedure using 10 + system blank as the standard concentration. (i.e., If the NPOC system blank value is 0.521, recalibrate using the 10 mg/L standard but enter 10.521 as its concentration.) After recalibration, press [8] on the "Calibration Modes" menu and enter the system blank values for the corresponding Calibration Mode. Reprint calibration factors. The instrument is now ready to run unknown samples.

9.3 Determining System Blanks:

The system blank represents the instrumental background contribution and must be determined for both NPOC and POC (if POC values are desired). Before running the system blank (which is UV-reactor fluid), make the following hardware changes:

a) Change the pump tubing:

Switch the teflon lines from the purple/black pump tubing in position #6 to the blue/yellow pump tubing in position #7.

b) Hook up blanking valve:

Disconnect the sample intake line (blue fitting) from the inside of the pump housing compartment and connect it to the blanking valve (see Diagram 1). When running the system blank, remember the sample pick-up time may need to be adjusted because of the pump tubing change. Open the blanking valve. Allow the reactor fluid to clean the lines for few minutes. When the baseline is steady, run the system blank as sample analysis (see 11.3). Repeat until reproducible data is obtained. Average the POC values and the NPOC values separately. Use these averages to recalibrate and enter them as the system blanks.

9.4 Check calibration daily by injection of 10 mg/L standard (Section 8.2.5). If the standard is off by more than 5%, then a recalibration is needed.

10.0 QUALITY CONTROL

10.1 Method detection limits are performed and calculated in accordance with the SOP for Conducting MDL Studies, IEA Doc# QAS02000.NET.

10.2 The Practical Quantitation Limit for this method is 1.0 mg/L.

10.3 Daily Instrument Calibration check is performed at the beginning of each day of instrument operation to verify proper functioning of the detector (Section 9.2).

10.4 Matrix spikes are performed by pipetting 100 uL of 2000 mg/L stock TOC standard (Sec. 8.2) into 25.0 mL of sample to make a 8.0 mg/L TOC spike.

10.5 QC Check Samples consist of Initial and Continuing Calibration Verification standards (ICV and CCV, respectively), Section 8.2.3.

10.6 Surrogates are not applicable to this method.

10.7 QC SCHEDULES:

ACTIVITY:	FREQUENCY:
Initial Calibration Verification	Onset of every analysis
Initial Calibration Blank	Onset of every analysis
Duplicate Sample Analysis	Every 20 samples, per batch
Matrix Spike	Every 20 samples, per batch
Continuing Calibration Blank	Every 10 samples, per analysis
Continuing Calibration Verification	Every 10 samples, per analysis

10.7.1 DEFINITIONS:

Batch: Every 20 samples leached, digested, or distilled, per day.
Analysis: Each calibration, each day.
Duplicate sample: A separate sample aliquot that is carried through preparation and analysis.

11.0 SAMPLE PREPARATION AND INSTRUMENTAL PROCEDURES

11.1 Instrumental conditions:

Section 9 outlines the specific procedures involved in starting up the Dohrmann DC-180 Carbon Analyzer for TOC analysis.

11.2 Sample Preparation:

No sample preparation is required.

11.3 Sample Analysis:

From "Sampling" menu ([+/-] [2] [2]), verify the "number of repeats" (number of times pick-up loop is filled with sample) and "# of inj./repeat", (number of times inj. loop is filled from pick-up loop). Verify loop volumes. Press [5] if multi-sampler use is desired. Press [1], [2], and [3] to toggle Multi-sampler option responses. When sampling options are specified, verify "sequence times" ([+/-] [2] [1]) in accordance with Table 4. From "Analysis Modes" menu ([+/-] [2] [3]), select [4] for TOC analysis. Answer [NO] to question at bottom of screen. If the baseline is steady, set up samples and press [RUN].

12.0 CALCULATIONS

12.1 ICV, and CCV Recovery:

$$\frac{\text{Actual mg/L}}{\text{Theor. mg/L}} \times 100 = \%\ \text{Recovery}$$

12.2 Spike Recovery:

$$\frac{(\text{Spiked sample} + \text{sample conc., mg/L}) - (\text{sample conc., mg/L})}{40.00\ \text{mg/L}} \times 100 = \%\ \text{Recovery}$$

12.3 Method blank, Initial Calibration and Continuing Calibration blanks:
Not to exceed 1.0 mg/L (MDL)

12.4 Relative Percent Difference:

$$\left[\frac{(\text{Sample 1} - \text{Sample 2})}{\text{avg., Sample 1 \& 2}}\right] \times 100 = \%\ \text{RPD}$$

13.0 ACCEPTANCE CRITERIA

13.1	ICB, CCB	< 1.0 mg/L
	ICV, CCV	85% - 115% Recovery
	Matrix spike	75% - 125% Recovery
	Duplicates	± 20 % RPD or ± 1 mg/L (MDL) if sample conc. < 5 x MDL

13.2 If any of the calibration criteria (ICV, CCV, ICB, CCB) are not met, analysis is terminated. The source of the error is investigated and corrected and all samples are reanalyzed up to the last valid standard before the anomaly occurred. Matrix spikes that do not fall within the specified range are reanalyzed to verify the analysis. If the spike still does not fall within the acceptance limits, another sample aliquot is re-spiked at the same level and reanalyzed. If the recovery is outside the acceptance limits a method discrepancy form or "analyst comment" noting the anomaly is documented in the project folder.

14.0 REPORTING OF RESULTS

14.1	Practical Quantitation Limits:	1.0 mg/L
	Units of Measure:	mg/L
	Significant Figures:	2 figures
	QC information included in report:	
	Level I Report	Results, QC Blank Data
	Level II Report	Results, QC Blank Data, Duplicate Data, and MS %Recovery Data

Level III Report	Modified CLP forms 1, 2, 3, 4, 6, 7 and all raw data to support the forms.

15.0 SUPPLEMENTAL DOCUMENTS

15.1 SOP for Conducting MDL Studies, IEA Doc# QAS02000.NET.

16.0 REFERENCES

16.1 Standard Methods For the Examination of Water and Wastewater, 16th edition, Method 505B, 1985.

16.2 DC - 180 Total Organic Carbon Analyzer Operation Manual, 5th Edition, October 1990, Part No. 915-233.

16.3 EPA Methods for Chemical Analysis of Water and Wastes, Method 415.1, 1983.

17.0 SUBSTANTIVE REVISIONS

Original Issue

GUIDELINES FOR SETTING UP SEQUENCE TIMES

Please note actual times may change slightly due to variances in gas pressure. Pay close attention to the Pick-Up Loop to Sparger and Injection Loop fill times when setting up your analysis.

TABLE 1

NPOC with Pick-Up Loop						
PICK-UP LOOP VOL. (mL)	TYPICAL TOC/POC/ IC RANGE	EMPTY SPARGER	PICK-UP LOOP FILL TIME (sec)	PU LOOP TO SPARGER (sec)	SPARGE TIME (sec)	INJECT. LOOP FILL TIME (sec)
10	10 ug/L - 16 mg/L	30	210 *	30	150	40
5	20 ug/L - 32mg/L	15	105 *	20	90	20
2	50 ug/L - 80 mg/L	7	45 *	10	60	14

* Add 30 seconds if using the Multi-sampler.

TABLE 2

NPOC with Inj. Loop				with Multisampler	
INJECTION LOOP VOL.	TYPICAL TOC RANGE (mg/L)	INJECTIO LOOP FILL TIME (sec) (at ~5 psi)	TOTAL # of INJECTIONS POSSIBLE	REPEATS POSSIBLE - 20 mL vial	REPEATS POSSIBLE - 50 mL vial
1 mL	0.1 - 160	5 - 7	3	1	3
200 uL	10 - 800	5 - 7	3	1	3
60 uL	80 - 2600	5 - 7	3	1	3
1 mL	0.1 - 160	3 - 5	2	3	5
200 uL	10 - 800	3 - 5	2	3	5
60 uL	80 - 2600	3 - 5	2	3	5
1 mL	0.1 - 160	1 - 2	1	5	10
200 mL	10 - 800	1 - 2	1	5	10
60 mL	80 - 2600	1 - 2	1	5	10

TABLE 3
EXPECTED CALIBRATION RANGES

Vol Injected (mL)	Std Conc. (mg/L)	Expected* Raw Counts Range	Expected CAL Factor Range
10	10	960000-1600000	6E-05 - 1E-04
5	10	480000-800000	6E-05 - 1E-04
2	10	192000-320000	6E-05 - 1E-04
1	10	96000-160000	6E-05 - 1E-04
0.2	400	768000-1280000	6E-05 - 1E-04
0.06	2000	1152000-1920000	6E-05 - 1E-04
0.04	2000	768000-1280000	6E-05 - 1E-04

* Gas flow rate is approx. 200 $\pm$ 10% mL/min.

TABLE 4
SEQUENCE TIMES

NPOC with Pick-Up Loop						
Pick-Up Loop Vol. (mL)	Typical TOC Range	Empty Sparger (sec)	Pick-Up Loop Fill Time (sec)	PU Loop to Sparger (sec)	Sparge Time (sec)	Inj. Loop Fill Time (sec)
10	10 ug/L - 16 mg/L	30	210*	30	150	40
5	20 ug/L - 32 mg/L	15	105*	20	90	20
2	50 ug/L - 80 mg/L	7	45*	10	60	14

* Add 30 seconds if using the Multisampler.

26. IEA SOP FOR TOC IN SEDIMENT

8.2 Standard solutions:

Working Standard (100,000 ppm C): Weigh 23.75 grams of reagent grade Sucrose ($C_{12}H_{22}O_{11}$) dried to a constant weight at 105°C and transferred quantitatively with volatile free DI water (8.1.1) to a 100 milliliter volumetric flask.

-Calibration Verification Standard (2000 mg/L): Purchased from Rosemount Analytical Division P/N 511-920.

9.0 CALIBRATION

9.1 The calibration curve is prepared as follows using the above working standard (8.2) and volatile-free reagent water (8.1.1) available:

Conc. of Std. to dilute (mg/L)	mL of Std.	Final volume (mL)	Final concentration(mg/L)
100000	20.00	100	20000
100000	10.00	100	10000
100000	5.00	100	5000
100000	2.00	100	2000
5000	2.00	100	100
BLANK	0.00	100	0

9.2 The working calibration range of this method is 100 mg/L to 20000 mg/L.

9.3 Instrument Calibration:

9.3.1 Dohrmann DC-180 Start-up:
Turn on the main power by depressing the square white button beside the display screen and verify that the system main menu is being displayed. Turn on the POWER switch on the front panel of the Boat Sampling Module. Flip Boat Gas toggle switch to the ON position. Verify that oxygen is bubbling through the pyrex sparger on the front of the boat module and through the Gas/Liquid separator in the reactor compartment. Both vessels should be 1/3 full of DI water adjusted to a pH < 2 with phosphoric acid. Wait 15 minutes to allow the boat module to stabilize at 800 degrees C. Once temperature has stabilized, advance the platinum sample boat into the furnace. Allow the boat to bake there for about 2 minutes. Then, retract the boat so that it resides again in the hatch block. From "Main Menu", select "monitor baseline". Wait for a stable baseline ($\pm$

5 ppm) before starting an analysis.

9.3.2 Calibration:
From "Analysis Modes" menu ([+/-] [2] [3]), sele
YES to boat inlet question. Press RUN. Th
milligrams; enter [40], but do not press ENTER
syringe injection port and inject 40 uL of a 10,(
Immediately press ENTER. Slide Boat into the
now!". When the run is completed, the instrum
Repeat the injection procedure. If the two co
"continue?". The calibration factor will be autom
curve (as if each standard were a sample, see 9.1)
2000, 5000, 10000, and 20000 ppm. Inject 40
This default value will normalize all analytical re
Calibration curve must be run at the beginning o

9.4 Internal standards are not applicable to this meth

9.5 Acceptance criteria for the calibration require
coefficient of the calibration standards to be grea

10.0 QUALITY CONTROL

10.1 Method detection limits are calculated based on
definition of method detection limit. A minimum o
by referenced methods including all processing step
where appropriate. Method detection limits are cal
deviation of all replicate results by student t values

10.2 Practical quantitation limits for this method is 10

10.3 Matrix spikes are performed by injecting 40 u
standard (Sec. 8.2) onto the platinum boat contai
ng carbon spike.

10.4 QC Check Samples consist of Initial and Continu
(ICV and CCV, respectively), Section 8.2.

10.5 Surrogates are not applicable to this method.

10.6 QC SCHEDULES:

ACTIVITY:	FREQUENCY:
Initial Calibration Verification	Onset of every analysis
Initial Calibration Blank	Onset of every analysis
Quadruplicate Sample Analysis	Every 20 samples, per batch
Matrix Spike	Every 20 samples, per batch
Continuing Calibration Blank	Every 10 samples, per analysis
Continuing Calibration Verification	Every 10 samples, per analysis

Note: Quadruplicate analyses are used to maintain a control chart.

10.6.1 DEFINITIONS:
Batch: Every 20 samples leached, digested, or distilled, per day.
Analysis: Each calibration, each day.
Quadruplicate sample: A separate sample aliquot is carried through preparation and analysis each time.

11.0 SAMPLE PREPARATION AND INSTRUMENTAL PROCEDURES

11.1 Sample Preparation:
Weigh out between 10 and 50 mg of well mixed sample into the platinum boat. Add 1:1 phosphoric acid drop wise until effervescence stops. Heat to 75°C.

11.2 Sample Analysis:
A calibration curve must be run at the beginning of each analysis day, prior to any sample analysis. (see 9.3) From "Analysis Modes" menu ([+/-] [2] [3]) select [7] for Syringe injection. Answer YES to "Boat inlet?" question. Place boat containing the prepped sample into hatch port and press RUN. Enter [1] as the sample weight and wait for prompt to say "inject now!". Slide boat into oven and wait 2-8 minutes for the analysis to finish. The instrument will give an answer in ppm carbon. In actuality, this value is total nanograms of carbon because the default value of 1 was entered as the sample weight. Actual concentration in mg/kg must be calculated (see 12.5). Move boat back into the hatch port and wait 30 seconds to allow the boat to cool before removing. Answer [NO] to the question "continue?".

12.0 CALCULATIONS

12.1 ICV and CCV Recovery:

$$\frac{\text{Actual ng}}{\text{Theor. ng}} \times 100 = \%\ \text{Recovery}$$

12.2 Spike Recovery:

$$\frac{(\text{Spiked sample} + \text{sample conc., ng}) - (\text{sample conc., ng})}{80000\ \text{ng}} \times 100 = \%\ \text{Recovery}$$

12.3 Method blank, Initial Calibration and Continuing Calibration blanks:
Not to exceed 100 mg/kg (MDL)

12.4 Relative Percent Difference:

$$\left[\frac{(\text{Replicate 1} - \text{Replicate 2})}{\text{avg., Replicate 1 \& 2}}\right] \times 100 = \%\ \text{RPD}$$

12.5 Sample Concentration:

$$\frac{\text{TOC result, ng}}{\text{Sample Wt., mg}} \times \frac{1000\ \text{mg}}{1\ \text{g}} \times \frac{1000\ \text{g}}{1\ \text{kg}} \times \frac{1\ \text{ug}}{1000\ \text{ng}} \times \frac{1\ \text{mg}}{1000\ \text{ug}} = \frac{\text{mg}}{\text{kg}}$$

13.0 ACCEPTANCE CRITERIA TOC ANALYSIS:

ICB, CCB	< 100 mg/kg
ICV, CCV	85% - 115% Recovery
Matrix spike	75% - 125% Recovery
Duplicates	$\pm$ 20 % RPD or $\pm$ 1 mg/L (MDL) if sample conc. < 5 x MDL

If any of the calibration criteria (ICV,CCV,ICB,CCB) are not met, analysis is terminated. The source of the error is investigated and corrected and all samples are reanalyzed up to the last valid standard before the anomaly occurred. Matrix spikes that do not fall within the specified range are reanalyzed to verify the analysis. If the spike still does not fall within the acceptance limits, another sample aliquot is re-spiked at the same level and reanalyzed. If the recovery is outside the acceptance limits a method

discrepancy form or "analyst comment" noting the anomaly is documented in the project folder.

14.0 REPORTING OF RESULTS

Practical Quantitation Limits: Same as MDL.
Units of Measure: mg/kg
Significant Figures: 2 figures
QC information included in report:

Level 1 QC: Blank values.
Modified CLP Deliverable Data Package:
- Modified CLP forms 1,2,3,4,6,7 and all raw data to support the forms.
- Standards recoveries, blank data, matrix spike recoveries, duplicate data.

15.0 SUPPLEMENTAL DOCUMENTS

DC-180 Total Organic Carbon Analyzer Operation Manual, Edition 5, October 1990, Part No. 915-233.

Determination of Total Organic Carbon in Sediment. July 27, 1988.
Lloyd Kahn, Quality Assurance Specialist. U.S.E.P.A., Region II

Installation and Operation of the 183 Boat Sampling Module. Part No. 915-240. January, 1991

16.0 REFERENCES

Determination of Total Organic Carbon in Sediment. July 27, 1988.
Lloyd Kahn, Quality Assurance Specialist. U.S.E.P.A., Region II.

17.0 SUBSTANTIVE REVISIONS

17.1 Revised Section 6.3 to "The holding time for TOC analysis is 14 days from sampling." Section 9.1 amended from standard to working standard. Section 9.3.2 amended reference to Section 3.3 changed to Section 9.1. Amendment to Section 10.1: Chapter 1 changed to Part 136, Appendix B. Section 10.3 amended: Standard changed to working standard. Section 12.4 amended to more clearly express absolute value.

27. IEA SOP FOR TOTAL PHOSPHOROUS ANALYSIS

1.0 APPROVALS

The signatures of the following individuals indicate that this SOP is complete and meets the requirements specified in corporate document # QAS00200.NET. In addition, it signifies that the content meets the specifications of the referenced "Test Code".

Laboratory Director

Quality Assurance Manager

UNCONTROLLED COPY
PROPRIETARY DOCUMENT

2.0 SCOPE AND APPLICATION

2.1 Polyphosphates and organic phosphorus compounds are converted to orthophosphate by a sulfuric acid/persulfate digestion. Ammonium molybdate and antimony potassium tartrate then react in an acid medium with orthophosphate to form an antimony-phospho-molybdate complex. This complex is reduced to an intensely blue-colored complex by ascorbic acid. The color is proportional to the phosphorus concentration.

2.2 This procedure is applicable to drinking, surface, saline, and ground waters; domestic and industrial wastes, both solid and liquid; and soils and sludges.

2.2 This procedure is applicable to Test Codes P_01 and P_02.

3.0 SUMMARY OF METHOD

3.1 An aliquot or weight of sample is digested with sulfuric acid and potassium persulfate. The resulting solution is filtered, if necessary, to remove solid particles, and mixed with a color reagent containing sulfuric acid, antimony potassium tartrate, ammonium molybdate, and ascorbic acid. After 10 minutes but no more than 30 minutes the absorbance is read on a spectrophotometer at 880 nm. The concentration of phosphorus is determined from comparison with a calibration curve.

3.2 This procedure is based on Standard Methods 16th Edition 424.C.III and 424.F, and on EPA Method 365.2.

3.3 This procedure deviates from the above methods as follows:

3.3.1 100 ml of sample is digested instead of 50 ml.

3.3.2 The pH is not adjusted prior to digestion.

3.3.3 2 ml of 11N sulfuric acid and 1 g of potassium persulfate are used instead of 1 ml and 0.5 g, respectively.

3.3.4 The pH is adjusted after digestion using phenolphthalein, 6N sodium hydroxide, and 11N sulfuric acid, to clear.

3.3.5 A 50 ml aliquot is taken from the 100 ml digested solution for coloring.

3.3.6 Antimony potassium tartrate is stored in a dark bottle at 4 degrees C. Ammonium molybdate date is stored in a plastic bottle at 4 degrees C.

3.3.7 Phosphorus stock solution is 500 mg/L; intermediate standard is 50 mg/L; working standard is 2.5 mg/L.

4.0 INTERFERENCES

4.1 High iron concentrations can cause precipitation of and subsequent loss of phosphorus. Samples may be screened for iron content using CHEMetrics Iron Analysis kit. Dilution of sample prior to digestion may be necessary to reduce iron interference.

4.2 Arsenates react with the molybdate reagent to produce a blue color similar to that formed with phosphate. Concentrations as low as 0.1 mg As/L interfere with the phosphate determination.

4.3 Hexavalent chromium and nitrite interfere to give results about 3% low at concentrations of 1 mg/L and 10 to 15% low at 10 mg/L.

5.0 SAFETY

5.1 Concentrated sulfuric acid can cause severe burns. Goggles or face shield, lab coat, and gloves are necessary when using this reagent. Gloves are recommended when using dilute solutions of sulfuric acid.

5.2 Potassium persulfate is a strong oxidizer; contact with combustible materials may cause

fire. This reagent is harmful if inhaled or swallowed, and may cause skin irritation. Gloves are recommended when handling this reagent.

5.3 Sodium hydroxide can cause severe burns. Goggles or face shield, lab coat, and gloves are recommended when preparing solutions of this reagent. Gloves are recommended when using dilute solutions of sodium hydroxide.

5.4 Gloves are recommended when handling antimony potassium tartrate, ammonium molybdate, and ascorbic acid; avoid inhalation and contact with skin.

6.0 SAMPLE CONTAINERS, COLLECTION AND PRESERVATION

6.1 Sample containers: Containers may be plastic or glass, thoroughly rinsed and dried before use and are not to be reused.

6.2 Samples are preserved with sulfuric acid (2 ml/liter of sample) to pH less than 2 at time of collection. Samples are stored at 4 degrees C until analysis.

6.3 Samples must be preserved with sulfuric acid to pH less than 2.

6.4 The holding time for phosphorus analysis is 28 days from sampling. If samples are delayed in transit or received after the 28 day holding time, the client is notified and a resampling schedule is arranged.

7.0 APPARATUS AND MATERIALS

7.1 Spectronic 1201 spectrophotometer; 1 cm square cuvet

7.2 Top-loading balance, reading to 0.1 g

7.3 250 ml beakers, funnels, 100 ml volumetric flasks, and 125 ml Erlenmeyer flasks prerinsed with 50% hydrochloric acid and d.i. water

7.4 #40 filter paper

7.5 Oxford pipetter, 5-10 ml

7.6 Assorted beakers, graduated cylinders, transfer pipets, volumetric pipets, scoopulas

8.0 REAGENTS AND STANDARD PREPARATION

8.1 Reagents:

8.1.1 Reagent water, 16.6 meg-ohm, deionized

8.1.2 Potassium dihydrogen phosphate (KH_2PO_4), dried at 105 degrees C, ACS grade

8.1.3 Concentrated sulfuric acid (H_2SO_4), ACS grade

8.1.4 Potassium persulfate ($K_2S_2O_8$), ACS grade

8.1.5 Phenolphthalein indicator

8.1.6 Ethanol, 95%, not denatured

8.1.7 Sodium hydroxide (NaOH), ACS grade

8.1.8 Antimony potassium tartrate ($K(SbO)C_4H_4O_6$: 1/2 H_2O), ACS grade

8.1.9 Ammonium molybdate ($(NH_4)_6Mo_7O_{24}$: 4 H20), ACS grade

8.1.10 L-ascorbic acid, ACS grade

8.2 Reagent prep:

8.2.1 11N H2SO4: Carefully add 310 ml concentrated H2SO4 to 500 ml d.i. water in a 1-liter volumetric flask; swirl to mix; cool and dilute to mark.

8.2.2 Phenolphthalein indicator solution: Dissolve 0.5 g phenolphthalein in 50 ml 95% ethanol and 50 ml d.i. water.

8.2.3 6N NaOH: Dissolve 120 g NaOH pellets in d.i. water; cool and dilute to 500 ml.

8.2.4 5N H2SO4: Add 70 ml concentrated H_2SO_4 to 350 ml d.i. water in a 500 ml volumetric flask; swirl to mix; cool and dilute to mark.

8.2.5 Antimony potassium tartrate: Dissolve 1.3715 g in d.i. water; dilute to 500 ml. Store at 4 degrees C in a dark bottle.

8.2.6 Ammonium molybdate: Dissolve 20 g in 500 ml d.i. water. Store at 4 degrees C in a plastic bottle.

8.2.7 Ascorbic acid: Dissolve 1.76 g in 100 ml d.i. water. Prepare fresh at time of analysis.

8.2.8 Combined color reagent: Mix, in order, per 100 ml of reagent:

50 ml 5N H_2SO_4
5 ml antimony potassium tartrate
15 ml ammonium molybdate
30 ml ascorbic acid

Mix after addition of each reagent. Reagents should be at room temperature before mixing and must be mixed in the order given. Prepare fresh at time of analysis.

8.3 **Standard prep**:

8.3.1 Stock phosphorus solution, 500 mg/L: Dissolve 1.0985 g dried KH2PO4 in d.i. water; dilute to 500 ml. Prepare annually.

8.3.2 Intermediate standard, 50 mg/L: Dilute 10.0 ml of Stock to 100 ml. Prepare quarterly.

8.3.3 Working standard, 2.5 mg/L: Dilute 10.0 ml of Intermediate standard to 200 ml. Prepare fresh at time of analysis.

8.3.4 Initial and Continuing Calibration Verification standards: Purchased from Hach Co., Cat# 14243-10, 15 mg/L PO4 (= 4.89 mg/L P). ICV: Empty 10 ml voluette into a pre-rinsed 250 ml beaker; add 90 ml d.i. water. CCV: Empty 10 ml voluette into a pre-rinsed 100 ml volumetric flask; adjust pH as for calibration standards and bring to volume.

8.3.5 Matrix spiking solution: 2.5 mg/L Working standard, 10.0 ml added to sample prior to digestion.

9.0 CALIBRATION

9.1 Calibration Standard Curve

ml 2.5 mg/L standard to 100 ml	Concentration, mg/L
0	0
2.0	0.050
10.0	0.250
25.0	0.625
40.0	1.000

The pH of each standard is adjusted with one drop each of phenolphthalein, 6N NaOH, and 11N H_2SO_4 prior to bringing to volume.

9.2 The working calibration range of this method is 0.02 mg/L to 1.00 mg/L.

9.3 The absorbance of each standard is plotted against its concentration, using a calculator with linear regression.

9.4 Internal standards are not applicable to this method.

9.5 Acceptance criteria for the calibration requires the correlation coefficient of the absorbance vs. concentration to be at least 0.9975.

9.6 Correlation coefficients are calculated on a calculator using linear regression.

10.0 QUALITY CONTROL

10.1 Method detection limits are calculated based on the 40 CFR chapter 1 definition of method detection limit. A minimum of seven replicate standards prepared by referenced methods including all processing steps including distillation and digestion where appropriate. Method detection limits are calculated by multiplying the standard deviation of all replicate results by student t values listed in 40 CFR.

10.2 Practical quantitation limit for this method is the same as the method detection limit.

10.3 Matrix spikes are performed by pipetting 10.0 ml of 2.5 mg/L Working standard (8.3.3) into sample prior to digestion, to make a 0.25 mg/L spike.

10.4 QC Check Samples consist of Initial and Continuing Calibration Verification standards (ICV and CCV, respectively), Section 8.3.4.

10.5 Surrogates are not applicable to this method.

10.6 QC SCHEDULES:

ACTIVITY:	FREQUENCY:
Initial Calibration Verification	Onset of every analysis
Initial Calibration Blank	Onset of every analysis
Preparation blank	Every 10 samples, per batch
Continuing Calibration Verification	Every 10 samples, per analysis
Continuing Calibration Blank	Every 10 samples, per analysis
Matrix Spike	Every 10 samples, per batch

10.6.1 DEFINITIONS:
Batch: Every 10 samples leached, digested, or distilled, per day.
Analysis: Each calibration, each day.

11.0 SAMPLE PREPARATION AND INSTRUMENTAL PROCEDURES

11.1 INSTRUMENTAL CONDITIONS
Turn on Spectronic 1201. Allow instrument to complete Self-Check and set wavelength at 880 nm. Allow instrument to warm up at least 30 minutes before using. Place square cuvet holder in chamber.

11.2 SAMPLE PREPARATION

11.2.1 Pour 100 ml of sample into a pre-rinsed 250 ml beaker. (A smaller aliquot may be used and sufficient volume of d.i. water added to bring volume to 100 ml.)

11.2.2 For ICB, add 100 ml d.i. water instead of sample.

11.2.3 For ICV, empty 10 ml voluette of Hach standard into beaker; add 90 ml d.i. water.

11.2.4 Add 2 ml 11N H_2SO_4 and 1 g potassium persulfate; swirl to mix. Place beaker on hot plate and boil gently until volume is reduced to 10-15 ml. Remove and cool.

11.2.5 Rinse sides of beaker with d.i. water. Add 2 drops of phenolphthalein. Add 6N NaOH to pink color. Add 11N H_2SO_4 dropwise to clear.

11.2.6 Check sample for turbidity and solid matter. Filter if necessary through #40 paper into a pre-rinsed 100 ml volumetric flask. Rinse beaker 3 times with d.i. water, adding rinses to filter. Rinse filter 3 times with d.i. water and allow to filter through. Remove filter and rinse funnel into flasks.

11.2.7 If filtration is not needed, transfer sample to 100 ml volumetric flask; rinse beaker 3 times and add rinses to flask.

11.2.8 Bring to volume; cap and shake well. Note any turbidity, color, or precipitate in prepared sample. Do not refilter to remove turbidity or precipitate.

11.3 ANALYTICAL PROCEDURE

11.3.1 Transfer 50 ml of digested solution into a pre-rinsed 125 ml Erlenmeyer flask. Add 8 ml of combined color reagent and swirl to mix well.

11.3.2 After at least 10 minutes but no more than 30 minutes, set absorbance on Spectronic 1201 at zero using the 0 mg/L calibration standard.

11.3.3 Read each solution in succession, starting with the calibration standards; record the corresponding absorbance.

12.0 CALCULATIONS

12.1 Analyte:

$$\text{mg/L P} = \text{mg/L from curve}$$

$$\text{mg/L } PO_4 = (\text{mg/L P})(3.066)$$

where $$3.066 = \frac{\text{FW } PO_4}{\text{FW P}} = \frac{94.97\text{ g}}{30.97\text{ g}}$$

12.2 ICV, and CCV Recovery:

$$\frac{\text{Actual mg/L}}{4.89\ \text{mg/L}} \times 100 = \%\ \text{Recovery}$$

12.2 Spike Recovery:

$$\frac{(\text{Spiked sample conc., mg/L}) - (\text{unspiked sample conc., mg/L})}{0.25\ \text{mg/L}} \times 100 = \%\ \text{Recovery}$$

12.3 Method blank, Initial Calibration and Continuing Calibration blanks: Not to exceed 0.02 mg/L (MDL)

12.4 Relative Percent Difference:

$$\left[\frac{(\text{Sample 1 - Sample 2})}{\text{avg., sample 1 \& 2}}\right] \times 100 = \%\ \text{RPD}$$

13.0 ACCEPTANCE CRITERIA PHOSPHORUS ANALYSIS:

ICB, CCB	< 0.02 mg/L
ICV, CCV	85% - 115% Recovery
Matrix spike	75% - 125% Recovery
Duplicates	± 20 % or ± 1 mg/L (MDL) if sample conc. < 5 x MDL

14.0 REPORTING OF RESULTS

Practical Quantitation Limits: Same as MDL.
Units of Measure: mg/L
Significant Figures: 2 figures
Data Reporting:
Below quantitation limit: BQL

QC information included in report:

Level 1 QC: Blank values.

Level 2 QC: Blank values, Duplicate data (%RPD).

Level 3 QC: Blank values, Duplicate data (%RPD), matrix spike recoveries, specific to client's samples.

15.0 SUPPLEMENTAL DOCUMENTS [Not applicable]

16.0 REFERENCES

16.1 Standard Methods 16th Edition 424.C III and 424.F

16.2 EPA Method 365.2

17.0 SUBSTANTIVE REVISIONS

17.1 Addendum to Section 6.4 to clarify holding time. Amendment to Section 8.1.1 from 18.0 to 16.6 mega-ohm d.i. reagent water. Section 12.4 amended to more clearly express absolute value.

28. IEA SOP FOR ORTHOPHOSPHATE ANALYSIS

1.0 APPROVALS

The signatures of the following individuals indicate that this SOP is complete and meets the requirements specified in corporate document # QAS00200.NET. In addition, it signifies that the content meets the specifications of the referenced "Test Code".

Laboratory Director

Quality Assurance Manager

UNCONTROLLED COPY
PROPRIETARY DOCUMENT

2.0 SCOPE AND APPLICATION

2.1 This method describes the procedure for the analysis of aqueous samples for orthophosphate.

2.2 This procedure is applicable to drinking, surface, saline, and ground waters, and domestic and industrial wastes.

2.2 This procedure is applicable to Test Code OPO4_1.

3.0 SUMMARY OF METHOD

3.1 Ammonium molybdate and antimony potassium tartrate react in an acid medium with dilute solutions of phosphorus to form antimony-phospho-molybdate complex. This complex is reduced to an intensely blue-colored complex by ascorbic acid. The color is proportional to the phosphorus concentration. Only orthophosphate forms a blue color in this test.

3.2 This procedure is based on EPA Method 365.2 and on Standard Methods, 17th Edition, Method 4500-P E.

3.3 This procedure deviates from the above methods as follows:

3.3.1 A 50 ml aliquot of sample is colored.

3.3.2 Antimony potassium tartrate is stored in a dark bottle at 4 °C. Ammonium molybdate is stored in a plastic bottle at 4 °C.

3.3.3 Phosphorus stock solution is 500 mg/L; intermediate standard is 50 mg/L; working standard is 2.5 mg/L.

4.0 INTERFERENCES

4.1 High iron concentrations can cause precipitation of and subsequent loss of phosphorus. Samples may be screened for iron content using CHEMetrics Iron Analysis kit. Dilution of sample may be necessary to reduce iron interference.

4.2 Arsenates react with the molybdate reagent to produce a blue color similar to that formed with phosphate. Concentrations as low as 0.1 mg As/L interfere with the phosphate determination.

4.3 Hexavalent chromium and nitrite interfere to give results about 3% low at concentrations of 1 mg/L and 10 to 15% low at 10 mg/L.

5.0 SAFETY

5.1 Concentrated sulfuric acid can cause severe burns. Goggles or face shield, lab coat, and gloves are necessary when using this reagent. Gloves are recommended when using dilute solutions of sulfuric acid.

5.2 Sodium hydroxide can cause severe burns. Goggles or face shield, lab coat, and gloves are recommended when preparing solutions of this reagent. Gloves are recommended when using dilute solutions of sodium hydroxide.

5.3 Gloves are recommended when handling antimony potassium tartrate, ammonium molybdate, and ascorbic acid; avoid inhalation and contact with skin.

6.0 SAMPLE CONTAINERS, COLLECTION AND PRESERVATION

6.1 Sample containers: Containers may be plastic or glass, thoroughly rinsed and dried before use and are not to be reused.

6.2 Samples must be preserved with sulfuric acid (2 ml/liter of sample) to pH less than 2 at time of collection. Samples are stored at 4 degrees C until analysis.

6.3 The holding time for ortho-phosphate analysis is 48 hours from sampling.

7.0 APPARATUS AND MATERIALS

7.1 Spectronic 1201 spectrophotometer; 1 cm square cuvet

7.2 250 ml beakers, funnels, 100 ml volumetric flasks, 500 mL volumetric flasks and 125 ml Erlenmeyer flasks prerinsed with 50% hydrochloric acid and d.i. water

7.3 #40 filter paper

7.4 Oxford pipetter, 5-10 ml

7.5 Assorted beakers, graduated cylinders, transfer pipets, volumetric pipets, scoopulas

8.0 REAGENTS AND STANDARD PREPARATION

8.1 Reagents

8.1.1 Reagent water, 16.6 mega-ohm, deionized

8.1.2 Potassium dihydrogen phosphate (KH_2PO_4), dried at 105 degrees C, ACS grade

8.1.3 Concentrated sulfuric acid (H_2SO_4), ACS grade

8.1.4 Sodium hydroxide (NaOH), ACS grade

8.1.5 Antimony potassium tartrate ($K(SbO)C_4H_4O_6$: 1/2 H2O), ACS grade

8.1.6 Ammonium molybdate ($(NH_4)_6Mo_7O_{24}$ * 4 H20), ACS grade

8.1.7 L-ascorbic acid, ACS grade

8.2 Reagent prep:

8.2.1 6N NaOH: Dissolve 120 g NaOH pellets in distilled water; cool and dilute to 500 mL.

8.2.2 1 N NaOH: Dissolve 40 g NaOH pellets in distilled water; cool and dilute to 1 liter.

8.2.3 0.02 N NaOH: Dilute 5 mL 1 N NaOH to 250 mL with distilled water.

8.2.4 1 N H_2SO_4: Dilute 28 mL concentrated H_2SO_4 to 1 liter with distilled water.

8.2.5 0.02 N H_2SO_4: Dilute 5 mL 1 N H_2SO_4 to 250 mL with distilled water.

8.2.6 5 N H_2SO_4: Add 70 mL concentrated H_2SO_4 to 350 mL distilled water in a 500 mL volumetric flask; swirl to mix; cool and dilute to mark.

8.2.7 Antimony potassium tartrate: Dissolve 1.3715 g in distilled water; dilute to 500 mL. Store at 4 °C in a dark bottle.

8.2.8 Ammonium molybdate: Dissolve 20 g in 500 mL distilled water. Store at 4 °C in a plastic bottle.

8.2.9 Ascorboc acid: Dissolve 1.76 g in 100 mL distilled water. Prepare fresh at time of analysis.

8.2.10 Combined color reagent: Mix, in order, per 100 ml of reagent:

50 ml 5N H_2SO_4
5 ml antimony potassium tartrate
15 ml ammonium molybdate
30 ml ascorbic acid

Mix after addition of each reagent. Reagents should be at room temperature before mixing and must be mixed in the order given. Prepare fresh at time of analysis.

8.3 Standard prep:

8.3.1 Stock phosphorus solution, 500 mg/L: Dissolve 1.0985 g dried KH2PO4 in d.i. water; dilute to 500 ml. Prepare annually.

8.3.2 Intermediate standard, 50 mg/L: Dilute 10.0 ml of Stock to 100 ml. Prepare quarterly.

8.3.3 Working standard, 2.5 mg/L: Dilute 10.0 ml of Intermediate standard to 200 ml. Prepare fresh at time of analysis.

8.3.4 Initial and Continuing Calibration Verification standards: Purchased from Hach Co., Cat # 21092-10, 25 mg/L. Empty 10 ml voluette into a prerinsed 500 mL volumetric flask

containing 400 mL d.i. water; dilute to volume. This constitutes a 0.50 mg/L standard.

8.3.5 Matrix spiking solution: 50 mg/L Intermediate standard, 250 ul added to 50 ml sample before addition of color reagent.

9.0 CALIBRATION

9.1 Calibration Standard Curve

ml 2.5 mg/L standard to 100 ml	Concentration, mg/L
0	0
1.0	0.025
5.0	0.125
15.0	0.375
25.0	0.625
40.0	1.000

9.2 The working calibration range of this method is 0.02 mg/L to 1.00 mg/L.

9.3 The absorbance of each standard is plotted against its concentration, using a calculator with linear regression.

9.4 Internal standards are not applicable to this method.

9.5 Acceptance criteria for the calibration requires the correlation coefficient of the absorbance vs. concentration to be at least 0.9975.

9.6 Correlation coefficients are calculated on a calculator using linear regresion.

10.0 QUALITY CONTROL

10.1 Method detection limits are calculated based on the 40 CFR, Part 136, Appendix B, definition of method detection limit. A minimum of seven replicate standards prepared by referenced methods including all processing steps including distillation and digestion where appropriate. Method detection limits are calculated by multiplying the standard deviation of all replicate results by student t values listed in 40 CFR.

10.2 Practical quantitation limit for this method is the same as the method detection limit.

10.3 Matrix spikes are performed by pipetting 250 ul of 50 mg/L Intermediate standard (8.3.2) into a 50 ml aliquot of sample prior to the addition of color reagent, making a 0.25 mg/l spike.

10.4 QC Check Samples consist of Initial and Continuing Calibration Verification standards (ICV and CCV, respectively), Section 8.3.4.

10.5 Surrogates are not applicable to this method.

10.6 QC SCHEDULES:

ACTIVITY:	FREQUENCY:
Initial Calibration Verification	Onset of every analysis
Initial Calibration Blank	Onset of every analysis
Preparation blank	Every 10 samples, per batch
Continuing Calibration Verification	Every 10 samples, per analysis
Continuing Calibration Blank	Every 20 samples, per analysis
Matrix Spike	Every 20 samples, per batch
Duplicate	Every 20 samples, per batach

10.6.1 DEFINITIONS:
Batch: Every 20 samples leached, digested, or distilled, per day.
Analysis: Each calibration, each day.

11.0 SAMPLE PREPARATION AND INSTRUMENTAL PROCEDURES

11.1 INSTRUMENTAL CONDITIONS
Turn on Spectronic 1201. Allow instrument to complete Self-Check and set wavelength at 880 nm. Allow instrument to warm up at least 30 minutes before using. Place square cuvet holder in chamber.

11.2 SAMPLE PREPARATION

11.2.1 Calibrate pH meter using appropriate analytical SOP.

Pour approximately 150 ml of sample into a pre-rinsed 250 ml beaker. Add stir bar, place beaker on stir plate, and place probes in solution. Adjust pH of solution to between 6.8 and 7.2, using 6 N, 1 N and 0.02 N NaOH, and 1 N and 0.02 N H_2SO_4, as

necessary.

11.2.2 Remove beaker from pH meter. If sample has turned golden-yellow and/or formed flocc during pH adjustment, add 11 N H_2SO_4 dropwise until color and/or flocc re-dissolves.

11.2.3 For ICB, add 150 ml of d.i. water to a labeled beaker. Adjust pH as above.

11.2.4 For ICV, add 150 mL of ICV solution to labeled beaker. Adjust pH as above.

11.2.5 For CCV, add 150 mL of ICV solution to labeled beaker. Adjust pH as above.

11.3 ANALYTICAL PROCEDURE

11.3.1 Transfer 50 ml of prepared solution into a pre-rinsed 125 ml Erlenmeyer flask. Add 8 ml of combined color reagent and swirl to mix well.

11.3.2 After at least 10 minutes but no more than 30 minutes, set absorbance on the Spectronic 1201 at zero using the 0 mg/L calibration standard.

11.3.3 Read each solution in succession, starting with the calibration standards; record the corresponding absorbance. If sample absorbance is off the curve, dilute another aliquot of the sample and re-color as above.

12.0 CALCULATIONS:

12.1 Analyte: $\text{mg/L from curve} \times \frac{100\ \text{mL}}{\text{mL sample}} = \text{mg/L as P}$

12.2 ICV and CCV Recovery:

$$\frac{\text{Actual mg/L}}{0.50\ \text{mg/L}} \times 100 = \%\ \text{Recovery}$$

12.3 Spike Recovery:

$$\frac{(\text{Spiked sample conc., mg/L}) - (\text{unspiked sample conc., mg/L})}{0.25\ \text{mg/L}} \times 100 = \%\ \text{Recovery}$$

12.4 Method blank, Initial Calibration and Continuing Calibration blanks: Not to exceed 0.02 mg/L (MDL).

12.5 Relative Percent Difference:

$$\left[\frac{\text{(Sample 1 - Sample 2)}}{\text{avg., sample 1 \& 2}} \right] \times 100 = \% \text{ RPD}$$

13.0 ACCEPTANCE CRITERIA PHOSPHORUS ANALYSIS:

ICB, CCB	< 0.02 mg/L
ICV, CCV	85% - 115% Recovery
Matrix spike	75% - 125% Recovery
Duplicates	± 20 % or ± 0.02 mg/L (MDL) if sample conc. < 5 x MDL

14.0 REPORTING OF RESULTS

Practical Quantitation Limits: Same as MDL.
Units of Measure: mg/L
Significant Figures: 2 figures
Data Reporting:
Below quantitation limit: BQL
QC information included in report:
Level 1 QC: Blank values.
Level 2 QC: Blank values, Duplicate data (%RPD).
Level 3 QC: Blank values, Duplicate data (%RPD), matrix spike recoveries, specific to client's samples.

15.0 SUPPLEMENTAL DOCUMENTS [None at this time]

16.0 REFERENCES

16.1 EPA Method 365.2.

16.2 Standard Methods, 17th Edition, Method 4500-P E.

16.2 40 CFR, Part 136, Appendix B.

17.0 SUBSTANTIVE REVISIONS

17.1 Amendment made to Section 2.1. Revision made to Section 3.1. Amendment made to Section 3.3. Addendum to Section 7.2. Amendments made to Sections 8.1 and 8.2. Amendment to 10.1. Revisions to Sections 11.2.1 and 11.2.2. Amendments to Sections 11.2.5 and 11.3.3. Revisions to Section 12.1 and Section 12.2. Amendment to Section 12.5. Amendment to Section 13.0. Amendment to Section 16.2.

29. IEA SOP FOR TOTAL KJELDAHL NITROGEN

1.0 APPROVALS

The signatures of the following individuals indicate that this SOP is complete and meets the requirements specified in corporate document # QAS00200.NET. In addition, it signifies that the content meets the specifications of the referenced "Test Code".

Laboratory Director

Quality Assurance Manager

2.0 SCOPE AND APPLICATION

2.1 This method describes the determination of Total Kjeldahl Nitrogen in drinking and surface waters, domestic, industrial wastes, and solids. The procedure converts nitrogen components of biological origin such as amino acids, proteins and peptides to ammonia, but may not convert nitrogenous compounds of some industrial wastes such as amines, nitro compounds, hydrazones, oximes, semicarbazones and some refractory tertiary amines. The applicable range of this method is 0.1 to 2.0 mg/L TKN for liquid matrices and 25 to 5000 mg/kg TKN for solid matrices. The range may be extended with sample dilution.

2.2 This method is applicable to Test Codes TKN_01 and TKN_02.

3.0 SUMMARY OF METHOD

3.1 The sample is heated in the presence of sulfuric acid, K_2SO_4 and $HgSO_4$ for two and one half hours. The residue is cooled, diluted to 25 mL and placed on the Lachat QuikChem AutoAnalyzer for ammonia determination. This digested sample may also be used for phosphorous determination.

3.2 This procedure is based on EPA method 351.2 (Colorimetric, Semi-Automated Block Digester, AAII).

3.3 This procedure deviates from EPA method 351.2 in the following way:

3.3.1 The high temperature digestion is for a period of 3.5 hours instead of 2 1/2 hours as specified in the method.

4.0 INTERFERENCES

4.1 Samples must not consume more than half of the sulfuric acid during the digestion. The buffer will accommodate a range of 2 - 4% (v/v) [or 3.5 - 7% (w/w)] H_2SO_4 in the diluted digestion sample with no change in signal intensity.

5.0 SAFETY

5.1 Sulfuric acid is used in the digestion process which is an extreme corrosive and reacts severely with water. Safety glasses, gloves, and lab coat should be worn at all times.

5.2 Extreme caution should be taken when making the mercuric oxide stock solution for the digestion reagent. Precautions should be taken to avoid breathing the dust when weighing and gloves should be worn.

6.0 SAMPLE CONTAINERS, COLLECTION AND PRESERVATION

6.1 Sample containers: Containers must be 500 mL HDPE bottles, thoroughly rinsed and dried before use and are not to be reused.

6.2 Waters samples must be preserved to a pH below 2 using H_2SO_4. Water samples are cooled to 4 °C for transport to the laboratory and are stored at 4 °C until analysis.

6.3 Soil samples are cooled to 4 °C for transport to the laboratory and are stored at 4 °C until analysis.

6.4 Samples must be analyzed within 28 days of collection.

7.0 APPARATUS AND MATERIALS

7.1 Lachat QuikChem Autoanalyzer.

7.2 Lachat TKN chemistry manifold (10-107-06-2-F).

7.3 Block Digestor.

7.4 Chemware TFE (Teflon Boiling Stones).

7.5 20 mL repipettor.

7.6 Vortex mixer.

8.0 REAGENTS AND STANDARD PREPARATION

8.1 Reagents

8.1.1 Reagent Water, 16.6 megohm or higher, deionized.

8.1.2 Stock Mercuric Sulfate Solution: To a 100 mL volumetric flask, add 40 mL water, 10 mL of concentrated sulfuric acid and 8 g mercuric oxide. Stir until dissolved, dilute to volume, and invert three times.

8.1.3 Digestion Solution: In a 1 L volumetric flask, dissolve 133 g K_2SO_4 in about 700 ml reagent water and 200 mL concentrated H_2SO_4. Add 25 mL of stock mercuric sulfate solution and dilute to volume. Invert three times.

8.1.4 Buffer, pH 13.5: In a 1 L volumetric flask, dissolve 50.0 g sodium potassium tartrate, 65 g sodium hydroxide, and 26.8 g dibasic sodium phosphate heptahydrate in about 900 mL of reagent water. Dilute to volume and invert three times. Degas with helium.

8.1.5 Salicylate - Nitroprusside Color Reagent: In a 1 L volumetric flask, dissolve 150.0 g sodium salicylate and 1.00 g sodium nitroprusside in about 800 mL of water. Dilute to volume and invert three times. Degas with helium. Store in a light-proof bottle. Prepare at least monthly.

8.1.6 Hypochlorite reagent: In a 1 L volumetric flask, dilute 60 mL of regular Chlorox bleach (5.25% sodium hypochlorite, The Chlorox Company, Oakland, CA) to volume with water. Invert three times and degas with helium.

8.1.7 Ammonium Chloride Carrier: In a 1 L volumetric flask, dissolve 22 g of sodium chloride (NaCl). Degas with helium.

8.1.8 All reagent receipt and preparation is documented in the Reagent Receipt and Preparation Logbook (Attachment I).

8.2 Standard Solutions

8.2.1 Stock Standard, 1000 mg N/L as NH_3: In a 1 L volumetric flask dissolve 3.819 g of ammonium chloride (NH_4Cl) that has been dried for two hours at 110 deg C, in about 800 mL of water. Dilute to volume and invert three times. Store tightly stoppered.

8.2.2 Working Standard Solution A, 100 mg N/L: In a 100 mL volumetric flask, dilute 10.0 mL of stock standard (8.2.1) to volume with water. Invert three times.

8.2.3 Working Standard Solution B, 5.00 mg N/L: In a 100 mL volumetric flask, dilute 5.00 mL of working standard (8.2.2) to volume with water. Invert three times.

8.2.4 Working Standard Solution C, 0.50 mg N/L: In a 100 mL volumetric flask, dilute 10.00 mL of the working standard (8.2.3) to volume with water. Invert three times.

8.2.5 Ammonia Nitrogen Standard (ICV, CCV), (Hach Co., Loveland CO., cat# 1891-4923EF): 1.00 mg/L.

8.3 Calibration Standards

8.3.1 1.00 mg N/L standard: In a 75 mL digestion tube, add 5.0 mL of the 5.00 mg N/L working standard (8.2.3). Add reagent water for a final volume of approximately 25 mL. Digest as in Section 11.2.

8.3.2 2.00 mg N/L standard: In a 75 mL digestion tube, add 10.0 mL of the 5.00 mg N/L working standard (8.2.3). Add reagent water for a final volume of approximately 25 mL. Digest as in Section 11.2.

8.3.3 0.100 mg N/L standard: In a 75 mL digestion tube, add 5.0 mL of the 0.50 mg N/L working standard (8.2.4). Add reagent water for a final volume of approximately 25 mL. Digest as in Section 11.2.

8.3.4 0.200 mg N/L standard: In a 75 mL digestion tube, add 10.0 mL of the 0.50 mg N/L working standard (8.2.4). Add reagent water for a final volume of approximately 25 mL. Digest as in Section 11.2.

8.4 All standard receipt and preparation is documented in the Standard Receipt and Preparation Logbook (Attachment II).

9.0 CALIBRATION

9.1 Instrument Startup

9.1.1 Switch the power strip located behind the system computer to "on" position. When the computer has completed the self-diagnostics, the CPU monitor will display the main menu, with the METHODS category illuminated.

9.1.2 Install the TKN chemistry manifold onto the Lachat module, making proper connections to reagent lines as shown on the diagram in the Lachat method manual.

9.1.3 Place all reagent lines into the proper reagent containers and adjust the tension levers on the peristaltic pump until the first "click" is heard.

9.1.4 While the system is pumping reagents through the manifold, proceed with METHOD DOWNLOADING.

9.2 Method Downloading

9.2.1 To download the chemistry program to the system computer, observe the "METHODS" category illuminated at the CPU monitor, press "ENTER".

9.2.2 The monitor will display "Download Method" and "Method Definition". Using the arrow keys, highlight the "Download Method" and press "ENTER". A menu will appear listing pre-programmed methods in the system.

9.2.3 Move the cursor until "Total Kjeldahl Nitrogen" is highlighted, then press ENTER. The method will now start downloading the TKN chemistry to the system computer.

9.3 Calibration

9.3.1 To set up standard curve on sampler, obtain a vial for each standard starting with the 1.00 mg/L standard, fill the vial and place it in the standards rack in the front left spot. Continue with the other standards, in order of decreasing concentration.

9.3.2 The computer monitor will display "EDIT". Using the arrow keys highlight "SUBMIT" and press "ENTER". "Submit Current Tray" will be highlighted. Using the arrow keys highlight "Calibrate Now", and "ENTER".

9.3.3 The screen will give the prompt, "Should a calibration be started now?", press "Y". The Lachat will begin calibrating. When the calibration is complete the system will indicate whether the pass/fail criteria has been met by displaying "calibration failed!" or "calibration approved".

Note: Analysis cannot proceed until the calibration is approved.

9.4 Acceptance criteria for the calibration requires the correlation coefficient of the absorbance vs. standard concentration to be at least 0.9975 or better.

9.5 Correlation coefficients are calculated in the Lachat software based on a least squares linear regression. A correlation coefficient of $\geq$ 0.9975 is required.

9.6 The working range of the calibration is 0.100 to 2.00 mg N/L.

10.0 QUALITY CONTROL

10.1 Method Detection Limits (MDLs) are performed in accordance with the SOP for Conducting MDL Studies, IEA Doc# QAS02000.NET.

10.2 Practical Quantitation Limits for this method are 0.1 mg/L for water samples and 20 mg/kg for soil samples.

10.3 Daily analyzer performance tests are performed automatically. The Lachat software defaults to a diagnostics check on the valves and detectors after the initial powering-up of the system computer.

10.4 Matrix spikes are performed by pipetting 0.200 mL of working standard (8.2.2) into 20 mL of sample for liquid matrices or 0.10 g for solid matrices prior to digestion. This constitutes a matrix spike concentration of 1.0 mg N/L for liquid matrices and 200 mg/kg for solid matrices. Recovery criteria can be found in Section 13.1.

10.5 QC Check Samples consist of 20 mL of Initial and Continuing Calibration Verification standards (ICV and CCV, respectively), (Section 8.2.5). Recovery criteria can be found in Section 13.1.

10.6 Initial Calibration Blanks and Continuing Calibration Blanks (ICB and CCB, respectively) consist of 20 mL of reagent water. Acceptance criteria can be found in Section 13.1.

10.7 Preparation Blanks (PBW for water samples and PBS for soil samples) consist of 20 mL of reagent water that is prepared for analysis as samples are prepared. Acceptance criteria can be found in Section 13.1.

10.8 A duplicate sample is prepared for analysis every 10 samples. Acceptable Relative Percent Difference can be found in Section 13.1.

10.9 Surrogates are not applicable to this method.

10.10 QC Schedules

ACTIVITY:	FREQUENCY:
Initial Calibration Verification	Onset of every analysis
Initial Calibration Blank	Onset of every analysis
Preparation blank	Every 20 samples, per batch
Continuing Calibration Verification	Every 10 samples, per analysis
Continuing Calibration Blank	Every 10 samples, per analysis
Matrix Spike	Every 20 samples, per batch
Duplicate	Every 10 samples, per analysis

10.10.1 Definitions:

Batch: Every 20 samples digested and analyzed, per day.
Analysis: Each calibration, each day.

11.0 SAMPLE PREPARATION AND INSTRUMENTAL PROCEDURES

11.1 Instrumental Conditions: Sections 9.3.1-9.3.3 outline the specific procedures for startup and calibration for the Lachat Autoanalyzer for TKN analysis.

11.2 Sample Preparation

11.2.1 Pipet 20.0 mL of sample for liquid matrices or weigh 0.10 g for solid matrices into a 75 mL digestion tube. Add 5 mL of digestion solution (8.1.3) into digestion tube. Record the volume or weight of sample used in the Lachat Runlog (Attachment III). Mix well with the vortex mixer.

11.2.2 Add 4 to 8 boiling stones.

11.2.3 Place tubes in the preheated block digestor for one hour at 160 °C.

11.2.4 Continue to digest 3.5 hours at 380 °C.

11.2.5 Cool sample and dilute to 25.0 mL, volumetrically, with deionized water (8.1.1).

11.3 Sample Analysis

11.3.1 After obtaining an approved calibration (Section 9.3), press the [Esc] key until the "Sample Tray" program menu appears. Use the arrow keys to highlight "Edit" then hit [ENTER].

11.3.2 A sub menu will appear with "Identification" highlighted. Press [ENTER]. Enter the sample ID's in the exact order that they are arranged in the sample tray.

11.3.3 After the last sample has been entered, type two periods " .. " as the next sample entry; this indicates the end of the tray. Press the [ESC] key to back out to the "Sample tray" menu again.

11.3.4 Using the arrow keys, highlight "SUBMIT" and then press [ENTER]. The display will prompt the operator to submit the tray for analysis.

Note: Real time display of sample results may be accessed by pressing the [CTRL], [SHIFT], and [1] keys simultaneously. To return to the program menu, simply press [CTRL], [SHIFT], and [2] simultaneously. DO NOT use the arrow keys to scroll the screen in this mode; it will cause the computer to lock up. This will require the system to be shut down and the method downloading and calibration to be executed again.

12.0 CALCULATIONS

12.1 ICV and CCV Recovery:

$$\frac{\text{Actual mg/L}}{\text{Theor. mg/L}} \times 100 = \% \text{ Recovery}$$

12.2 Spike Recovery:

12.2.1 Liquid matrices:

$$\frac{(\text{Spiked sample conc., mg/L} - \text{sample conc., mg/L})}{1.0 \text{ mg/L}} \times 100 = \% \text{ Recovery}$$

12.2.2 Solid matrices:

$$\frac{(\text{Spiked sample conc., mg/kg} - \text{sample conc., mg/kg})}{200 \text{ mg/kg}} \times 100 = \% \text{ Recovery}$$

12.3 Relative Percent Difference:

$$\left[\frac{(\text{Sample 1} - \text{Sample 2})}{\text{avg., sample 1 \& 2}} \right] \times 100 = \% \text{ RPD}$$

13.0 ACCEPTANCE CRITERIA

13.1

ICB, CCB & Preparation Blank	< PQL
ICV, CCV	85% - 115% Recovery; 90-110% Recovery for NC Wastewater samples
Matrix spike	75% - 125% Recovery
Duplicates	± 20 % RPD or ± PQL or if sample conc. < 5 x MDL

14.0 REPORTING OF RESULTS

14.1 Practical Quantitation Limits: 0.1 mg/L for water samples; 20 mg/kg for soil samples
Units of Measure: mg/L for water samples; mg/kg for soil samples
Significant Figures: 2 figures
Data Reporting: Below quantitation limit: BQL
QC information included in report:
Level 1 QC: Blank values, Sampling COC.
Level 2 QC: Blank values, Duplicate data (%RPD), and Matrix spike recoveries, Sampling COCs.

Level 3 QC: Blank values, Duplicate data (%RPD), and Matrix spike recoveries (specific to client's samples), raw data, copy of log book page(s), Sampling and Laboratory COCs.

15.0 SUPPLEMENTAL DOCUMENTS

15.1 SOP for Conducting MDL Studies, IEA Doc# QAS02000.NET.

16.0 REFERENCES

16.1 Methods for Chemical Analysis of Water and Waste, 1983, EMSL, EPA-600/4-79-020, Method 351.2.

16.2 Lachat Method #10-107-06-2-F.

17.0 SUBSTANTIVE REVISIONS

17.1 Original Issue (12/29/94)

17.2 Sections 3.3, 6.3, 6.4, 8.1.8, 8.4, 10.6, 10.7, 10.8, 15.1 and 16.1 added. Sections 9.0 and 11.3 revised for clarification. Sections 10.2 and 14.1 revised to reflect current PQLs. Section 11.2.1 revised to 20 mL of initial sample volume. Section 11.2.4 revised to 3 hours for the high temperature digestion time. Matrix spike concentration revised in Sections 10.4 and 12.2.1. Equations corrected in Section 12.2. Section 13.1 revised to reflect current ICV/CCV recovery criteria for North Carolina Wastewater samples. (08/08/96)

Attachment I

Reagents Preparation and Receiving Log
Wet Chemistry Laboratory

Date	Analyst	Time	Reagent #	Reagent Name	Preparation Method	Expiration Date
			R			
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Secondary Review By:_____ Date:_____

Attachment II

Standards Preparation and Receiving Log
Inorganics Laboratory

Date	Analyst	Time	Standard #	Standard Name	Vendor	Preparation Method	Expiration Date

Secondary Review By:_____ Date:_____

Attachment III

Lachat Runlog

Analyst: _____ Instrument-#*

Preparation Log: _____

Filename: _____

Sample ID.	Sample Amount (g,ml)	Leachate/Dig Volume		Dilution		DF	Analysis mg/l,ug/l	Result mg/l,mg/kg	Comments
		g/ml	Final Volume	Vi	Vf				

Secondary Review By:_____ Date:_____

30. IEA SOP FOR NITRATE/NITRITE ANALYSIS

1.0 APPROVALS

The signatures of the following individuals indicate that this SOP is complete and meets the requirements specified in corporate document # QAS00200.NET. In addition, it signifies that the content meets the specifications of the referenced "Test Code".

Laboratory Director

Quality Assurance Manager

UNCONTROLLED COPY
PROPRIETARY DOCUMENT

2.0 SCOPE AND APPLICATION

2.1 This method pertains to the determination of nitrite singly, or nitrite and nitrate combined in surface and saline waters, and domestic and industrial wastes. Nitrate concentrations may be determined by calculcating the difference between nitrate and nitrite combined, and nitrite. The applicable range of this method is 0.02 mg/L to 2 mg/L nitrate-nitrite nitrogen. The range may be extended with sample dilution.

2.2 This procedure is applicable to test codes NO2_01, NO3_01, NOX_01, and NOX_03.

3.0 SUMMARY OF METHOD

3.1 Nitrate is quantitatively reduced to nitrite by passage of the sample through a copperized cadmium column. The nitrite (reduced nitrite plus the original nitrite) is then determined by diazotizing with sulfanilamide followed by coupling with N-(1-naphthyl)ethylenediamine dihydrochloride. The resulting water soluble dye has a magenta color which is read at 520 nm. Nitrate alone also can be determined by removing the cadmium column and calculating the difference between nitrate/nitrite singly.

3.2 This procedure is based on EPA Method 353.2.

3.3 This procedure does not deviate from EPA Method 353.2.

4.0 INTERFERENCES

4.1 Build up of suspended matter in the reduction column will restrict sample flow. Since nitrate-nitrogen is soluble, the sample may be pre-filtered.

4.2 Low results would be obtained for samples that contain iron, copper or other metals. In this method, EDTA is added to the buffer to reduce this interference.

4.3 Samples that contain large concentrations of oil and grease will coat the surface of the cadmium. This interference is eliminated by pre-extracting the sample with an organic solvent.

5.0 SAFETY

5.1 The cadmium in the cadmium column is very toxic and carcinogenic. Gloves must be worn when working with this column and all waste should be contained and disposed of in accordance with local and federal regulations.

6.0 SAMPLE CONTAINERS, COLLECTION AND PRESERVATION

6.1 Sample containers: Containers may be of plastic or glass, thoroughly rinsed and dried before use and are not to be reused.

6.2 Sample Collection: Nitrate samples are collected without preservatives as well as nitrite. Combined nitrate and nitrite samples are preserved with H_2SO_4 at a pH less than 2. Samples are packaged and sent to the laboratory and stored at 4 deg. C until analysis.

6.3 The holding time for nitrate and nitrite separately is 48 hours. The holding time for combined nitrate/nitrite is 28 days.

7.0 APPARATUS AND MATERIALS

7.1 Lachat QuikChem Autoanalyzer

7.1.1 Lachat Chloride chemistry manifold (Method # 10-117-07-1-B).

8.0 REAGENTS AND STANDARD PREPARATION

8.1 Reagents:

8.1.1 Reagent Water, 16.6 megohm, deionized.

8.1.2 Ammonium chloride buffer, pH 8.5
In the hood, to a 1 L volumetric flask add 500 mls water, 105 mls concentrated HCl, 95 mls ammonium hydroxide and 1.0 g disodium EDTA. Dissolve and dilute to the mark and invert three times.

8.1.3 Sulfanilamide color reagent,
To a 1 L volumetric flask add about 600 mls of water. Then add 100 mls of 85% phosphoric acid, 40.0 g sulfanilamide, and 1.0 g n-1-napthylethylenediamine dihydrochloride (NED). Shake to wet and stir to dissolve 20 minutes. Dilute to the mark and invert three times. Store in a dark bottle. This solution is stable for a month.

8.2 Standard solutions:

8.2.1 Stock nitrate solution (2000 mg/L): In a 1 L volumetric flask dissolve 14.44 g potassium nitrate (KNO_3) in about 600 mLs water. Add 2 mLs of chloroform. Dilute to the mark and invert three times. This solution is stable for six months.

8.2.2 Stock nitrite solution (2000 mg/L): In a 1 L volumetric flask dissolve 9.86 g sodium nitrite ($NaNO_2$) or 12.14 g of potassium nitrite (KNO_2) in approximately 800 mLs water. Add 2 mLs of chloroform. Dilute to the mark and invert three times. Refrigerate. This solution is stable for six months.

8.2.3 Intermediate stock standard (20 mg/L): To prepare the intermediate standard of both the nitrate and nitrite solutions, dilute 10 mLs of the respective 2000 mg/L stock solution to 1 L volumetrically. Prepare fresh daily.

8.2.4 Initial and Continuing Calibration Standards: Purchased from Hach Co. (Loveland, CO.) For nitrate, dilute 10.0 mL volumetrically to 100 mL to make a 1.2 mg/L calibration verification standard. For nitrite, dilute 0.5 mls of 250 ug/ml standard to 100 mls volumetrically to make a 1.25 mg/L calibration verification standard.

8.2.5 Matrix spiking solution: 20 mg/L Intermediate stock standard of the nitrate and of the nitrite stock respectively, 80 uL spiked into 8.0 mL of sample, constituting a 0.2 mg/L nitrate or nitrite spike.

9.0 CALIBRATION

9.1 Calibration standards: The calibration curve is prepared as follows for both NO_3 and NO_2:

Conc.of Std. to Dilute (mg/L)	mL of Std.	Final Volume (mL)	Final Concentration (mg/L)
20	10.0	100	2.0
20	4.0	100	0.8
20	2.0	100	0.4
2.0	5.0	100	0.1
0.40	10.0	100	0.04
0.40	5.0	100	0.02
BLANK	0	100	0

9.2 The working calibration range of this method is 0.02 to 2 mg/L.

9.3 The calibration procedure is performed as outlined in section 11.4.

9.4 Internal standards are not applicable to this method.

9.5 Acceptance criteria for the calibration requires the correlation coefficient of the absorbance vs. standard concentration to be at least 0.9975 or better.

9.6 Correlation coefficients are calculated in the Lachat software based on a linear least squares plot.

10.0 QUALITY CONTROL

10.1 Method detection limits are calculated based on the 40 CFR, Part 136, Appendix B, definition of method detection limit. A minimum of seven replicate standards prepared by referenced methods including all processing steps including distillation and digestion where appropriate. Method detection limits are calculated by multiplying the standard deviation of all replicate results by student t values listed in 40 CFR.

10.2 Practical quantitation limits for this method are the same as the method detection limits.

10.3 Daily analyzer performance tests are performed automatically. The Lachat software defaults to a diagnostics check on the valves and detectors when the instrument is powered up.

10.4 Matrix spikes are performed by pipetting 80 uL of stock nitrate or nitrite standard (Section 8.2.3) into 8.0 mL of sample to make a 0.2 mg/L nitrate or nitrite spike.

10.5 QC Check Samples consist of Initial and Continuing Calibration Verification standards (ICV and CCV, respectively), Section 8.2.

10.6 Surrogates are not applicable to this method.

10.8 QC SCHEDULES:

ACTIVITY:	FREQUENCY:
Initial Calibration Blank	Onset of every analysis
Continuing Calibration Blank	Every 10 samples, per analysis
Initial Calibration Verification	Onset of every analysis
Continuing Calibration Verification	Every 10 samples, per analysis
Matrix Spike	Every 10 samples, per batch
Duplicate	Every 10 samples, per batch

10.8.1 DEFINITIONS:
Batch: Every 10 samples leached, digested, or distilled, per day.
Analysis: Each calibration, each day.

11.0 SAMPLE PREPARATION AND INSTRUMENTAL PROCEDURES

11.1 Sample preparation is minimal since the analysis does not require digestion or disillation. Turbid samples may be filtered through a 4.5 micron filter membrane. Highly turbid or colored samples may reqire dilution to negate interferences.

11.2 Lachat Autoanalyzer Startup:
Swith the power switch located behind the system computer to the "ON" position. When the computer has completed the self-diagnostics, the CPU monitor will display the main menu, with the METHODS category illiminated. Install the chloride chemistry manifold onto the Lachat module, making proper connections to reagent lines as shown in the diagram. Place all reagent lines into proper reagent containers and adjust the tension levers on the peristaltic pump until the first "click" is heard. While the system is pumping reagents through the manifold, proceed with METHOD DOWNLOADING.

11.3 Method Downloading: To download the chemistry program to the system computer, observe the "METHODS" category illuminated at the CPU monitor, press "ENTER". The monitor will display "Download Method" and "Method Definition", using the arrow keys, highlight the "Download Method" and hit "ENTER". The method will now start downloading the nitrate/nitrite or nitrite chemistry to the system computer.

11.4 Calibration:

To set up std. curve on sampler: Obtain a vial for each std. Starting with the 2.0 mg/L std., fill the vial and place it in the standards rack in the front left spot. Continue with the other standards, in order of decreasing concentration. The computer monitor will display "EDIT". Using the arrow keys highlight "SUBMIT" and hit "ENTER". "SUBMIT CURRENT TRAY" will be highlighted. Using the arrow keys, highlight "CALIBARATE NOW", and hit "ENTER". The screen will give the prompt, "SHOULD A CALIBRATION BE STARTED NOW?", hit "Y". The Lachat will begin calibrating.

When the calibration is complete, the system will indicate whether the pass/fail criteria has been met by displaying "CALIBRATION FAILED!" or "CALIBRATION APPROVED". Note: Analysis cannot proceed until the calibration is approved. Once approved, move the cursor on the monitor to highlight "EDIT", then hit "ENTER".

11.5 Analyzing Samples: After the "EDIT" choice has been made, the new screen will change, move the cursor to "IDENTIFICATION' and hit "ENTER". Sample identifications may be entered here in the order of desired analysis. Each entry will be assigend a unique position in the sample tray. When all entries are complete, type a double period (..) in the location immediatley following the last sample to indicate the end of that tray. Hit "ESCAPE" and move the cursor to the "SUBMIT" category, hit "ENTER". The follwoing screen will indicate how many sample entries are in the computer. It will also ask "SHOULD THE TRAY BE SUBMITTED NOW?" Y/N? Analysis of the samples will now begin.

12.0 CALCULATIONS

12.1 ICV,and CCV Recovery:

$$\frac{\text{Actual mg/L}}{\text{1.2 mg/L (for nitrate) 1.25 for nitrite}} \times 100 = \% \text{ Recovery}$$

12.2 Spike Recovery:

$$\frac{\text{(Spiked sample + sample conc., mg/L) - (sample conc., mg/L)}}{0.2 \text{ mg/L}} \times 100 = \% \text{ Recovery}$$

12.3 Method blank, Initial Calibration and Continuing Calibration blanks:
Not to exceed 0.02 mg/L (MDL).

12.4 Relative Percent Difference (RPD):

$$\left[\frac{\text{(Sample 1 - Sample 2)}}{\text{avg., sample 1 \& 2}} \right] \times 100 = \% \text{ RPD}$$

12.5 Sample calculations are performed by computer software (see Sec. 9.6).

13.0 ACCEPTANCE CRITERIA NITRATE/NITRITE ANALYSIS:

ICB, CCB	< 0.02 mg/L
ICV, CCV	85% - 115% Recovery
Matrix spike	75% - 125% Recovery
Duplicates	± 20 % or
	± 1 mg/L (MDL) if sample conc. < 5 x MDL

14.0 REPORTING OF RESULTS

Practical Quantitation Limits: Same as MDL.
Units of Measure: mg/L
Significant Figures: 2 figures
Data Reporting:
Below quantitation limit: BQL

QC information included in report:
Level 1 QC: Blank values.
Level 2 QC: Blank values, Duplicate data (%RPD)
Level 3 QC: Blank values, Duplicate data (%RPD)
Matrix spike recoveries, specific to client's samples.

15.0 SUPPLEMENTAL DOCUMENTS

Lachat nitrate + nitrite, nitrite Method # 10-107-04-1-C.

16.0 REFERENCES

EPA Method 353.2.

17.0 SUBSTANTIVE REVISIONS

17.1 Amendments to Sections 2.1 and 2.2. Amendments made to Sections 3.1, 3.2 and 3.3. Amendment to Section 8.1.1. Revisions to Sections 8.2.1, 8.2.2 and 8.2.3. Revision to Section 9.1. Revisions to Sections 11.1, 11.2, 11.3, 11.4 and 11.5.

31. IEA SOP FOR AMMONIA ANALYSIS

1.0 APPROVALS

The signatures of the following individuals indicate that this SOP is complete and meets the requirements specified in corporate document # QAS00200.NET. In addition, it signifies that the content meets the specifications of the referenced "Test Code".

Laboratory Director

Quality Assurance Manager

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PROPRIETARY DOCUMENT

2.0 SCOPE AND APPLICATION

2.1 This procedure is applicable to the measurement of ammonia-nitrogen in drinking, surface and saline waters, domestic and industrial wastes.

2.2 This procedure is applicable to Test Code Amm_01 for ammonia analysis in water.

3.0 SUMMARY OF METHOD

3.1 The ammonia is determined potentiometrically using an ion selective electrode and a pH meter having an expanded millivolt scale or a specific ion meter.

3.2 The ammonia electrode uses a hydrophobic gas-permeable membrane to separate the sample solution from an ammonium chloride internal solution. Ammonia in the sample diffuses through the membrane and alters the pH of the internal solution, which is sensed by a pH electrode. The constant level of chloride in the internal solution is sensed by a chloride selective ion electrode which acts as the reference electrode.

3.3 This procedure is based on EPA Method 350.3 and Standard Method 4500.

3.4 This procedure deviates from EPA method 350.3 in the follow way:

A solution of 50 mL of sample and 5 mL of ISA pH adjusting solution is used for the pH measurement instead of 100 mL of sample and 1 mL 10 N NaOH.

3.5 This procedure deviates from Standard Method 4500 in the following way:

10 mL of borate buffer is added to the distillation flask instead of 25 mL.

4.0 INTERFERENCES

4.1 Volatile amines act as a positive interference. Mercury interferes by forming a strong complex with ammonia. The samples therefore cannot be preserved with mercuric chloride.

5.0 SAFETY

5.1 Consult Material Safety Data Sheets (MSDS) for precautions and proper handling of the ISA Buffer.

6.0 SAMPLE CONTAINERS, COLLECTION AND PRESERVATION

6.1 Sample containers: Containers may be either glass or plastic, thoroughly rinsed and dried before use and are not to be reused.

6.2 Sample Collection: Samples are collected into a plastic or glass container to which 2-3 drops of sulfuric acid has been added. Samples are cooled and maintained at 4 degrees Celsius and shipped to the laboratory for analysis.

6.3 The holding time for ammonia analysis is 28 days from sampling.

6.4 If the EPA-specified holding time of 28 days is exceeded, replenishment is requested.

7.0 APPARATUS AND MATERIALS

7.1 APPARATUS:

7.1.1 Electrometer (pH meter) with expanded mV scale or specific ion meter.

7.1.2 Ammonia ion selective electrode, such as Orion Model 95-10 or EIL Model 8002-2

7.1.3 Magnetic stirrer, thermally insulated, and Teflon coated stirring bar.

7.1.4 50 mL graduated cylinder

7.1.5 100 mL glass beaker

7.1.6 Plastic rinse bottle, with deionized water.

7.1.7 Magnetic stir bar retriever.

7.1.8 100 mL volumetric flasks, 4.

7.1.9 10.0 mL volumetric pipets, 5

7.1.10 Oxford pipetter or equivalent, with tips.

8.0 REAGENTS AND STANDARD PREPARATION

8.1 Reagents:

8.1.1 Reagent water, 16.6 megohm or greater, deionized.

8.1.2 Ammonia pH adjusting ISA buffer (Orion Cat# 951211).

8.1.3 Borate buffer solution: Add 88 mL 0.1N NaOH solution to 500 mL approximately 0.025M sodium tetraborate ($Na_2B_4O_7$) solution (9.5 grams $Na_2B_4O_7$*10 H2O) and dilute to 1.0 L.

8.1.4 Sulfuric acid, 0.04N: dilute 1.0 mL conc. H_2SO_4 to 1.0 L.

8.2 **Standard solutions:**

8.2.1 Stock Ammonia 25 N Standard, 1000 mg/L (Orion# 951007).

8.2.2 Ammonia nitrogen standard (ICV,LCS), (Hach Co., Loveland CO., cat# 1891_11 18cy): 1.0 mg/L.

9.0 CALIBRATION

9.1 Calibration standards are prepared by serial dilution in the following manner:

mg/L std to be diluted	mL to dilute to 100.0 mL	final Conc. (mg/L)
1000	10.0	100.0
100	10.0	10.0
10	10.0	1.0
1.0	10.0	0.1

9.2 The working range of the calibration is 0.1 to 100 mg/L NH_3-N

9.3 The calibration is performed by submerging the ammonia ion-selective electrode into each standard, beginning with the 0.1 ppm standard as described in section 9.1. Press the STDBY key. While stirring, add 5 mL of ISA buffer. Observe the multi-meter display until the signal stabilizes approx. 0.1 mV within 10 - 20 seconds. Record the voltage. Repeat the above procedure with the remaining standards. Enter the log of the concentration in mg/L for "x" and the mV reading in "y" into a calculator with a linear regression function.

9.4 Example calculations with typical data would be as follows:

Std. Conc.(mg/L)	mV	Log(Conc)
0.10	100.0	-1.00
1.0	43.7	0.00
10.0	-16.9	1.00
100.0	-76.5	2.00

Correlation Coefficient (r) = -0.99988

9.5 Internal standards are not appliacble to this method.

9.6 Acceptance criteria for the calibration requires that the correlation coefficient be 0.995 or greater.

10.0 QUALITY CONTROL

10.1 Method detection limits are calculated based on the 40 CFR, Part 136, Appendix B, definition of method detection limit. A minimum of seven replicate standards are prepared by referenced methods including all processing steps including distillation and digestion where appropriate. Method detection limits are calculated by multiplying the standard deviation of all replicate results by student t values in listed in 40 CFR.

10.2 Practical quantitation limits for this method are the same as the method detection limits.

10.3 Daily performance tests consist of noting and recording the correlation coefficient of the calibration curve.

10.4 Matrix spikes are performed by pipetting 1.0 mL of stock ammonia standard (section 8.2.1) into 50 mL. This constitutes a matrix spike concentration of 20 mg/L NH_3-N.

10.5 QC Check Samples consist of Initial and Continuing Calibration Verification standards (ICV and CCV, respectively).

10.6 Surrogates are not applicable to this method.

10.7 QC SCHEDULES:

ACTIVITY:	FREQUENCY:
Initial Calibration Verification	Onset of every analysis
Initial Calibration Blank	Onset of every analysis
Preparation blank	Every 20 samples, per batch
Continuing Calibration Verification	Every 10 samples, per analysis
Continuing Calibration Blank	Every 10 samples, per analysis
Matrix Spike	Every 20 samples, per batch
Duplicate	Every 10 samples, per analysis

10.7.1 DEFINITIONS:
Batch: Every 20 samples leached, digested, or distilled, per day.
Analysis: Each calibration, each day.

11.0 SAMPLE PREPARATION AND INSTRUMENTAL PROCEDURES

11.1 Instrumental Conditions: The ammonia ion selective electrode is a maintenance free, solid state electrode. The reference electrode is a single junction, sleeve type electrode. Electrolyte is added through the side opening near the top of the reference electrode.

11.2 **DISTILLATION:** To a sample volume of 500 mL in a 1000 mL boiling flask add 25 mL borate buffer (section 8.1.3) and adjust to a pH of 9.5 with 6N NaOH using a pH meter. Collect the distillate in a 500 mL erlenmeyer flask containing 50 mL plain sulfuric acid solution (section 8.1.4). Collect at least 200 mL of the distillate. Dilute the distillate to 500 mL with water.

Note: Distillation is not required for every sample analyzed. Samples which are regulated wastewaters (NPDES) and samples containing large amounts of solids may require distillation. NPDES samples need to be distilled only once per year per sample matrix. If comparison of distilled vs. non distilled results indicates little or no difference then distillation may be omitted.

11.3 Sample measurement: Measure 50 mL of sample into a 150 mL beaker. Immerse the ammonia ion-selective electrode in the sample. With constant stirring, add 5.0 mL ISA pH adjusting buffer. Allow the potential to stabilize, record the measurement. Sample calculations are described in section 12.5.

12.0 CALCULATIONS

12.1 ICV and CCV Recovery:

$$\frac{\text{Actual mg/L}}{\text{Theor. mg/L}} \times 100 = \%\ \text{Recovery}$$

12.2 Spike Recovery:

$$\frac{(\text{Spiked sample} + \text{sample conc., mg/L}) - (\text{sample conc., mg/L})}{20.0\ \text{mg/L}} \times 100 = \%\ \text{Recovery}$$

12.3 Method blank, Initial Calibration and Continuing Calibration blanks: Not to exceed 0.1 mg/L (MDL)

12.4 Relative Percent Difference:

$$\left[\frac{\text{(Sample 1 - Sample 2)}}{\text{avg., sample 1 \& 2}} \right] \times 100 = \% \text{ RPD}$$

12.5 Sample concentration determination from mV probe readings:

First the curve is entered into the calculator as described under section 9.0. Then for each sample to be determined the mV reading is entered as "y" and the "x" value for that point on the curve is determined by the calculator. This "x" value is the log of the concentration of ammonia in mg/L, therefore the concentration is 10 to the "x" power. An example follows.

Using the curve data from section 9.6 a sample reading of -55.7 would yield a "x" value of 1.657.

"y" = -55.7, therefore "x" = 1.657, Conc(NH_3) = 10^x = $10^{1.657}$ = 45.39 mg/L

13.0 ACCEPTANCE CRITERIA

ICB, CCB	< 0.1 mg/L
ICV, CCV	85% - 115% Recovery
Matrix spike	75% - 125% Recovery
Duplicates	$\pm$ 20 % or $\pm$ 0.1 mg/L (MDL) if sample conc. < 5 x MDL

14.0 REPORTING OF RESULTS

Practical Quantitation Limits: Same as MDL.
Units of Measure: mg/L
Significant Figures: 2 figures
Data Reporting:
Below quantitation limit: BQL
QC information included in report:
Level 1 QC: Blank values.
Level 2 QC: Blank values, Duplicate data (%RPD)
Level 3 QC: Blank values, Duplicate data (%RPD), and Matrix spike recoveries, specific to client's samples.

15.0 SUPPLEMENTAL DOCUMENTS

15.1 Standard Methods for the Examination of Water and Wastewater, 16th Edition, Method 417A, (1985).

16.0 REFERENCES

17.0 SUBSTANTIVE REVISIONS

17.1 Section 2.2 revised: Test Code Amm_02 omitted. Addenda to Section 3.0: Sections 3.3 - 3.5 added. Amendment to 8.1.1: 18 megohm D.I. water changed to 16.6. Addenda to 9.3: Calculator calculation added. Section 9.6 revised: Example calculation revised to incorporate calculator use. Section 10.3 amended: Calculation of the ICV and CCV and recording the slope between the top three standards are omitted. Addenda and amendment to Section 10.7: Preparation blank analyzed every 20 samples instead of 10. Duplicate and frequency added. Duplicate analyzed every 10 samples. Addendum to Section 12.0: Section 12.5 added. Amendment to Section 13.0: MDL reference in duplicate acceptance criteria changed to 0.01 mg/L. Section 16.1 omitted.

32. IEA SOP FOR CHLORIDE IN WATER

1.0 APPROVALS

The signatures of the following individuals indicate that this SOP is complete and meets the requirements specified in corporate document # QAS00200.NET. In addition, it signifies that the content meets the specifications of the referenced "Test Code".

Laboratory Director

Quality Assurance Manager

UNCONTROLLED COPY
PROPRIETARY DOCUMENT

2.0 SCOPE AND APPLICATION

2.1 This automated method is applicable to drinking, surface, and saline waters, domestic and industrial wastes and soils. The applicable range is 1 to 100 mg Cl/L.

2.2 This method is applicable to Test Codes CL_01 and CL_02.

3.0 SUMMARY OF METHOD

3.1 Thiocyanate ion (SCN) is liberated from mercuric thiocyanate through sequestration of mercury by chloride ion to form un-ionized mercuric chloride. In the presence of ferric ion, the liberated SCN forms highly colored ferric thiocyanate in concentration proportional to the original chloride concentration.

3.2 This method is based on EPA method number 325.2 for Automated Ferricyanide AAI Chloride Analysis and is adapted for analysis using the Lachat QuikChem Autoanalyzer.

3.3 This method deviates from Method 325.2 in the following manner:

3.3.1 This method has been modified to accommodate the analysis of soils samples. Soil samples are leached in reagent water and then centrifuged or filtered prior to analysis.

4.0 INTERFERENCES

4.1 Substances which reduce iron (III) to iron (II) and mercury (III) to mercury (II). (e.g. sulfite, thiosulfate) and halides which form strong complexes with mercury (e.g. Br-, I-) may interfere.

4.2 Highly turbid or colored samples may require centrifugation, filteration or dilution prior to analysis.

4.3 Note: If any question of interferences arise, calibration curves should be prepared in water and in the suspected interfering matrix. If the two curves differ significantly, then there is interference, and the standards must be prepared in the interfering matrix instead of water.

5.0 SAFETY

5.1 The Chloride Color Reagent contains mercury, methanol and complexed cyanide. Safety glasses, lab coat and gloves are necessary to prevent contact with this solution. Waste reagent should be labelled hazardous and disposed of properly.

6.0 SAMPLE CONTAINERS, COLLECTION AND PRESERVATION

6.1 Sample containers: Containers may be either plastic or glass, thoroughly rinsed and dried before use and are not to be reused.

6.2 Sample Collection: Samples are collected without preservatives. Samples are packaged and sent to the laboratory and stored at 4 deg. C until analysis.

6.3 Sample preservation is not necessary for this analysis.

6.4 The holding time for chloride analysis is 28 days from sampling. If samples are delayed in transit or are received after the 28 day holding time, the client is notified and a resampling schedule is arranged.

7.0 APPARATUS AND MATERIALS

7.1 Lachat QuikChem Autoanalyzer.

7.1.1 Lachat Chloride chemistry manifold (Method # 10-117-07-1-B).

8.0 REAGENTS AND STANDARD PREPARATION

8.1 Reagents

8.1.1 Reagent Water, 18 megohm-cm, deionized.

8.1.2 Chloride Color Reagent (Sigma Diagnostics, cat # 955-6).

8.1.3 Sodium chloride, NaCl crystals.

8.1.4 Reagent receipt and preparation is documented in the Reagent Preparation and Receiving Logbook (Attachment I).

8.2 Standard Solutions Preparation

8.2.1 Calibration Standard Preparation

8.2.1.1 Chloride Stock Standard: Dry 4-5 grams of NaCl (Section 8.1.3) at 140 degrees Celsius for 2 hours. Dissolve 8.241 grams of dried NaCl to one liter volumetrically. The final concentration is 5000 mg/L.

8.2.1.2 Intermediate Chloride Stock Standard: Dilute 100 mL of the Chloride Stock Standard (Section 8.2.1.1) to 1 liter volumetrically. The final concentration is 500 mg/L.

8.2.1.3 The calibration curve is prepared from the dilution of the Intermediate Chloride Stock Standard (Section 8.2.1.2) as follows:

Conc. of Std. to be diluted (mg/L)	mL of standard	Final volume (mL)	Final concentration (mg/L)
500	20.0	100	100
500	10.0	100	50
500	2.0	100	10
500	1.0	100	5
500	0.20	100	1
BLANK	0	100	0

8.2.2 Initial and Continuing Calibration (ICV/CCV) Standard Preparation

8.2.2.1 Initial and Continuing Calibration Stock Standard is purchased from Hach Co. (Loveland, CO.) Cat # 183-11, 1000 mg/L.

8.2.2.2 Dilute 5.0 mL of the ICV/CCV stock standard (Section 8.2.2.1) volumetrically to 100 mL to prepare a 50 mg/L calibration verification standard.

8.2.3 All standard receipt and preparation is documented in the Standards Receipt and Preparation Logbook (Attachment II).

9.0 CALIBRATION

9.1 To set up Std Curve on sampler: Obtain a vial for each Std. starting with the 100 mg/L Std., fill the vial and place it in the Standards rack in the front left spot. Continue with the other Standards, in order of decreasing concentration. The computer monitor will display "EDIT". Using the arrow keys highlight "SUBMIT" and hit "ENTER". "Submit Current Tray" will be highlighted; using the arrow keys highlight "Calibrate Now", and "ENTER". The screen will give the prompt, "Should a calibration be started now?",hit "Y". The Lachat will begin calibrating.

Current Tray" will be highlighted; using the arrow keys highlight "Calibrate Now", and "ENTER". The screen will give the prompt, "Should a calibration be started now?",hit "Y". The Lachat will begin calibrating.

9.2 When the calibration is complete the system will indicate whether the pass/fail criteria has been met by displaying "calibration failed!" or "calibration approved". Note: Analysis cannot proceed until the calibration is approved. Once approved, move the cursor on the monitor to highlight "EDIT", then hit "ENTER".

9.3 The working calibration range of this method is 1 to 100 mg/L.

9.4 Acceptance criteria for the calibration requires the correlation coefficient of the absorbance vs. standard concentration to be at least 0.9975 or better.

9.5 Correlation coefficients are calculated in the Lachat software based on a least squares linear regression.

10.0 QUALITY CONTROL

10.1 Method detection limits are performed in accordance with the SOP for Conducting MDL Studies, IEA Doc# QAS02000.NET which is based on the 40 CFR, Part 136, Appendix B definition of method detection limit. A minimum of seven replicate standards prepared by referenced methods including all processing steps including distillation and digestion where appropriate. Method detection limits are calculated by multiplying the standard deviation of all replicate results by student t values listed in 40 CFR.

10.2 The practical quantitation limit for this method is 1.0 mg/L for waters and 10 mg/kg for soils.

10.3 Daily analyzer performance tests are performed automatically. The Lachat software defaults to a diagnostics check on the valves and detectors when the instrument is powered up.

10.4 Matrix spikes are performed by pipetting 400 uL of intermediate chloride stock standard (Section 8.2.1.2) into 7.6 mL for a final spike concentration of 26.32 mg/L. Matrix spikes are analyzed every 20 samples, per batch and must recover within 20% RPD or $\pm$ the MDL if the sample concentration is less than 5x the MDL.

10.5 Sample duplicates are performed every 10 samples, per analysis. Duplicate must recover within 20% RPD.

10.6 QC Check Samples consist of Initial and Continuing Calibration Verification standards (ICV and CCV, respectively), Section 8.2. The ICV is analyzed at the onset of each analysis and the CCV is analyzed every 10 samples, per anaysis. The ICV and CCV must

10.7 Initial Calibration Blanks (ICB) and analyzed at the onset of analysis and Continuing Calibration Blanks (CCB) are analyzed every ten samples, per analysis. ICB/CCBs must not exceed the PQL, 1.0 mg/L, for chloride.

10.8 Surrogates are not applicable to this method.

10.9 Internal standards are not applicable to this method.

10.10 QC SCHEDULES:

ACTIVITY:	FREQUENCY:
Initial Calibration Blank	Onset of every analysis
Continuing Calibration Blank	Every 10 samples, per batch
Initial Calibration Verification	Onset of every analysis
Continuing Calibration Verification	Every 10 samples, per analysis
Matrix Spike	Every 20 samples, per batch
Duplicate	Every 10 samples, per analysis

10.10.1 DEFINITIONS:
Batch: Every 20 samples leached or distilled, per day.
Analysis: Each calibration, each day.

11.0 SAMPLE PREPARATION AND INSTRUMENTAL PROCEDURES

11.1 Sample Preparation

11.1.1 10 grams of soil sample is leached in 100 mL reagent water for 30 minutes.

11.1.2 The supernatant is decanted and centrifuged and/or filtered through a glass fiber filter (GF-D grade or better). Further clarification is accomplished by filtration through 0.45 micron filter membrane. Highly turbid or colored samples may require dilution to negate interferences.

11.1.3 Water samples require no preparation.

11.2 Instrumental Procedures

11.2.1 Switch the power strip located behind the system computer to "on" position. When the computer has completed the self-diagnostics, the CPU monitor will display the main menu, with the METHODS category illuminated.

11.2.2 Install the chloride chemistry manifold onto the Lachat module, making proper connections to reagent lines.

11.2.3 Place all reagent lines into proper reagent containers and adjust the tension levers on the peristaltic pump until the first "click" is heard. Make sure that the carrier line is placed in reagent water and the color reagent pickup line is in the Chloride Color Reagnet (Section 8.12.). While the system is pumping reagents through the manifold, proceed with METHOD DOWNLOADING.

11.2.4 To download the chemistry program to the system computer, observe the "METHODS" category illuminated at the CPU monitor, press "ENTER". The monitor will display "Download Method" and "Method Definition", using the arrow keys, highlight the "Download Method" and hit "ENTER". The method will now start downloading the chloride chemistry to the system computer.

11.2.5 The instrument is now ready for calibration as in Section 9.0.

11.2.6 After the "EDIT" choice has been made (Section 9.2), the new screen will change, move the cursor to "IDENTIFICATION" and hit "ENTER".

11.2.7 Sample identifications may be entered here in the order of desired analysis. Each entry will be assigned a unique position in the sample tray. When all entries are complete type a double period (..) in the location immediately following the last sample to indicate the end of that tray.

11.2.8 Hit "ESC" and move the cursor to the "SUBMIT" category, hit "ENTER". The following screen will indicate how many sample entries are in the computer, it will also ask "Should the tray be submitted now?" Y / N ? Enter "Y". Analysis of the samples will now begin.

11.2.9 All analyses including calibration, samples and QC samples are documented in the instrument specific Lachat Runlog (Attachment III).

12.0 CALCULATIONS

12.1 ICV, and CCV Recovery:

$$\frac{\text{Actual mg/L}}{50\ \text{mg/L}} \times 100 = \%\ \text{Recovery}$$

12.2 Spike Recovery:

$$\frac{(\text{Spiked sample} + \text{sample conc., mg/L}) - (\text{sample conc., mg/L})}{26.32\ \text{mg/L}} \times 100 = \%\ \text{Recovery}$$

12.3 Relative Percent Difference (RPD):

$$\left[\frac{\text{(Sample 1 - Sample 2)}}{\text{avg., sample 1 \& 2}}\right] \times 100 = \% \text{ RPD}$$

12.4 Sample calculations are performed by computer software.

13.0 ACCEPTANCE CRITERIA CHLORIDE ANALYSIS:

13.1

ICB, CCB	< 1.0 mg/L
ICV, CCV	85% - 115% Recovery
Matrix spike	75% - 125% Recovery
Duplicates	± 20 % or
	± MDL if sample conc. < 5 x MDL

14.0 REPORTING OF RESULTS

14.1 Practical Quantitation Limits: 1.0 mg/L for waters and 10 mg/kg for soils.
Units of Measure: mg/L for waters and mg/kg for soils
Significant Figures: 2 figures
Data Reporting:
Below quantitation limit: BQL
QC information included in report:
Level 1 QC: Blank values, sampling COCs
Level 2 QC: Blank values, Duplicate data (%RPD), sampling COCs
Level 3 QC: Blank values, Duplicate data (%RPD), Matrix spike recoveries, specific to client's samples, sampling and laboratory COCs.

15.0 SUPPLEMENTAL DOCUMENTS

15.1 SOP for Conducting MDL Studies, IEA Doc# QAS02000.NET.

16.0 REFERENCES

16.1 EPA Methods for Chemical Analysis of Water and Wastes, Method 325.1, 1983.

16.2 Lachat QuickChem Chloride Method# 10-117-07-1-B.

17.0 SUBSTANTIVE REVISIONS

17.1 Amendments to Sections 3.2. and 3.3 to correct EPA method from 325.1 to 325.2. Addendum to Section 6.4 to clarify holding time. Section 8.1 amended from 18.0 to 16.6 mega-ohm d.i. reagent water. Section 12.4 amended to more clearly express absolute value.

17.0 SUBSTANTIVE REVISIONS

17.1 Amendments to Sections 3.2. and 3.3 to correct EPA method from 325.1 to 325.2. Addendum to Section 6.4 to clarify holding time. Section 8.1 amended from 18.0 to 16.6 mega-ohm d.i. reagent water. Section 12.4 amended to more clearly express absolute value.

17.2 Section 8.0 and Section 11.0 revised for clarification. Section 8.2.1.3 revised: 20 and 2 mg/L standards omitted as calibration points. Preparation of the 5 and 1 mg/L calibration standards corrected. Sections 10.5 and 10.7 added. Sections 10.2, 13.1 and 14.1 revised to reflect the correct PQL. (03/21/96)

Reagents Preparation and Receiving Log
Wet Chemistry Laboratory

Date	Analyst	Time	Reagent #	Reagent Name	Preparation Method	Expiration Date
			R			
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Secondary Review By:____________________**Date:**___________

Attachment I

Standards Preparation and Receiving Log
Inorganics Laboratory

Date	Analyst	Time	Standard #	Standard Name	Vendor	Preparation Method	Expiration Date

Secondary Review By:_____ **Date:**_____

Attachment II

Attachment III

Lachat Runlog

Analyst: _____ Instrument-#*

Preparation Log: _____

Filename: _____

Sample ID.	Sample Amount (g,ml)	Leachate/Dig Volume		Dilution		DF	Analysis mg/l,ug/l	Result mg/l,mg/kg	Comments
		g/ml	Final Volume	Vi	Vf				

Secondary Review By:_____ Date:_____

33. IEA SOP FOR FLUORIDE ANALYSIS

1.0 APPROVALS

The signatures of the following individuals indicate that this SOP is complete and meets the requirements specified in corporate document # QAS00200.NET. In addition, it signifies that the content meets the specifications of the referenced "Test Code".

Laboratory Director

Quality Assurance Manager

UNCONTROLLED COPY
PROPRIETARY DOCUMENT

2.0 SCOPE AND APPLICATION

2.1 This procedure is applicable to the measurement of fluoride in drinking, surface and saline waters, domestic and industrial wastes.

2.2 This procedure is applicable to Test Code Fl_01 for fluoride analysis in water.

3.0 SUMMARY OF METHOD

3.1 Fluoride is determined potentiometrically using an ion selective electrode in conjunction with a standard single junction sleeve-type reference electrode and a pH meter having an expanded millivolt scale or a specific ion meter. The fluoride electrode consists of a lanthanum fluoride crystal across which a potential is developed by fluoride ions. The cell is represented by a Ag/AgCl junction.

3.2 This procedure is based on EPA Method 340.2.

3.3 This procedure does not deviate from EPA Method 340.2.

4.0 INTERFERENCES

4.1 Extreme pHs interfere; sample pH should be between 5 and 9. Polyvalent cations of Si^{+4}, Fe^{+3}, and Al^{+3} interfere by forming complexes with fluoride. The degree of interference depends upon the concentration of the complexing cations, the concentration of fluoride and the pH of the sample. The addition of a pH 5.0 buffer containing a strong chelating agent (Section 8.1.2) preferentially complexes aluminum (the most common interference), silicon and iron and eliminates the pH problem.

5.0 SAFETY

5.1 Consult Material Safety Data Sheets (MSDS) for precautions and proper handling of the TISAB III Buffer.

6.0 SAMPLE CONTAINERS, COLLECTION AND PRESERVATION

6.1 Sample containers: Containers may be either glass or plastic, thoroughly rinsed and dried before use and are not to be reused.

6.2 Sample Collection: Samples are collected into plastic or glass containers. Samples are cooled and maintained at 4 degrees Celsius and shipped to the laboratory for analysis.

6.3 The holding time for fluoride analysis is 28 days from sampling.

6.4 If the EPA-specified holding time of 28 days is exceeded, a request for sample replenishment is requested.

7.0 APPARATUS AND MATERIALS

7.1 Electrometer (pH meter) with expanded mV scale or specific ion meter.

7.2 Fluoride ion selective electrode, such as Orion Model 94-09 or EIL Model 8002-2.

7.3 Reference electrode, single junction, sleeve-type, such as Orion No.90-01.

7.4 Magnetic stirrer, thermally insulated, and Teflon coated stirring bar.

7.5 50 mL graduated cylinder.

7.6 100 mL glass beaker.

7.7 Plastic rinse bottle, with deionized water.

7.8 Magnetic stir bar retriever.

7.9 100 mL volumetric flasks (4).

7.10 10.0 mL volumetric pipets (5).

7.11 Oxford pipetter or equivalent, with tips.

8.0 REAGENTS AND STANDARD PREPARATION

8.1 Reagents:

8.1.1 Reagent water, 18 megohm-cm, deionized.

8.1.2 Orion pH adjusting buffer TISAB III (Orion # 940911).

8.1.3 Reagent receipt and preparation is documented in the Reagent Preparation and Receiving Logbook (Attachment I).

8.2 Standard solutions:

8.2.1 Stock Fluoride Solution: 0.1M NaF (Orion 940906), equivalent to 1900 mg/L.

8.2.2 Fluoride standard (ICV, LCS), (Hach Co., Loveland CO., cat# 405_02): 0.20 mg/L.

8.2.3 Calibration standards: Prepare the calibration curve by successive (serial) dilution of the stock fluoride solution as follows.

8.2.3.1 Dilute 5.0 mL of 1900 mg/L stock fluoride (Section 8.2.1) to 100 mL volumetrically to prepare the 95 mg/L standard.

8.2.3.2 Dilute 10 mL of 100 mg/L standard (Section 8.2.3.1) to 100 mL volumetrically to prepare the 9.5 mg/L standard.

8.2.3.3 Dilute 10.0 mL of the 10 mg/L standard (Section 8.2.3.2) to 100 mL volumetrically to prepare the 0.95 mg/L standard.

8.2.3.4 Dilute 10.0 mL of the 1.0 mg/L standard (Section 8.2.3.3) to 100 mL volumetrically to prepare the 0.095 mg/L standard.

8.2.4 All standard receipt and preparation is documented in the Standards Receipt and Preparation Logbook (Attachment II).

9.0 CALIBRATION

9.1 The working range of the calibration is 0.095 to 95 mg/L fluoride.

9.2 The calibration is performed by adding 5 mL of the TISAB III buffer to 50 mL of standard, then submerging the fluoride ion-selective electrode into each standard, beginning with the 0.095 mg/L standard. Depress the STDBY key, observe the multimeter display until the signal stabilizes approx. 0.1 mV within 10 - 20 seconds. Record the voltage in the Potentiometric Analysis of Fluoride Logbook (Attachment III). Repeat the above procedure with the remaining standards.

9.3 Acceptance criteria for the calibration requires the difference (slope) in mV readings to be 56.0 mV $\pm$ 1 mV between each of the three highest standards.

10.0 QUALITY CONTROL

10.1 Method detection limits are performed in accordance with the SOP for Conducting MDL Studies, IEA Doc# QAS02000.NET which is based on the 40 CFR, Part 136, Appendix B definition of method detection limit. A minimum of seven replicate standards prepared by referenced methods including all processing steps including distillation and digestion where appropriate. Method detection limits are calculated by multiplying the standard deviation of all replicate results by student t values listed in 40 CFR.

10.2 The practical quantitation limit for this method is 0.1 mg/L.

10.3 Daily performance tests consist of noting and recording the slope of the calibration curve between the top three standards and calculation of laboratory control standards (ICV, CCV).

10.4 Sample duplicates are performed every 10 samples, per batch. Duplicates must meet 20% RPD or $\pm$ MDL if the sample concentration is less than 5x the MDL.

10.5 Matrix spikes are performed by pipetting 1.0 mL of stock fluoride solution (Section 8.2.1) into a 50 mL aliquot of sample. This constitutes a matrix spike concentration of 38.0 mg/L fluoride. Matrix spikes must recover within 75-125% of the true value.

10.6 QC Check Samples consist of Initial and Continuing Calibration Verification standards (ICV and CCV, respectively). The ICV and CCV must recover within 85-115% of the true value.

10.7 Surrogates are not applicable to this method.

10.8 Internal standards are not applicable to this method.

10.9 QC SCHEDULES:

ACTIVITY:	FREQUENCY:
Initial Calibration Verification	Onset of every analysis
Initial Calibration Blank	Onset of every analysis
Preparation blank	Every 10 samples, per batch
Continuing Calibration Verification	Every 10 samples, per analysis
Continuing Calibration Blank	Every 10 samples, per analysis
Sample Duplicate	Every 10 samples, per batch
Matrix Spike	Every 10 samples, per batch

10.9.1 DEFINITIONS:

Batch: Every 20 samples leached, or distilled, per day.
Analysis: Each calibration, each day.

11.0 SAMPLE PREPARATION AND INSTRUMENTAL PROCEDURES

11.1 Instrumental Conditions: The fluoride ion selective electrode is a maintenance free, solid state electrode. The reference electrode is a single junction, sleeve type electrode. Electrolyte is added through the side opening of the reference electrode near the top.

11.2 Sample Preparation: Measure 50 mL of sample into a 150 mL beaker. Add 5.0 mL of TISAB lll buffer.

11.3 Sample measurement: Immerse the fluoride ion-selective electrode in the solution in the beaker as prepared in Section 11.2. Stir constantly. Allow the potential to stabilize, record the measurement. Sample calculations are described in Section 12.4.

11.4 All analyses including calibration, samples and QC samples are documented in the Potentiometric Analysis of Fluoride Logbook (Attachment III).

12.0 CALCULATIONS

12.1 ICV,and CCV Recovery:

$$\frac{\text{Actual mg/L}}{\text{Theor. mg/L}} \times 100 = \%\ \text{Recovery}$$

12.2 Spike Recovery:

$$\frac{\text{(Spiked sample + sample conc., mg/L) - (sample conc., mg/L)}}{37.2 \text{ mg/L}} \times 100 = \% \text{ Recovery}$$

12.3 Relative Percent Difference:

$$\left[\frac{\text{(Sample 1 - Sample 2)}}{\text{avg., sample 1 \& 2}}\right] \times 100 = \% \text{ RPD}$$

12.4 Example calculations with typical data would be accomplished as follows:

Std. Conc.(mg/L)	mV	Slope (difference)
0.10	100.0	
		NA
1.0	43.7	
		56.3
10.0	-16.9	
		60.0
100.0	-76.5	

The equation for samples and control standards (ICV, CCV) is a logarithmic correlation and is described below:

$$\text{mg/L fluoride} = 10^{(\text{mV std.} - \text{mV sample})/(\text{slope})} \times \text{mg/L std}$$

where:

mV std. = the cell potential (in mV) of the standard closest to the sample.

mV sample = the cell potential (in mV) of the sample.

"mg/L" = the true concentration (mg/L) of the standard used for the "mV Std" used above.

13.0 ACCEPTANCE CRITERIA

13.1

Method Blank, ICB, CCB	0.1 mg/L
ICV, CCV	85% - 115% Recovery
Matrix spike	75% - 125% Recovery
Duplicates	$\pm$ 20% or $\pm$ MDL if sample conc. $<$ 5 x MDL

14.0 REPORTING OF RESULTS

14.1 Practical Quantitation Limits: 0.1 mg/L
Units of Measure: mg/L
Significant Figures: 2 figures
Below quantitation limit: BQL

14.2 QC information included in report:
Level 1 QC: Blank values.
Level 2 QC: Blank values, Duplicate data (%RPD), Sampling COC
Level 3 QC: Blank values, Duplicate data (%RPD), Sampling and Laboratory COC, Matrix spike recoveries specific to client's samples.

15.0 SUPPLEMENTAL DOCUMENTS

15.1 SOP for Conducting MDL Studies, IEA Doc# QAS02000.NET.

16.0 REFERENCES

16.1 EPA Methods for Chemical Analysis of Water and Wastes, Method 340.2, 1983.

16.2 Handbook of Electrode Technology, Orion Research, 1982.

17.0 SUBSTANTIVE REVISIONS

17.1 Section 6.3 corrected for reference to fluoride. Section 8.1.1 corrected for units. Sections 8.1.3, 9.1.1 and 11.5 added to include logbook references. Section 10.1 and 15.1 added to include SOP reference. Section 9.6 moved to Section 12.4. Section 16.1 revised to reflect proper reference format. Section 10.1 corrected for reference to Appendix B. COCs added to QC reports for level 2 and 3.

17.2 Section 8.3 revised for clarification and correction of standard concentrations. Section 9.1 revised to correct the working range calibration in accordance with the correction of the standard concentrations. Section 10.2 and 14.1 revised to correct the PQL. Sections 10.3 added and 10.8 revised to describe sample duplicates. (Revised 03/21/96)

Attachment I

Reagents Preparation and Receiving Log
Wet Chemistry Laboratory

Date	Analyst	Time	Reagent #	Reagent Name	Preparation Method	Expiration Date
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Secondary Review By:____________________ Date:__________

Attachment II

Standards Preparation and Receiving Log
Inorganics Laboratory

Date	Analyst	Time	Standard #	Standard Name	Vendor	Preparation Method	Expiration Date

Secondary Review By:_____ **Date:**_____

Attachment III

POTENTIOMETRIC ANALYSIS

Method Name:_____ Date:_____ Analyst:_____

DAILY STANDARDS

Standard ID	Stock Lot#	Stock Vol.	Stock Conc.	Diluent	Final Vol.	Final Conc.
ICV						
LCS/QC Check						
Working Standard						

STANDARD CURVE Samples/Standards Final Volume Required:_____ mL

Standard ID	Std Concentration	mV Reading

Curve Correlation Coefficient (must be $\geq$ 0.995):_____ Slope:_____

Sample ID	Volume Used (mLs)	mV Reading	Curve Conc. mg/L	Final Conc. (mg/L)	QC Sample (Recovery/RPD)
ICV					
ICB					
LCS/QC CHECK					
CCV					
CCB					

CALCULATIONS:

$RPD = \frac{|(\text{Sample} - \text{Sample Dup.})|}{\text{Avg., Sample} + \text{Dup.}} \times 100$

$\% \text{ Recovery} = \frac{\text{Spiked Sample Result} - \text{Sample Result}}{\text{Spike Concentration}} \times 100$

Concentration =

Comments:_____

_____

Secondary Review By:_____ Date:_____

34. IEA SOP FOR SULFATE IN WATER

1.0 APPROVALS

The signatures of the following individuals indicate that this SOP is complete and meets the requirements specified in corporate document # QAS00200.NET. In addition, it signifies that the content meets the specifications of the referenced "Test Code".

UNCONTROLLED COPY
PROPRIETARY DOCUMENT

Laboratory Director

Quality Assurance Manager

2.0 SCOPE AND APPLICATION

2.1 This method is applicable to drinking and surface waters, domestic and industrial wastes. Sulfate ion (SO_4) is precipitated in acid medium containing barium chloride ($BaCl_2$) will react and form barium sulfate($BaSO_4$) crystals. The $BaSO_4$ suspension is then determined spectrophotometrically and compared to a standard curve.

2.2 This method is applicable to Test Codes SO4_01 and SO4_02.

3.0 SUMMARY OF METHOD

3.1 Sulfate ion is converted to barium sulfate suspension under controlled conditions. The resulting turbidity is determined by a spectrophotometer and compared to a curve prepared from standard sulfate solutions.

3.2 This method deviates from EPA method 375.4 by the following:

3.2.1 Sodium sulfate has been substituted for sodium carbonate in Section 8.1.7 as the primary standard with sulfuric acid.

4.0 INTERFERENCES

4.1 Color and/or suspended matter in vast amounts will interfere. Running a blank from which the barium chloride has been omitted will correct the problem.

4.2 Silica in concentrations over 500 mg/L will also interfere.

5.0 SAFETY

5.1 Consult the Material Safety Data Sheet (MSDS) for Barium Chloride precautions.

6.0 SAMPLE CONTAINERS, COLLECTION, AND PRESERVATION

6.1 Samples are to be collected in new 500 mL plastic bottles. Sample bottles are not to be reused.

6.2 No field preservation is required.

6.3 Samples are cooled to 4 °C during shipment to the laboratory and until analysis.

6.4 Samples have 28 day holding time from date of collection. If samples are delayed in transit or are received after the 28 day holding time, the client is notified and a resampling schedule is arranged.

7.0 APPARATUS AND MATERIALS

7.1 Magnetic stirrer.

7.2 Stopwatch.

7.3 Measuring spoon, capacity 0.2 to 0.3 mL.

7.4 200 mL beaker.

7.5 Stir bar.

7.6 Spectrophotometer, 420 mm wavelength.

7.7 5 cm pathlength cell.

8.0 REAGENTS AND STANDARD PREPARATION

8.1 Reagents

8.1.1 Barium chloride, $BaCl_2$, crystals, 20 to 30 mesh.

8.1.2 Concentrated hydrochloric acid.

8.1.3 Reagent water 16.6 megohm-cm, or higher, deionized.

8.1.4 95% ethanol or isopropanol.

8.1.5 Sodium chloride, NaCl, crystals.

8.1.6 Glycerol.

8.1.7 Anhydrous sodium sulfate, Na_2SO_4, crystals.

8.1.8 Conditioning reagent: Place 30 mL conc. HCl, 300 mL reagent water, 100 mL 95% ethanol or isopropanol and 75 g NaCl in solution in a container. Add 50 mL glycerol and mix.

8.2 Standards

8.2.1 Calibration Standard Preparation

8.2.1.2 Sulfate Stock Solution: Na_2SO_4: Dissolve 1.479 g anhydrous Na_2S0_4 (Section 8.1.7) in reagent water in a liter volumetric flask and dilute to volume with reagent water. The final stock concentration is 1000 mg/L.

8.2.1.2 Intermediate Stock Standard: 10 mL of 1000 mg/L sulfate stock standard (Section 8.2.1.2) is diluted to 100 mL volumetrically for a final concentration of 100 mg/L.

8.2.1.3 Calibration Standards: Prepare calibration standards 0, 5, 10, 15, 20 and 25 mg/L in 100 mL volumetric flasks using 0, 5, 10, 15, 20 and 25 mL of the 100 mg/L intermediate stock standard, respectively.

8.2.2 Initial Calibration Verification/Continuing Calibration Verification standard is Hach Co. Sulfate standard, 50 mg/L, Cat. #2578-11.

8.2.3 All standard receipt and preparation is recorded in the Standards Receipt and Preparation Logbook (Attachment II).

9.0 CALIBRATION STANDARDS

9.1 The working calibration range of the method is 0 mg/L to 25 mg/L.

9.2 The calibration is performed by filling the 5.0 cm cell with the 0 mg/L standard and placing in the cell holder of the spectrophotometer. When the absorbance reading stabilizes, press the "2nd function" key, then press "zero" key, and "enter". This establishes the origin of the calibration plot. Repeat the above procedures by placing each subsequent standards into the cell and recording the absorbance reading from the instrument.

9.3 The acceptance criteria for the calibration requires the correlation coefficient of the absorbance vs. standard concentration to be at least 0.9975 or better.

9.4 Correlation coefficients are calculated based on a absorbance vs. concentration plot based on the equation for a linear regression calculation.

10.0 QUALITY CONTROL

10.1 Method detection limits are performed in accordance with the SOP for conducting MDL Studies, IEA Doc# QAS02000.NET which is based on the 40 CFR, Part 136, Appendix B definition of method detection limit. A minimum of seven replicate standards prepared by referenced methods including all processing steps including distillation and digestion where appropriate. Method detection limits are calculated by multiplying the standard deviation of all replicate results by student t values in listed in 40 CFR.

10.2 The practical quantitation limit for this method is 2.0 mg/L.

10.3 Daily Analyzer performance tests are performed automatically by the spectrophotometer. A diagnostic check is performed by the spectrophotometer to verify that the light source is functioning properly.

10.4 Sample duplicates are performed every 10 samples, per batch. Duplicates must recover within 20% RPD or $\pm$ MDL if the sample concentration is less than 5x the MDL.

10.5 Matrix spikes are performed by pipetting 1.0 mL of 1000 mg/L stock standard (Section 8.2.1.2) into 99 mL of sample to obtain a spike concentration of 10 mg/L. Matrix spikes are performed every 20 samples, per batch and must recover within 75-125% of the true value.

10.6 QC Check Samples consist of Initial and Continuing Calibration Verification standards (ICV and CCV, respectively). The ICV and CCV (Section 8.2.2) must recover within 85-115% of the true value, except for North Carolina Wastewater samples which must recover within 90-110% of the true value.

10.7 Initial Calibration Blanks are analyzed at the onset of analysis and Continuing Calibration Blanks are analyzed every 10 samples, per analysis. ICB/CCBs must be less than the PQL, 2.0 mg/L, for sulfate.

10.8 Surrogates are not applicable to this method.

10.9 Internal standards are not employed in this method.

10.10 QC SCHEDULES:

ACTIVITY:	FREQUENCY:
Initial Calibration Blank	Onset of every analysis
Continuing Calibration Blank	Every 10 samples, per analysis
Initial Calibration Verification	Onset of every analysis
Continuing Calibration Verification	Every 10 samples, per analysis
Sample Duplicates	Every 10 samples, per analysis
Matrix Spike	Every 20 samples, per batch

10.10.1 DEFINITIONS:

Batch: Every 20 samples, per day.
Analysis: Each calibration, each day.

11.0 SAMPLE PREPARATION AND INSTRUMENTAL PROCEDURE

11.1 Sample Preparation

11.1.1 Measure 100 mL or an aliquot diluted to 100 mL into a 150 mL beaker, add 5 mL of conditioning reagent (Section 8.1.8) and mix at constant stirring with a magnetic stirring bar. As solution is being stirred, add a measured spoonful of $BaCl_2$ crystal and begin timing with a stopwatch immediately. Stir for 60 seconds.

11.2 Sample Analysis

11.2.1 Turn on spectrophotometer; to set wavelength enter 420 then press the wavelength button. Allow spectrophotometer to warm up 15-20 minutes.

11.2.2 Pour the solution prepared in Section 11.1.1 into the absorbance cell. Check the absorbance every 30 seconds for 4 minutes. Record the maximum absorbance (turbidity) in the Spectrophotometric Analysis Logbook (Attachment III).

11.2.3 All standards are analyzed first as in Section 9.0. A calibration plot of Absorbance vs. mg/L sulfate is generated and all subsequent samples are treated similarly, comparing the absorbance with those of the known standards.

11.2.4 Since turbidity and intense color interfere with the analysis, samples often require dilution or blank corrections for samples by omitting the Barium Chloride reagent in a duplicate analysis of the sample. Subtracting background absorbance corrects for turbidity and color interferences. Filtration is generally not employed since some precipitated sulfate may be removed.

12.0 CALCULATIONS

12.1 ICV, and CCV Recovery:

$$\frac{\text{Actual mg/L}}{50\ \text{mg/L}} \times 100 = \%\ \text{Recovery}$$

12.2 Spike Recovery:

$$\frac{(\text{Spiked sample, mg/L} - \text{sample conc., mg/L})}{10\ \text{mg/L}} \times 100 = \%\ \text{Recovery}$$

12.3 Relative Percent Difference (RPD):

$$\left[\frac{(\text{Sample 1} - \text{Sample 2})}{\text{avg., sample 1 \& 2}}\right] \times 100 = \%\ \text{RPD}$$

13.0 ACCEPTANCE CRITERIA

13.1

ICB, CCB	< 2.0 mg/L
ICV, CCV	85% - 115% Recovery; 90-110% for NC Wastewater samples
Matrix spike	75% - 125% Recovery
Duplicates	± 20 % or ± MDL if sample conc. < 5 x MDL

14.0 REPORTING OF RESULTS

14.1 Practical Quantitation Limits: 2.0 mg/L
Units of Measure: mg/L
Significant Figures: 2 figures
Data Reporting:
Below quantitation limit: BQL
QC information included in report:
Level 1 QC: Blank values, sampling COC
Level 2 QC: Blank values, Duplicate data (%RPD), sampling COC
Level 3 QC: Blank values, Duplicate data (%RPD) Matrix Spike data specific to client's samples, sampling and laboratory COC

15.0 SUPPLEMENTAL DOCUMENTS

15.1 SOP for Conducting MDL Studies, IEA Doc# QAS02000.NET.

16.0 REFERENCES

16.1 EPA Methods for Chemical Analysis of Water and Wastes, Method 375.4, 1983.

16.2 Standard Methods for the Examination of Water and Wastewater, 16th ed. Method 426C.

17.0 SUBSTANTIVE REVISIONS

17.1 Addendum to Section 3.0 to define deviations from EPA method 375.4. Addendum to Section 6.4 to clarify holding time. Section 12.4 amended to more clearly express absolute value.

17.2 Section 8.2 revised to clarify standard preparation. Addendum to Section 8.1: Section 8.1.1. to 8.1.7 added. Section 10.4 and 10.7 added. Section 10.10 revised to correct Matrix spikes as every 20 samples. Section 11.0 revised for clarification. Sections 10.2, 13.1 and 14.1 revised to correct the PQL. (Revised 03/21/96)

17.3 Sections 10.6 and 13.1 revised to reflect North Carolina Wastewater recovery criteria for ICVs and CCVs. (08/05/96)

Attachment I

Reagents Preparation and Receiving Log
Wet Chemistry Laboratory

Date	Analyst	Time	Reagent #	Reagent Name	Preparation Method	Expiration Date
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Secondary Review By:____________________ **Date:**___________

IEA, Inc. Form# CVL01200.NC

Attachment II

Standards Preparation and Receiving Log
Inorganics Laboratory

Date	Analyst	Time	Standard #	Standard Name	Vendor	Preparation Method	Expiration Date

Secondary Review By:_____ Date:_____

Attachment III

SPECTROPHOTOMETRIC ANALYSIS

Method Name:_____ Date:_____ Sample Matrix:_____

Test Code:_____ Prep. Date:_____ Analyst:_____

Daily Standards Preparation							
Standard ID	Stock Lot#	Stock Conc	Stock Vol	Diluent	Final Volume	Final Conc	Std #
ICV							
LCS/QC Check							
Working Standard							
Spike							

STANDARD CURVE

Samples/Standards Final Volume Required:_____mL (B)

Volume Std Used	Std #	Std Concentration	Absorbance Reading

Curve Correlation Coefficient (must be $\geq$ 0.995):_____ Y-Intercept:_____

Sample ID	Volume Used (mLs) (C)	Absorbance	Curve Conc. ug/mL (A)	Final Conc. (ug/mL)	QC Sample (Recovery/RPD)
ICV					
ICB					
LCS/QC CHECK					
CCV					
CCB					

CALCULATIONS

Concentration = ug/mL = (A) (B/C)

$$RPD = \frac{|(\text{Sample} - \text{Sample Dup.})| \times 100}{\text{Avg.. Sample} + \text{Dup.}}$$

$$\%\ \text{Recovery} = \frac{\text{Spiked Sample Result} - \text{Sample Result}}{\text{Spike Concentration}} \times 100$$

Comments:_____

Spike Witness:_____ Date:_____

Secondary Review By:_____ Date:_____

35. IEA SOP FOR SULFITE ANALYSIS

1.0 APPROVALS

The signatures of the following individuals indicate that this SOP is complete and meets the requirements specified in corporate document # QAS00200.NET. In addition, it signifies that the content meets the specifications of the referenced "Test Code".

Laboratory Director

Quality Assurance Manager

2.0 SCOPE AND APPLICATION

2.1 This method gives the procedure for the analysis of water samples for sulfite.

2.2 This procedure is applicable to aqueous samples, including drinking and surface waters, sewage, and industrial wastes.

2.3 This procedure is applicable to Test Code SO3_01.

3.0 SUMMARY OF METHOD

3.1 An acidified sample containing a starch indicator is titrated with a standard potassium iodide-iodate titrant to a faint permanent blue endpoint which appears when the reducing power of the sample has been completely exhausted.

3.2 This procedure is based on Standard Methods 16th Edition 428A and on EPA Method 377.1.

3.3 This procedure deviates from the above methods in the following ways:

3.3.1 Sample is measured out in a graduated cylinder instead of a pipet.

4.0 INTERFERENCES

4.1 The temperature of the sample must be below 50 degrees C.

4.2 Care must be taken to allow as little contact with air as possible. For example, do not filter the sample.

4.3 Other oxidizable substances, such as organic compounds, ferrous iron, and sulfide are positive interferences. Sulfide may be removed by adding 0.5 g of zinc acetate and analyzing the supernatant of the settled sample.

4.4 Nitrite gives a negative interference by oxidizing sulfite when the sample is acidified; this is corrected by either using a proprietary indicator which eliminates nitrite or by adding sulfamic acid.

4.5 Copper and possibly other heavy metals catalyze the oxidation of sulfite; EDTA should be used to complex metals.

5.0 SAFETY

5.1 Concentrated sulfuric acid can cause severe burns. Goggles or face shield, lab coat, and gloves are necessary when using this reagent.

6.0 SAMPLE CONTAINERS, COLLECTION AND PRESERVATION

6.1 Sample containers: Containers may be of plastic or glass, thoroughly rinsed and dried before use and are not to be reused.

6.2 Sample collection: Samples are collected without headspace in bottles to which 1 ml EDTA solution has been added per 100 ml of volume. Samples are packaged and sent to the laboratory and stored at 4 degrees C until analysis.

6.3 Sample preservation: Samples are to be preserved with EDTA solution, 1 ml per 100 ml of sample and stored at 4 degrees C.

6.4 Samples are to be analyzed immediately. If this is not possible, they are to be preserved as above and analyzed as soon as possible.

7.0 APPARATUS AND MATERIALS

7.1 25 ml buret, graduated in 0.1 ml

7.2 Stir plate, stir bar, magnetic stir bar retriever

7.3 125 ml Erlenmeyer flasks

7.4 Assorted beakers, graduated cylinders, transfer pipets, volumetric pipets and volumetric flasks, scoopula

8.0 REAGENTS AND STANDARD PREPARATION

8.1 Reagents:

8.1.1 Reagent water, 16.6 megohm or higher, deionized

8.1.2 Concentrated sulfuric acid (H_2SO_4), ACS grade

8.1.3 Sulfamic acid, ACS grade

8.1.4 Soluble starch, ACS grade

8.1.5 Salicylic acid, ACS grade

8.1.6 Ethylenediamine tetraacetate (EDTA), ACS grade

8.1.7 Potassium iodide (KI), ACS grade

8.1.8 Potassium iodate (KIO_3), primary grade, dried 4 hours at 120 °C

8.1.9 Sodium bicarbonate($NaHCO_3$), ACS grade

8.2 Reagent prep:

8.2.1 1+1 sulfuric acid: Carefully add a volume of concentrated sulfuric acid to an equal volume of d.i. water; mix well; cool.

8.2.2 Starch indicator: Dissolve 5 g soluble starch and 0.5 g salicylic acid in 250 ml hot d.i. water.

8.2.3 EDTA solution: Dissolve 2.5 g disodium EDTA in 100 ml d.i. water.

8.2.4 Standard potassium iodide-iodate titrant, 0.0125N: Dissolve 0.4458 g potassium iodate, 4.25 g potassium iodide, and 0.310 g sodium bicarbonate in d.i. water and dilute to 1 liter.

8.3 Standard solutions:

8.3.1 Initial and Continuing Calibration Standards: Purchased from Hach Co., Cat # 22674, 5000 mg/L. Dilute a 10 ml voluette to 100 ml volumetrically, to make a 500 mg/L calibration verification standard.

8.3.2 Matrix Spiking solution: Use the 500 mg/L calibration verification standard.

9.0 CALIBRATION

Not applicable to this method

10.0 QUALITY CONTROL

10.1 Method detection limits are calculated based on the 40 CFR, Part 136, Appendix B definition of method detection limit. A minimum of seven replicate standards prepared by referenced methods including all processing steps including distillation and digestion where appropriate. Method detection limits are calculated by multiplying the standard deviation of all replicate results by student t values listed in 40 CFR.

10.2 Practical quantitation limit for this method is the same as the method detection limit.

10.3 Matrix spikes are performed by pipetting 1000 ul of 500 mg/L calibration verification standard into an aliquot of sample, making a 10 mg/L spike, based on 50 ml (500 ug total spike).

10.4 QC Check Samples consist of Initial and Continuing Calibration Verification standards (ICV and CCV, respectively), Section 8.3.1.

10.5 Surrogates are not applicable to this method.

10.6 QC SCHEDULES:

ACTIVITY:	FREQUENCY:
Initial Calibration Verification	Onset of every analysis
Initial Calibration Blank	Onset of every analysis
Preparation blank	Every 10 samples, per batch
Continuing Calibration Verification	Every 10 samples, per analysis
Continuing Calibration Blank	Every 10 samples, per analysis
Matrix Spike	Every 20 samples, per batch
Duplicate	Every 20 samples, per batch

10.6.1 DEFINITIONS:
Batch: Every 20 samples leached, digested, or distilled, per day.
Analysis: Each calibration, each day.

11.0 SAMPLE PREPARATION AND INSTRUMENTAL PROCEDURES

11.1 Analytical Procedure

11.1.1 Rinse and fill a 25 ml buret with standard potassium iodide/iodate titrant.

11.1.2 Add to a 250 ml beaker, in order: 1 ml 1+1 sulfuric acid, 0.1 g sulfamic acid, 50 ml sample, and 5-6 drops starch indicator.

11.1.3 Record initial volume of titrant. Titrate to first permanent blue color. Record final volume of titrant.

11.1.4 For ICB and CCB, add 50 ml deionized H2O instead of sample. Titrate as above.

11.1.5 For ICV and CCV, pipet 5.0 ml of 500 mg/L calibration verification standard into 250 ml beaker; add 45 ml d.i. H2O. Titrate as above.

12.0 CALCULATIONS

12.1 Analyte:

$$mg/L = \frac{(Vt - Vb)(Nt)(meq\ wt)(1000)}{ml\ sample}$$

where: Vt = volume of titrant used for sample
Vb = volume of titrant used for ICB
Nt = normality of titrant (0.0125 meq/ml)
meq wt = 40.00 mg/meq

12.2 ICV and CCV Recovery:

$$\frac{Actual\ mg/L}{500\ mg/L} \times 100 = \%\ Recovery$$

12.3 Spike Recovery:

$$\frac{(Spiked\ sample\ conc.,\ mg/L) - (sample\ conc.,\ mg/L)}{10\ mg/L} \times 100 = \%\ Recovery$$

12.4 Method blank, Initial Calibration and Continuing Calibration blanks: Not to exceed 2.0 mg/L (MDL)

12.5 Relative Percent Difference:

$$\left[\frac{(Sample\ 1 - Sample\ 2)}{avg.,\ sample\ 1\ \&\ 2}\right] \times 100 = \%\ RPD$$

13.0 ACCEPTANCE CRITERIA SULFITE ANALYSIS:

ICB, CCB	< 2 mg/L
ICV, CCV	85% - 115% Recovery
Matrix spike	75% - 125% Recovery
Duplicates	± 20 % or ± 2 mg/L (MDL) if sample conc. < 5 x MDL

14.0 REPORTING OF RESULTS

Practical Quantitation Limits: Same as MDL.
Units of Measure: mg/L
Significant Figures: 2 figures
Data Reporting:
Below quantitation limit: BQL
QC information included in report:
Level 1 QC: Blank values.
Level 2 QC: Blank values, Duplicate data (%RPD).
Level 3 QC: Blank values, Duplicate data (%RPD), matrix spike recoveries, specific to client's samples.

15.0 SUPPLEMENTAL DOCUMENTS

N/A

16.0 REFERENCES

16.1 Standard Methods 17th Edition, Method 4500-SO_3^{2-}B.

16.2 EPA Method 377.1

17.0 SUBSTANTIVE REVISIONS

17.1 Revision to Section 2.1. Amendment to Section 3.3. Amendments to Sections 8.1.1, 8.1.6, 8.1.7, 8.1.8, 8.1.9. Amendement to Section 10.6. Amendments to Sections 11.1.2 and 11.1.5. Amendment to Section 13.0.

17.2 Section 3.3.1 was deleted: bottles are sent to clients preserved with EDTA. Section 6.2 was updated to remove the collection without preservative. Section 6.3 was updated to include storage at 4 degrees C until analysis.

36. IEA SOP FOR ACIDITY ANALYSIS

STANDARD OPERATING PROCEDURES FOR ACIDITY

1.0 APPROVALS

The signature of the following individuals indicates that this Standard Operation Procedure (SOP) is complete and meets the requirements specified in Corporate Document # QAS00200.NET. In addition, it also signifies that the content meets the specifications of the referenced "Test Code".

Laboratory director _____

Quality Assurance Manager _____

DRAFT

2.0 SCOPE AND APPLICATION

2.1 This method describes the procedure for the analysis of aqueous and semi-aqueous samples for acidity, expressed as $CaCO_3$. The applicable range is 10 to 1000 mg/L.

2.2 This method is applicable to aqueous samples, including surface and groundwaters, and domestic and industrial wastes.

2.3 This method is applicable to Test Code ACD_01.

3.0 SUMMARY OF METHOD

3.1 The pH of the sample is determined and a measured amount of standard acid is added, as needed, to lower the pH to 4 or less. Hydrogen peroxide is added, the solution boiled for several minutes, cooled, and titrated electrometrically with standard alkali to pH 8.2.

3.2 This method is based on Standard Methods for the Examination of Water and Wastewater, 16th Edition, Method 402, and on EPA Method 305.1.

3.3 This method does not deviate from the above methods.

DRAFT

4.0 INTERFERENCES

4.1 Suspended solids or precipitates formed during titration may cause a sluggish electrode response. Titrant may be added slowly dropwise as the endpoint pH is approached, to offset this interference.

5.0 SAFETY

5.1 Hydrogen peroxide is a strong oxidizer. Gloves should be worn when handling, and great care must be taken when using it in the vicinity of a hot plate.

5.2 Goggles or face shield, lab coat, and gloves are necessary if preparing titrants from concentrated sulfuric acid and sodium hydroxide pellets.

5.3 Potassium hydrogen phthalate may cause skin and respiratory irritation. Gloves are recommended when handling; avoid breathing dust.

6.0 SAMPLE CONTAINERS, COLLECTION AND PRESERVATION

6.1 Sample containers: Containers may be glass or plastic, thoroughly rinsed and dried before use, and are not to be reused.

6.2 Sample collection: Samples are collected without preservatives. Samples are packaged and sent to the laboratory and stored at 4 °C until analysis.

6.3 Sample preservation is not necessary for this analysis.

6.4 The holding time for acidity analysis is 14 days from sampling.

7.0 APPARATUS AND MATERIALS

7.1 pH meter with combination electrode and temperature probe

7.2 25 ml buret, graduated in 0.1 ml

7.3 125 ml Erlenmeyer flasks

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7.4 250 ml beakers

7.5 50 ml graduated cylinders

7.6 Hot plate

7.7 Assorted beakers, volumetric pipets, volumetric flasks, watch glasses

7.8 Stir plate, stir bars, magnetic stir bar retriever

8.0 REAGENTS AND STANDARD PREPARATION

8.1 Reagents:

8.1.1 Reagent water, 16.6 mega-ohm or higher, deionized

8.1.2 Potassium hydrogen phthalate (KHP), ACS grade, dried

8.1.3 Phenolphthalein indicator

8.1.4 Ethanol 95%, not denatured

8.1.5 Sodium hydroxide pellets (NaOH), ACS grade

8.1.6 Optional: Purchased solutions of NaOH, 0.1 N and 0.02 N

8.1.7 Concentrated sulfuric acid (H_2SO_4), ACS grade

8.1.8 Optional: Purchased solutions of H_2SO_4, 0.1 N and 0.02 N

8.1.9 pH 4, pH 7, and pH 10 buffers, purchased

8.1.10 Hydrogen peroxide (H_2O_2), 30% solution

8.2 Reagent prep:

8.2.1 Phenolphthalein indicator solution: Dissolve 0.5 g phenolphthalein in 50 ml 95% ethanol and 50 ml d.i. water.

8.2.2 Standard NaOH titrant, 0.1 N: Dissolve 4 g of NaOH pellets in d.i. water and bring to 1 liter volume.

8.2.3 Standard NaOH titrant, 0.02 N: Dilute 400 ml of 0.1 N NaOH solution to 1 liter.

8.2.4 Standard H_2SO_4 titrant, 0.1 N: Dilute 2.8 ml of concentrated H_2SO_4 to 1 liter.

8.2.5 Standard H_2SO_4 titrant, 0.02 N: Dilute 400 ml of 0.1 N H_2SO_4 solution to 1 liter.

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8.3 Titrant standardization:

8.3.1 0.1 N standard NaOH:
Weigh 0.205 g dried KHP into a 125 ml Erlenmeyer flask; record weight to 0.1 mg. Add 50 ml d.i. water; swirl to dissolve. Prepare two more KHP solutions as above. Add 50 ml d.i water to a 125 ml Erlenmeyer flask as a Blank. Add 1 to 2 drops of phenolphthalein and a stir bar to each flask.
Rinse and fill a 25 ml buret with 0.1 N NaOH solution. Record initial volume. Titrate Blank and each KHP solution until the pink endpoint persists for 30 seconds. Record final volume.
Calculate normality of NaOH solution:

$$N\ NaOH = \frac{(g\ KHP)(1000)}{(204.2)(V_t - V_b)}$$

where V_t = final volume of titrant - initial volume of titrant
V_b = volume of titrant used for Blank

8.3.2 0.02 N NaOH: Standardize as for 0.1 N NaOH, using 0.041 g dried KHP.

8.3.3 0.1 N H_2SO_4:
Pipet 10.0 ml of standardized 0.1 N NaOH solution into each of three 125 ml Erlenmeyer flasks. Add 40 ml of d.i. water, 1 to 2 drops of phenolphthalein, and a stir bar to each flask.
Rinse and fill a 25 ml buret with 0.1 N H_2SO_4 solution. Record initial volume. Titrate each solution to a clear endpoint. Record final volume.
Calculate normality of H_2SO_4 solution:

$$N\ H_2SO_4 = \frac{(N\ NaOH)(ml\ NaOH)}{V_t}$$

where V_t = final volume of titrant - initial volume of titrant

8.3.4 0.02 N H_2SO_4: Standardize as for 0.1 N H_2SO_4, using 10.0 ml of 0.02 N NaOH solution.

8.4 Standard solutions:

8.4.1 Initial and Continuing Calibration Standards: Purchased from Hach Co., Cat # 14330-10, 25,000 mg/L as CaCO3. Dilute 10 ml voluette to 500 ml, to make a 500 mg/L calibration standard. Dilute 50 ml of this solution to 250 ml, to make a 100 mg/L

calibration standard.

8.4.2 Matrix spiking standard: 500 mg/L calibration standard. Add 3.0 ml to 50 ml of a sample with an initial pH below 4.0, to constitute a 30 mg/L spike.

9.0 CALIBRATION

9.1 pH meter calibration: Calibrate on two-point calibration using pH 4 and pH 10 buffers. Efficiency (slope) must be greater than 0.90; preferably greater than 0.95.

10.0 QUALITY CONTROL

10.1 Method detection limits are calculated based on the 40 CFR chapter 1 definition of method detection limit. A minimum of seven replicate standards prepared by referenced methods including all processing steps including distillation and digestion where appropriate. Method detection limits are calculated by multiplying the standard deviation of all replicate results by student t values listed in 40 CFR.

10.2 Practical quantitation limit for this method is the same as the method detection limit.

10.3 QC Check Samples consist of Initial and Continuing Calibration Verification standards (ICV and CCV, respectively), Section 8.4.1.

10.4 Surrogates are not applicable to this method.

10.5 QC SCHEDULES:

ACTIVITY:	FREQUENCY:
Initial Calibration Verification	Onset of every analysis
Initial Calibration Blank	Onset of every analysis
Continuing Calibration Verification	Every 10 samples, per analysis
Continuing Calibration Blank	Every 10 samples, per analysis
Matrix Spike	Every 20 samples, per batch
Duplicate	Every 20 samples, per batch

10.5.1 DEFINITIONS:
Batch: Every 20 samples leached, digested, or distilled, per day.
Analysis: Each calibration, each day.

11.0 SAMPLE PREPARATION AND INSTRUMENTAL PROCEDURES

11.1 Analytical Procedure

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11.1.1 Standardize the pH meter, using the pH 4 and pH 10 buffers; record the Efficiency. Read and record the pH 7, pH 10, and pH 4 buffers.

11.1.2 Add 50 ml sample to a 250 ml beaker; add stir bar. Place beaker on stir plate and adjust stirring. Place pH probe in sample. Allow the pH to stabilize; record the initial pH.

11.1.3 If sample pH is above 4.0, add standardized 0.02 N H_2SO_4 in 5.0 ml increments to lower the pH to 4.0 or less. If sample pH is less than 4.0, addition of H_2SO_4 is not necessary.

11.1.4 Add 5 drops of hydrogen peroxide and cover with a watch glass. Place beaker on hot plate.

11.1.5 Heat sample to boiling and continue boiling for 2 to 4 minutes. Remove beaker from hot plate and allow sample to cool to room temperature.

11.1.6 Rinse and fill a 25 ml buret with standardized 0.02 N NaOH. Place beaker on stir plate and adjust stirring. Allow pH to stabilize.

11.1.7 Record initial volume of titrant. Add titrant dropwise to raise pH to 8.2. Record final volume of titrant.

11.1.8 For ICV and CCV, pour 50 ml of calibration verification standard into a 250 ml beaker. Analyze as for sample.

12.0 CALCULATIONS

12.1 Analyte:

$$\frac{[(V_t)(N_t) - (V\ H_2SO_4)(N\ H_2SO_4)](\text{meq wt})(1000)}{\text{ml sample}} = \text{mg/L } CaCO_3$$

where: V_t = volume of titrant used for sample
N_t = normality of titrant
$V\ H_2SO_4$ = volume of H_2SO_4 added to reduce pH to 4 or less
$N\ H_2SO_4$ = normality of H_2SO_4
meq wt = 50.04 mg/meq

12.2 ICV and CCV Recovery:

$$\frac{\text{Actual mg/L}}{100\ \text{mg/L}} \times 100 = \%\ \text{Recovery}$$

12.3 Relative Percent Difference:

$$\frac{|(\text{Sample 1} - \text{Sample 2})|}{\text{avg., sample 1 \& 2}} \times 100 = \%\ \text{RPD}$$

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13.0 ACCEPTANCE CRITERIA ACIDITY ANALYSIS:

ICB, CCB	< 10 mg/L (MDL)
ICV, CCV	85% - 115% Recovery
Matrix spike	75% - 125% Recovery
Duplicates	± 20 % or ± 10 mg/L (MDL) if sample conc. < 5 x MDL

14.0 REPORTING OF RESULTS

Practical Quantitation Limits: Same as MDL.
Units of Measure: mg/L
Significant Figures: 2 figures
Data Reporting:
Below quantitation limit: BQL
QC information included in report:
Level 1 QC: Blank values.
Level 2 QC: Blank values, Duplicate data (%RPD).
Level 3 QC: Blank values, Duplicate data (%RPD), matrix spike recoveries, specific to client's samples.

15.0 SUPPLEMENTAL DOCUMENTS

None.

16.0 REFERENCES

16.1 Standard Methods for the Examination of Water and Wastewater, 16th Edition, Method 402.

16.2 EPA Method 305.1.

17.0 SUBSTANTIVE REVISIONS

No current revisions exist.

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37. IEA SOP FOR PH IN WATER

1.0 APPROVALS

The signature of the following individuals indicates that this SOP is complete and meets the requirements specified in corporate Document #QAS00200.NET. In addition, it also signifies that the content meets the specifications of the referenced "Test Code".

Laboratory Director _____

Quality Assurance Manager _____

2.0 SCOPE AND APPLICATION

2.1 This method is applicable to the measurement of pH in drinking, surface, and saline waters, domestic and industrial wastes and acid rain (atmospheric deposition).

2.2 This method is applicable to Test Code PH_01 for pH analysis in water.

3.0 SUMMARY OF METHOD

3.1 The pH of the sample is determined electrometrically using either a glass electrode in combination with a reference potential or a combination electrode.

3.2 This SOP is based upon EPA method 150.1.

3.3 This procedure does not deviate from EPA Method 150.1.

4.0 INTERFERENCES

4.1 The glass electrode, in general, is not subject to solution interferences from color, turbidity, colloidal matter, oxidants, reductants or high salinity.

4.2 Coatings of oily material or particulate matter can impair electrode response. These coatings can usually be removed by gentle wiping or detergent washing, followed by distilled water rinsing. An additional treatment with hydrochloric acid (1:9) may be necessary to remove any remaining film.

4.3 Temperature effects on the electrometric measurement of pH arise from two sources. The first is caused by the change in electrode output at various temperatures. This interference can be controlled with instruments having temperature compensation or by calibrating the electrode-instrument system at the temperature of the samples. The second source is the change of pH due to changes in the sample as the temperature

changes. This error is sample-dependent and cannot be controlled. It should be noted by reporting both the pH and temperature at the time of analysis.

5.0 SAFETY

5.1 Prolonged skin contact with pH buffers may cause dermatitis. Inhalation may cause respiratory irritation. Consult Material Safety Data Sheets (MSDS) for precautions and proper handling of buffer soultions.

6.0 SAMPLE CONTAINERS AND PRESERVATION

6.1 Sample Containers: Containers may be either glass or plastic and should be thoroughly rinsed and dried before use. Sample containers are not to be reused.

6.2 Sample Collection: Samples are collected into either glass or plastic containers. Samples are cooled to 4 degrees Celsius and are maintained at that temperature during shipment to the laboratory for analysis.

6.3 Sample Preservation: Sample preservation is not necessary. Samples should be refrigerated at 4 degrees Celsius until analysis.

6.4 Holding Times: The pH of a sample should be taken immediately after sampling or as soon thereafter as possible.

7.0 APPARATUS AND MATERIALS

7.1 APPARATUS:

7.1.1 Electrometer (pH meter) with expanded mV scale or specific ion meter

7.1.2 pH combination electrode

7.1.3 Temperature sensing electrode

7.1.4 Thermally insulated magnetic stirrer and Teflon coated stir bar

7.1.5 50 mL graduatd cylinder

7.1.6 100 mL glass beaker

7.1.7 Plastic rinse bottle with deionized water

7.1.8 Magnetic stir bar retriever

8.0 REAGENTS AND STANDARD PREPARATION

8.1 Reagents:

8.1.1 Reagent water, 16.6 mega-ohm, deionized

8.1.2 Standard pH buffer solutions, pH 4.00, 7.00 and 10.00, purchased

8.1.3 Hach Standard pH powder pillows: pH 4.00, 7.00 and 10.00. Catalog numbers 22268-66, 22270-64 and 22271-66, respectively. Loveland, CO.

9.0 CALIBRATION

9.1 Calibration Standards: The calibration standards consist of pH buffers 4.00, 7.00 and 10.00.

9.2 The working range of the calibration is 0 to 14 pH units.

9.3 Instrument Calibration:

9.3.1 Place the pH electrode and temperature sensor into a beaker containing sufficient pH 4.00 buffer to cover at least 1/2" of the probe tip, with constant stirring. From the pH meter keypad, press "STDBY/MEAS", press the "MODE" key in succession until "PH" units are displayed on the LED. Press "TWO POINT CAL". Allow sufficient time (at least 30 seconds) for the mV reading to stabilize: press "ENTER" to store the value. Press "STDY/MEAS" and raise the electrodes out of the buffer and rinse with deionized water, blot dry with a laboratory wipe.

9.3.2 Place the pH electrode and temperature sensor into a second beaker containing sufficient pH 10.00 buffer to cover at least 1/2" of the probe tip, with constant stirring. The display will read "ENTER SECOND BUFFER", press "10.00", then "ENTER". Allow the mV reading to stabilize: Press "ENTER" to store the value. When the display reads "EFF=", press "ENTER". Press "STDBY/MEAS" and raise the electrodes out of the buffer and rinse with deionized water, blot dry with a laboratory wipe.

9.3.3 Place the pH electrode and temperature sensor into a third beaker containing sufficient pH 7.00 buffer to cover at least 1/2" of the probe tip, with constant stirring. When the pH reading has stabilized, record the pH in the logbook.

9.4 Internal standards are not applicable to this method.

9.5 Acceptance criteria for the calibration requires that the efficiency (correlation) is 0.995

or greater and the pH 7.00 buffer reads in the range of 6.9 to 7.1 pH units.

10.0 QUALITY CONTROL

10.1 Method detection limits are calculated on the 40 CFR, Part 136, Appendix B definition of method detection limit. A minimum of seven replicate standards prepared by referenced methods including all processing steps. Method detection limits are calculated by multiplying the standard deviation of all replicate results by student t values listed in 40 CFR.

10.2 Practical quantitation limits for this method are the same as the method detection limit.

10.3 Daily performance tests consist of noting and recording the efficiency (correlation) of the calibration curve and the pH 7.00 buffer recovering in the 6.9 to 7.1 range.

10.4 Matrix spikes are not applicable to this method.

10.5 QC Check Samples consist of Initial and Continuing Calibration Verification standards (ICV and CCV, respectively), pH 7.00 buffer.

10.6 Surrogates are not applicable to this method.

10.7 QC SCHEDULES:

ACTIVITY:	FREQUENCY:
Initial Calibration Verification	Onset of every analysis
Continuing Calibration Verification	Every 10 samples analyzed

10.8 DEFINITIONS:

Batch: Every 10 samples leached, digested, or distilled, per day.
Analysis: Each calibration each day.

11.0 SAMPLE PREPARATION AND PROCEDURE

11.1 Instrumental Conditions: Instrument is calibrated using pH 4.00 and 10.00 buffers. The pH 7.00 buffer is read back. The criteria for acceptable functioning requires the efficieny to be greater than 0.995 and that the pH 7.00 buffer recovers in the range of 6.9 - 7.1 pH units.

11.2 Sample Preparation: Sample preparation is minimal since neither distillation nor

digestion are performed for this analysis.

11.3 Sample Measurement: Place approximately 50 mL of aqueous sample into a small beaker, enough to cover the tip of the electrode at least 1/2". With constant stirring, depress the "STDBY/MEAS" key. When the display stabilizes, record the pH in the appropriate logbook.

12.0 CALCULATIONS

12.1 ICV and CCV Recovery: 6.9 to 7.1 pH units

12.2 Relative Percent Difference:

$$\left[\frac{\text{(Sample 1 - Sample 2)}}{\text{avg., Sample 1 \& 2}} \right] \times 100 = \% \text{ RPD}$$

13.0 ACCEPTANCE OF DATA

ICV, CCV	6.9 to 7.1 pH units
Duplicates	$\pm$ 0.5 pH units

14.0 REPORTING OF RESULTS

Practical Quantitation Limits: 0.1 pH units
Units of Measure: pH units
Significant Figures: 2 figures
Data Reporting:
Below Quantitation Limit: BQL
QC Information Included in Report:
Level 1 QC: None
Level 2 QC: Duplicate data, ICV, CCV

15.0 SUPPLEMENTAL DOCUMENTS

15.1 EPA SW-846 Method 9040 addresses samples where the aqueous phase constitutes at least 20% of the total volume.

16.0 REFERENCES

16.1 EPA Method 150.1.

17.0 SUBSTANTIVE REVISIONS

17.1 Original document.

38. IEA SOP FOR PH IN SOIL

1.0 APPROVALS

The signature of the following individuals indicates that this Standard Operating Procedure (SOP) is complete and meets the requirements specified in Corporate Document # QAS00200.NET. In addition, it also signifies that the content meets the specifications of the referenced "Test Code".

Laboratory Director

Quality Assurance Manager

2.0 SCOPE AND APPLICATION

2.1 This method is applicable to the measurement of pH in leachates of soil samples.

2.2 This method is applicable to Test Code PH_02.

3.0 SUMMARY OF METHOD

3.1 The pH of a sample is determined electrometrically using either a glass electrode in combination with a reference potential or combination electrode.

3.2 This method is based on EPA SW-846 Method 9045A.

3.3 This procedure does not deviate from EPA SW-846 Method 9045A.

4.0 INTERFERENCES

4.1 The glass electrode, in general, is not subject to solution interferences from color, turbidity, colloidal matter, oxidants, reductants, or high salinity.

4.2 Coatings of oily material or particulate matter can impair electrode response. These coatings can usually be removed by gentle wiping or detergent washing, followed by rinsing with distilled water. An additional treatment with hydrochloric acid (1:9) may be necessary to remove any remaining film.

4.3 Temperature effects on the electrometric determination of pH arise from two sources. The first is caused by the change in electrode output at various temperatures. This interference can be controlled with instruments having temperature compensation or by calibration of the electrode-instrument system at the temperature of the samples. The second source of temperature effects is the change of pH due to changes in the sample as the temperature changes. This error is sample-dependent and cannot be controlled. It should therefore, be noted by reporting both the pH and temperature at the time of analysis.

5.0 SAFETY

5.1 Prolonged skin contact with pH buffers may cause dermatitis. Inhalation may cause respiratory irritation. Consult Material Safety Data Sheets (MSDS) for precautions and proper handling of the buffer solutions.

6.0 SAMPLE CONTAINERS, COLLECTION AND PRESERVATION

6.1 Containers may be either glass or plastic, thoroughly rinsed and dried before use and are not to be reused.

6.2 Samples are collected in either plastic or glass containers. Samples are cooled and maintained at 4 °C and shipped to the laboratory for analysis.

6.3 Sample preservation is not necessary. Samples should be refrigerated at 4 °C until analysis.

6.4 The pH of a sample should be taken immediately after sampling. Otherwise, the pH should be taken as soon as reasonably possible.

7.0 APPARATUS AND MATERIALS

7.1 Electrometer (pH meter) with expanded mV scale or specific ion meter.

7.2 pH combination electrode.

7.3 Temperature sensing electrode.

7.4 Magnetic stirrer, thermally insulated, and Teflon coated stirring bar.

7.5 50 mL graduated cylinder.

7.6 100 mL glass beaker.

7.7 Plastic rinse bottle, with reagent water.

7.8 Magnetic stir bar retriever.

8.0 REAGENTS AND STANDARD PREPARATION

8.1 Reagents

8.1.1 Reagent water, 16.6 megohm-cm or higher, deionized.

8.1.2 Calcium chloride, purchased from a commercial vendor.

8.1.2.1 Stock $CaCl_2$ solution: Dissolve 264.75 grams of $CaCl_2 * 2H_2O$ (Section 8.1.2) in reagent water (Section 8.1.1). Cool and dilute to 500 mL in a volumetric flask. Standardize by diluting 20 mL of this solution to 1 liter with reagent water and titrating a 25 mL aliquot with 0.1N $AgNO_3$, using 1 mL of 5% K_2CrO_4 as the indicator. Standardization is documented in the Titrant Standardization Logbook (Attachment IV).

8.1.3 Hydrochloric acid, purchased from a commercial vendor.

8.1.4 Calcium hydroxide, purchased from a commercial vendor.

8.1.5 0.01 M $CaCl_2$: Dilute 5 mL of stock calcium chloride solution (Section 8.1.2.1) to 1.8 L in reagent water. If the pH of this solution is not between 5.0 and 6.5, adjust by adding $Ca(OH)_2$ or HCl. The conductivity should be 2.32 $\pm$ 0.08 mmho per cm at 25 °C.

8.1.6 Hydrochloric acid, 1:3 volume/volume.

8.1.7 Reagent receipt and preparation are documented in the Reagent Receipt and Preparation Logbook (Attachment I).

8.2 Standards

8.2.1 Calibration Standards

8.2.1.1 Standard pH buffer solutions, pH = 4.00, 7.00 and 10.00, purchased from a commercial vendor.

8.2.2 Laboratory Control Standards

8.2.2.1 Hach Standard pH powder pillow: pH 7.00. Catalog # 22270-64. Loveland, CO.

8.2.3 Standard receipt and preparation are documented in the Standard Receipt and Preparation Logbook (Attachment II).

9.0 CALIBRATION

9.1 The working range of the calibration is 0 to 14 pH units.

9.2 Instrument Calibration

9.2.1 Place the pH electrode and temperature sensor into a beaker containing sufficient pH 4.00 buffer to cover at least 1/2" of the electrode, with constant stirring. From the pH meter keypad, press "STDBY/MEAS", press the "MODE" key in succession until "PH" units are displayed on the LED. Press "TWO POINT CAL". Allow sufficient time (at least 30 seconds) for the mv reading to stabilize: press ENTER to store the value. Press "STDBY/MEAS" and raise the electrodes out of the buffer and rinse with reagent water, blot dry with a laboratory wipe.

9.2.2 Place the pH electrode and temperature sensor into a second beaker containing sufficient pH 10.00 buffer to cover at least 1/2" of the electrode, with constant stirring. The display will read "Enter second buffer", press "10.00", then "ENTER". Allow the mv reading to stabilize: press ENTER to store the value. When the display reads "EFF=", press ENTER. Press "STDBY/MEAS" and raise the electrodes out of the buffer and rinse with reagent water, blot dry with a laboratory wipe.

9.2.3 Place the pH electrode and temperature sensor into a third beaker containing sufficient pH 7.00 buffer to cover at least 1/2" of the electrode, with constant stirring. When the pH reading has stabilized, record the pH in the Potentiometric Determination of pH Logbook (Attachment III).

9.2.4 Acceptance criteria for the calibration requires that the efficiency (correlation coefficient) is 0.995 or greater and the pH 7.00 buffer reads in the range of 6.9 to 7.1 pH units.

10.0 QUALITY CONTROL

10.1 Method detection limits are performed and calculated in accordance with SOP for Conducting MDL Studies, IEA Doc# QAS02000.NET.

10.2 The Practical Quantitation Limit (PQL) for this method is 0.1 pH units.

10.3 Daily performance tests consist of noting and recording the efficiency (correlation coefficient) of the calibration curve and the pH 7.00 buffer recovering in the 6.9 to 7.1 pH range.

10.4 Matrix spikes are not applicable to this method.

10.5 Laboratory Control Samples consist of Initial and Continuing Calibration Verification standards (ICV and CCV, respectively), pH 7.00 buffer (Section 8.2.2.1).

10.6 Surrogates are not applicable to this method.

10.7 QC SCHEDULES

ACTIVITY:	FREQUENCY:
Duplicate Sample	Every Batch or 10 samples
Initial Calibration Verification	Onset of every analysis
Continuing Calibration Verification	Every 10 samples analyzed

10.8 DEFINITIONS

Batch: Every 10 samples leached, per day.
Analysis: Each calibration, each day.

11.0 SAMPLE PREPARATION AND INSTRUMENTAL PROCEDURES

11.1 Calibrate the pH meter as in Section 9.0.

11.2 Determination of Calcareous versus Noncalcareous Soils

11.2.1 Place 0.5 grams of sample on a piece of aluminum foil.

11.2.2 Add one or two drops of 1:3 HCl to the sample. Bubbling or an audible fizz indicates that the sample is calcareous.

11.3 Sample Preparation for Noncalcareous Soils

11.3.1 Weigh 50 grams of sample into a 150 mL beaker and record the weight to the nearest 0.01 grams in the Potentiometric Analysis of pH Logbook (Attachment III).

11.3.2 Add 50 mL of reagent water and stir at room temperature for 30 minutes.

11.3.3 Let the sample stand for one hour prior to analysis to allow the suspension to settle.

11.3.4 Analyze as in Section 11.5.

11.4 Sample Preparation for Calcareous Soils

11.4.1 Weigh 25 grams of sample into a 150 mL beaker and record the weight to the nearest 0.01 grams in the Potentiometric Analysis of pH Logbook.

11.4.2 Add 50 mL of 0.01 M $CaCl_2$ and stir at room temperature for 30 minutes.

11.4.3 Let the sample stand for one hour prior to analysis to allow the suspension to settle.

11.4.4 Analyze as in Section 11.5.

11.5 Sample Analysis

11.5.1 Place approximately 50 mL of aqueous or 0.01 M $CaCl_2$ leached sample into a small beaker, enough to cover the tip of the electrode at least 1/2".

11.5.2 With constant stirring, depress the "STDBY/MEAS" key.

11.5.3 When the display stabilizes, record the pH in the Potentiometric Analysis of pH Logbook.

12.0 CALCULATIONS

12.1 Relative Percent Difference

$$\left| \frac{(\text{Sample 1} - \text{Sample 2}) \times 100}{\text{avg., sample 1 \& 2}} \right| = \% \text{ RPD}$$

13.0 ACCEPTANCE CRITERIA

13.1	ICV, CCV	6.9 to 7.1 pH units
	Duplicates	$\pm$ 20% RPD

14.0 REPORTING OF RESULTS

14.1	Practical Quantitation Limits:	0.1 pH units
	Units of Measure:	pH units
	Significant Figures:	2 figures
	Data Reporting:	Level I: Results, Sampling COCs. Level II: Results, Duplicate Data, Sampling COCs. Level III: Results, Duplicate Data, Sampling and Laboratory COCs.

14.2 Noncalcareous soils are reported as "soil pH measured in water"

14.3 Calcareous soils are reported as "soil pH measured in 0.01 M $CaCl_2$"

15.0 SUPPLEMENTAL DOCUMENTS

15.1 SOP for Conducting MDL Studies, IEA Doc# QAS02000.NET.

16.0 REFERENCES

16.1 Test Methods for Evaluating Solid Wastes, Method 9045A, 3rd Edition, September 1994 Revision.

17.0 SUBSTANTIVE REVISIONS

17.1 Original Issue (04/20/95)

17.2 Section 3.2, 3.3 and 15.1 revised to current the SW-846 9045A method. Section 8.0 revised for clarification. Section 8.1.1 revised to megohm-cm. Section 11.0 revised for clarification. Section 11.4 corrected to calcareous preparation. (08/09/96)

ATTACHMENT I

Reagents Preparation and Receiving Log
Wet Chemistry Laboratory

Date	Analyst	Time	Reagent #	Reagent Name	Preparation Method	Expiration Date
			R			
			R			
			R			
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Secondary Review By:____________________ Date:__________

ATTACHMENT II

Standards Preparation and Receiving Log
Inorganics Laboratory

Date	Analyst	Time	Standard #	Standard Name	Vendor	Preparation Method	Expiration Date

Secondary Review By:_____ Date:_____

ATTACHMENT III

POTENTIOMETRIC ANALYSIS

*Method Name:*_____ *Date:*_____ *Analyst:*_____

DAILY STANDARDS

Standard ID	Stock Lot#	Stock Vol.	Stock Conc.	Diluent	Final Vol.	Final Conc.
ICV						
LCS/QC Check						
Working Standard						

STANDARD CURVE

Samples/Standards Final Volume Required:_____ mL

Standard ID	Std Concentration	pH Reading

Curve Correlation Coefficient (must be $\geq$ 0.995):_____ Slope:_____

Sample ID	Volume Used (mLs)	pH Reading	Temperature °C	Final pH Units	QC Sample (RPD)
ICV					
LCS/QC CHECK					
CCV					

CALCULATIONS:

$$RPD = \frac{|(\text{Sample} - \text{Sample Dup.})|}{\text{Avg., Sample + Dup.}} \times 100$$

Comments:_____

Secondary Review By:_____ Date:_____

ATTACHMENT IV

TITRANT STANDARDIZATION

DATE:_____ Analyst:_____

TITRANT:_____ Date Prepared:_____

STANDARD:_____ Std Concentration:_____ Std #:_____

ID	mL Std	Titrant V_I (mL)	Titrant V_F (mL)	Titrant V_T (mL)	Conc (meq/mL)
Blk					
A					
B					
C					
Ave. A, B & C					

Concentration:

meq/mL =

DATE:_____ Analyst:_____

TITRANT:_____ Date Prepared:_____

STANDARD:_____ Std Concentration:_____ Std #:_____

ID	mL Std	Titrant V_I (mL)	Titrant V_F (mL)	Titrant V_T (mL)	Conc (meq/mL)
Blk					
A					
B					
C					
Ave. A, B & C					

Concentration:

meq/mL =

Comments:_____

Secondary Review:_____ Date:_____

ATTACHMENT B

CORRECTIVE ACTION SUMMARY

TABLE B-1

CORRECTIVE ACTIONS SUMMARY FOR METHOD TO-13

Method	Parameter	Calibration	Frequency	Acceptance Criteria	Corrective Action
TO-13 (GC/MS)	Semi-volatile Organics	Check of instrument tuning criteria using DFTPP	Every 12 hours	Refer to method narrative (SW846)	(1) Retune instrument (2) Repeat DFTPP analysis
		Multipoint calibration (minimum five points).	Initially and as required	% RSD for ≤ 30%; Avg. RF ≥ 0.050 for SPCCs	(1) Evaluate system (2) Recalibrate*
		Continuing calibration check standard	Every 12 hours	RF ≥ 0.050 for SPCCs; % Difference ≤ 30% for CCCs	(1) Evaluate system (2) Recalibrate* (3) Re-analyze samples run after the failed standard
		Method blank	Daily after calibration or LCS analyses	<RL of target compounds	(1) Re-analyze blank and repeat (2) Clean system (3) Evaluate system (a) Examine chromatography, integration parameters and calculations (b) Confirm integrity of solution (c) Check for interferences (d) Verify instrument settings and system operation (4) Re-analyze calibration standard once and compare to acceptance criteria

* Repeat Initial Calibration

TABLE B-2

CORRECTIVE ACTIONS SUMMARY FOR METHOD TO-14

Method	Parameter	Calibration	Frequency	Acceptance Criteria	Corrective Action
TO-14 (GC/MS)	Volatile Organic Compounds	Check of instrument tuning criteria using BFB	Every 12 hours	Refer to method narrative	(1) Retune instrument (2) Repeat BFB analysis
		Multipoint calibration (minimum three points)	Initially and as required	90% of target compounds ≤ 30% RSD. Min RF = 0.05	(1) Evaluate system (2) Repeat calibration
		Continuing calibration check standard	Every 12 hours	90% of target compounds ≤ 30% Diff; Min RF = 0.05	(1) Evaluate system (2) Repeat calibration check (3) Recalibrate* (4) Re-analyze affected samples
		Zero air blanks	Daily after calibration or LCS analyses	< RL of target compounds	(1) Run system blank, if necessary (2) Re-analyze air blank and repeat (3) Clean system (4) Evaluate System: • Examine Chromatography, integration parameters and calculations • Confirm integrity of solution • Check for interferences • Verify instrument settings and system operation (5) Reanalyze calibration standard once and compare to acceptance criteria.

* Repeat Initial Calibration

TABLE B-3

CORRECTIVE ACTION SUMMARY FOR METHOD SW846-8260A

Analytical Method	Parameter	QC Element	Frequency	Acceptance Criteria	Corrective Action
SW-846, 8260A; (GC/MS)	Volatile Organic Compounds	Tune instrument with a 4-bromofluorobenze standard	Every 12 hours	Must meet key ions and ion abundance criteria in Section 13.6 of Doc. #MSS01100.NC	• Return instrument, • Repeat standard analysis
		Initial multi-point calibration	Prior to analysis. and as required, determined by results of other standard analysis.	% relative standard deviation for CCC ≤ 30%; Average RF ≥ 0.30 (0.25 for CHBr3) for SPCC	• Evaluate the system • Repeat calibration
		Continuing calibration verification (CCV)	Every 12 hours	% difference ≤ 20% for CCC; RF ≥ 0.30 (0.25 for CHBr3) for SPCC	• Evaluate the system • Repeat calibration check standard • Evaluate standard • Repeat the initial calibration as necessary • Reanalyzed affected sample
		System blank	As required	< RL	• Run until system is clean • Base out system • Perform maintenance
		Method blanks	1 per 12 hours	<PQL of target compounds	• Assess impact on data • Reanalyze method blank if necessary • Run a system blank • Reprep/analyze batch as necessary
		Surrogate spike	Every sample, method blank, LCS MS/MSD	% Recovery within project QC acceptance criteria	• Evaluate the system • Reanalyze the sample • Reextract if >1 surrogate fraction outside acceptance limits • Narrate all outliers

TABLE B-3

CORRECTIVE ACTION SUMMARY FOR METHOD SW846-8260A

Analytical Method	Parameter	QC Element	Frequency	Acceptance Criteria	Corrective Action
		Matrix spike	1 per instrument, per sample batch, per day; up to 20 reportable samples	% Recovery within project QC acceptance criteria	• Assess data (4x rule) • Reanalyze once, report both • Narrate all outliers
		Matrix spike duplicate	1 per instrument, per sample batch, per day; up to 20 reportable samples	% Recovery and RPD within project QC acceptance criteria.	• Same as MS
		Laboratory control sample	1 per instrument, per sample batch, per day; up to 20 reportable samples	% Recovery within project QC acceptance criteria for all target analytes	• Reanalyze LCS • Assess impact on data • Reprep/reanalyze batch as necessary • Narrate all outliers

TABLE B-4

CORRECTIVE ACTION SUMMARY FOR SW 846-8270B

Analytical Method	Parameter	QC Element	Frequency	Acceptance Criteria	Corrective Action
SW-846, 8270B GC/MS	Base/Neutral Acid Extractable	Tune the instrument using a deca-fluorotriphenylphosine standard	Every 12 hours	Must meet the ion abundance criteria in SW 846	• Retune instrument, • Repeat standard analysis
		Initial calibration (5 point minimum)	Prior to analysis and as required	% Relative standard deviation for CCC ≤ 30%; Average RF ≥0.05 for SPCC	• Evaluate the system • Repeat calibration
		Continuing calibration check	Every 12 hours	Percent difference for CCC ≤ 30%; RF ≥ 0.05 for SPCC.	• Evaluate system • Reanalyze calibration check standard • Evaluate standard • Repeat the initial calibration as necessary • Reanalyze affected samples
		Method blank	1 per preparation batch (≤ 20 samples)	<PQL; except 5x CRQL for common laboratory contaminants. Surrogate recoveries must be within control limits	• Assess impact on data • Reanalyze blank • Run a system blank if necessary • Reprep/analyze batch as necessary
		Surrogate spike	Every sample, method blank, LCS and MS/MSD	No more than one surrogate per fraction outside of project acceptance criteria; No surrogate below 10% recovery	• Evaluate the system and data • Reanalyze the sample once • Reextract if >1 surrogate per fraction outside acceptance limits • Narrate all outliers
		Matrix spike	1 per 20 samples	% Recovery within project QC acceptance criteria	• Assess data (4x rule) • Reanalyze once, report both sets • Narrate all outliers
		Matrix spike duplicate	1 per 20 samples	% Recovery and RPD within project QC acceptance criteria	• Same as MS
		Laboratory control sample	1 per preparation batch (≤ 20 samples)	% Recovery within project QC acceptance criteria for all spiked analytes	• Reanalyze LCS • Assess impact on data • Reprep/reanalyze batch as needed • Narrate all outliers

TABLE B-5

CORRECTIVE ACTION SUMMARY FOR METHOD SW 846-8081

Analytical Method	Parameter	QC Element	Frequency	Acceptance Criteria	Corrective Action
SW-846, 8081 GC	Organochlorine Pesticides and PCBs	Initial multipoint calibration	Initially and as required	%RSD ≤ 20% or correlation coefficient (r) ≥ 0.995 or % RSE≤ 20% to accept curve	Recalibrate
		Initial calibration verification (ICV)	Prior to sample analysis	± 15% of expected concentration	Reanalyze ICV Recalibrate Reanalyze affected samples
		Continuing calibration verification (CCV)	Every 10 samples and end of run sequence	± 15% of expected concentration for each standard bracketing samples	Reanalyze CCV Reanalyze all affected samples Repeat initial calibration as necessary
		Degradation standard	Every 24 hours of analysis	Breakdown of endrin or 4,4'-DDT < 20%	Evaluate system Perform system maintenance Recalibrate as necessary
		Method blank	1 per preparation batch (≤20 samples)	< RL	Assess impact on data Reanalyze method blank Reprep/reanalyze batch as necessary
		System blank	As required	< RL	Run until system is clean Bake out system Perform maintenance
		Surrogate spike	Every sample, standard, method blank, LCS and MS/MSD	% recovery within acceptance windows for a minimum of one of two surrogate spikes	Assess data Reanalyze sample once Evaluate system Narrate all outliers

TABLE B-5

CORRECTIVE ACTION SUMMARY FOR METHOD SW 846-8081

Analytical Method	Parameter	QC Element	Frequency	Acceptance Criteria	Corrective Action
SW-846, 8081 GC (continued)		Matrix spike	1 per 20 samples	% Recovery within project QC acceptance limits	Assess data (4x rule) Reanalyze once, report both sets Narrate all outliers
		Matrix spike duplicate	1 per 20 samples	% Recovery and RPD within project QC acceptance limits	Same as MS
		Laboratory control sample	1 per preparation batch (≤20 samples)	% Recovery within project QC acceptance criteria for all spiked analytes	Reanalyze LCS Assess impact on data Reprep/reanalyze batch as necessary Narrate all outliers

TABLE B-6

CORRECTIVE ACTION SUMMARY FOR METHOD SW846-8151

Analytical Method	Parameters	QC Element	Frequency	Acceptance Criteria	Corrective Action
SW846, 8151 GC/ECD	Pentachlorophenol	Initial multipoint calibration (5 point minimum)	Initially and as required	%RSD < 20% or correlation coefficient (r) $\geq$ 0.995	• Evaluate system • Recalibrate
		Initial calibration verification (ICV)	After calibration and prior to sample analysis	± 15% difference from expected concentration	• Evaluate system • Reanalyze ICV • Recalibrate
		Continuing calibration verification (CCV)	Each day and end of run sequence, plus each 10th sample	± 15% difference from expected concentration for each standard bracketing samples	• Evaluate system • Reanalyze CCV • Recalibrate if necessary • Reanalyze affected samples
		Method blank	1 per preparation batch ($\leq$20 samples)	< Reporting Limit	• Assess impact on data • Reanalyze method blank • Reprep/reanalyze batch as necessary
		System blank	As required	< Reporting Limit	• Run until system is clean • Bake out system • Perform maintenance
		Surrogate spike	Every sample, standard, method blank, LCS, and MS/MSD	% Recovery within project QC acceptance criteria	• Assess data • Reanalyze sample once • Evaluate system • Narrate all outliers
		Matrix spike (MS)	1 per preparation batch ($\leq$20 samples)	% Recovery within project QC acceptance criteria	• Assess data (4x rule) • Reanalyze once, report both sets • Narrate all outliers

TABLE B-6

CORRECTIVE ACTION SUMMARY FOR METHOD SW846-8151

Analytical Method	Parameters	QC Element	Frequency	Acceptance Criteria	Corrective Action
SW846, 8151 GC/ECD (continued		Matrix spike duplicate (MSD)	1 per preparation batch (≤20 samples)	% Recovery and RPD within project QC acceptance criteria	• Same as matrix spike
		Laboratory control sample (LCS)	1 per preparation batch (≤20 samples)	% Recovery within project QC acceptance criteria for all spiked analytes	• Reanalyze LCS • Assess impact on data • Reprep/reanalyze batch as necessary • Narrate all outliers

TABLE B-7

DIOXIN/FURAN CORRECTIVE ACTION SUMMARY

Analytical Method	Parameter	QC Element	Frequency	Acceptance Criteria	Corrective Action
SW-8290 (GC/MS)	Dioxins/Furans	Tune using PFK	Once per 12 hours, prior to sample analysis	Resolving power ≥ 10,000 at m/z=304.9824 and m/x 380.9760 ≤5 ppm of expected mass	• Retune instrument • Reanalyze PFK
		Window defining mix (WDM) Column Performance Check Solution (CPSM)	Prior to ICAL, once per 12 hours prior to sample analysis	Used to set retention times. CPSM must have ≤25% valley resolution for 2378-TCDD	• Readjust windows • Evaluate system • Perform maintenance • Reanalyze WDN/CPSN
		Multipoint calibration (5 points, ICAL)	Initially and as required	Int std = %RSD≤30% Natives - %RSD≤20% Retention time must be within -1 to +3 seconds of labeled IS or 0.005 RRT units. Ion ratios within Table A limits, and S/N ≥ 2.5	• Evaluate system • Recalibrate
		Daily continuing calibration standard (CCAL)	Once per 12 hours, prior to sample analysis	%D of IS ≤30% from avg RRF (ICAL). %D of natives ≤29% from avg RRD (ICAL). RT must be within -1 to +3 seconds of labeled IS or 0.005 RRT units. Ion ratios are within Table A limits	• Evaluate system • Reanalyze CCAL • Recalibrate (ICAL) as necessary
SW-8290 (GC/HRMS)	Dioxins/Furans	Internal standards	Every sample, method blank, and LCS	Internal standard recovery within limits stated in Tables 2 and 3	• Check chromatogram for interference. If found, flag data • Check instrument and reanalyze the extract if a problem is found and corrected • Check S/W. If <10:1, reextract sample • Evaluate data usability and flag as appropriate • Reextract and reanalyze adversely affected samples
		Method blank	One per analytical batch, not to exceed 20 samples, per matrix	Ratio of a given unlabeled PCDD/PCDF isomer ≤5% of the appropriate internal standard	• Reanalyze method blank • If still exceeds and analyte concentration in sample <CRQL or >10x blank concentration, report results
		LCS (include natives)	At a frequency of 5% (1 per 20 samples analyzed)	Refer to Table 3	• Review Internal Standards, as above • Evaluate data for usability • If sample results are ND and CRQLs are met, no action required
		Duplicates	As per client request	Refer to Table 2 for internal std., RPD criteria	• Review Internal Standards, as above • If RPD exceeds, reinject extract • Narrate any outliers.
		Matrix spike	As per client request	Refer to Tables 2 and 3	• Review data for usability • Narrate outliers
		Matrix spike duplicate	As per client request	Refer to Tables 2 and 3	• Review data for usability • Narrate outliers

TABLE B-8

CORRECTIVE ACTION SUMMARY FOR ICP METALS AND COLD VAPOR ATOMIC ABSORPTION ANALYSIS

Analytical Method	Parameter	QC Element	Frequency	Acceptance Criteria	Corrective Action
METALS					
Inductively Coupled Plasma (ICP) and Cold Vapor Atomic Absorption (CVAA)		Initial laboratory mixed standard multipoint calibration	Daily prior to analysis	$r \geq 0.995$	Recalibrate
		Calibration blank	After initial calibration and each subsequent calibration	< PQL	Clean system Rerun blank
		ICP interference check	Beginning of daily run, after 8 hours, and/or end of run sequence	80-120% of true value for EPA check sample elements	Verify calibration Verify IEC and update as necessary Recalibrate
		Initial calibration verification (ICV)	After calibration, prior to sample analysis	± 10% expected concentration	Reanalyze ICV Recalibrate
		Continuing calibration verification (CCV)	Every 10 samples and end of run sequence	± 10% expected concentration	Reanalyze CCV Recalibrate Reanalyze affected samples back to the last acceptable CCV
		Method blank	1 per preparation batch (≤20 samples)	< PQL	Reanalyze Assess impact on data Redigest/reanalyze samples if blank fails twice
		Matrix spike	1 per 20 samples	% Recovery within project QC acceptance criteria	Assess data (4x rule) Reanalyze once, report both sets Narrate all outliers
		Matrix duplicate	1 per 20 samples	RPD within project QC acceptance criteria	Same as MS
		Laboratory control sample	1 per preparation batch (≤20 samples)	% Recovery within project QC acceptance criteria	Reanalyze LCS Assess impact on data Redigest/reanalyze affected samples Narrate all outliers

TABLE B-9

WET CHEMISTRY ANALYSES
CORRECTIVE ACTION SUMMARY

Analytical Method	Parameter	QC Element	Frequency	Acceptance Criteria	Corrective Action
SW-846, 7196 Colorimetric	Hexavalent Chromium	Initial multipoint calibration	Initially and as required	Correlation coefficient (r) ≥ 0.995	Recalibrate
EPA 351.2	Total Kjeldahl Nitrogen (TKN)	Initial calibration verification (ICV)	Daily, prior to sample analysis	± 15% expected concentration	Reanalyze ICV Recalibrate as necessary
		Continuing calibration verification (CCV)	Every 10 samples and end of run sequence	± 15% expected concentration	Reanalyze ICV Recalibrate as necessary Reanalyze all affected samples
		Calibration blank	Daily prior to sample analysis, and after each ICV and CCV	< PQL	Reanalyze blank Reanalyze samples back to last clean blank
		Method blank	1 per preparation batch (≤20 samples)	< PQL	Assess impact on data Reanalyze blank Redigest/reanalyze affected samples
		Matrix spike	1 per 20 samples	% Recovery within project QC acceptance criteria	Assess data (4x rule) Reanalyze once, report both sets Narrate all outliers
		Matrix duplicate	1 per 20 samples	RPD within project QC acceptance criteria	Same as MS
		Laboratory control sample	1 per preparation batch (≤20 samples)	% Recovery within project QC criteria	Reanalyze LCS Assess impact on data Reprep/reanalyze samples as necessary Narrate all outliers
EPA 325.2 EPA 365.1 EPA 365.2	Chloride O-phosphorous Total phosphorous	Initial multipoint calibration	Initially and as required	Correlation coefficient (r) ≥ 0.995	Check calculations Recalibrate
EPA 353.2	Nitrate Nitrite	Initial calibration verification (ICV)	Prior to sample analysis	± 15% expected concentration	Reanalyze ICV Recalibrate as necessary
EPA 340.2	Fluoride	Continuing calibration verification (CCV)	Every 10 samples and end of run	± 15% expected concentration	Reanalyze CCV Recalibrate as necessary
EPA 350.3 EPA 375.4 EPA 150.1 EPA 377.1 EPA 305.1	Ammonia Sulfate pH Sulfide Acidity	Calibration blank	Daily and after each ICV and CCV	< RL	Reanalyze blank Clean system Reanalyze samples back to last clean blank
		Method Blank	1 per preparation batch (≤20 samples)	< RL	Reanalyze reference standard Reanalyze ICV Recalibrate as necessary
		Matrix spike	1 per 20 samples	% Recovery within project QC acceptance criteria	Assess data (4x rule) Reanalyze once, report both sets Narrate all outliers

TABLE B-9

WET CHEMISTRY ANALYSES
CORRECTIVE ACTION SUMMARY

Analytical Method	Parameter	QC Element	Frequency	Acceptance Criteria	Corrective Action	Re
		Matrix duplicate	1 per 20 samples	RPD within project QC acceptance criteria	Same as MS	
		Laboratory control sample	1 per preparation batch (≤20 samples)	% Recovery within project QC acceptance criteria	Assess impact on data Reanalyze LCS Reprep/reanalyze samples as necessary Narrate all outliers	
		Method blank	1 per preparation batch (≤20 samples)	<PQL	Assess impact on data Reanalyze blank Reprep and analyze as necessary	
EPA 415.1	Total Organic Carbon	(1) Two point calibration	(1) Daily	(1) Measured value within ±0.1 pH unit	(1a) Recalibrate (1b) See Instrument Manual	
		(2) Acid standardization	(2) Daily	(2) Duplicate standardizations agree to +/- 0.2 ml	(2a) Recheck Na_2CO_3 standard prepare fresh standard solution if necessary (2b) Prepare fresh acid solutions	
		Method blank	1 per preparation batch	≤ RL	(1) Assess impact on data (2) Reanalyze blank if necessary (3) Reprep/reanalyze as necessary	
		Laboratory control sample	1 per batch	Refer to Table C-9	(1) Assess impact on data (2) Reanalyze LCS (3) Reprep/reanalyze as necessary	
		Matrix spike	5%	Refer to Table C-9	(1) Assess data (4x rule) (2) Reanalyze once, if necessary (3) Report as footnoted	
		Matrix duplicate	5%	Refer to Table C-9	Same as matrix spike	

ATTACHMENT C

PRACTICAL QUANTITATION LIMITS

TABLE C-1

PRACTICAL QUANTITATION LIMITS FOR MODIFIED METHOD TO-13

Parameter	Method	Analyte	Air (µg/sample)
Semivolatile Organic Compounds	Mod TO-13	**Base/Neutral Extractables**	
		Acenaphthene	10
		Acenaphthylene	10
		Anthracene	10
		Benzo (a) anthracene	10
		Benzo (b) fluoranthene	10
		Benzo (k) fluoranthene	10
		Benzo (g,h,i) perylene	10
		Benzo (a) pyrene	10
		Benzyl alcohol	10
		Bis (2-chloroethoxy) methane	10
		Bis(2-ethylhexyl)phthalate	10
		Bis(2-chloroethyl)ether	10
		Bis(2-chloroisopropyl)ether	10
		4-Bromophenyl phenyl ether	10
		Butyl benzyl phthalate	10
		4-Chloroaniline	10
		2-Chloronaphthalene	10
		4-Chlorophenyl phenyl ether	10
		Chrysene	10
		Dibenz (a,h) anthracene	10
		Dibenzofuran	10
		Di-n-butylphthalate	10
		1,2-Dichlorobenzene	10
		1,3-Dichlorobenzene	10
		1,4-Dichlorobenzene	10
		3,3'-Dichlorobenzidine	20
		Diethyl phthalate	10
		Dimethyl phthalate	10
		2,4-Dinitrotoluene	10
		2,6-Dinitrotoluene	10
		Di-n-octyl phthalate	10
		Fluoranthene	10
		Fluorene	10
		Hexachlorobenzene	10
		Hexachlorobutadiene	10
		Hexachlorocyclopentadiene	10
		Hexachloroethane	10
		Indeno(1,2,3-cd) pyrene	10
		Isophorone	10
		2-Methylnaphthalene	10
		Naphthalene	10
		2-Nitroaniline	10
		3-Nitroaniline	50

TABLE C-1

PRACTICAL QUANTITATION LIMITS FOR MODIFIED METHOD TO-13

Parameter	Method	Analyte	Air (µg/sample)
Semivolatile Organic Compounds (continued)	Mod TO-13	4-Nitroaniline	50
		Nitrobenzene	10
		N-Nitrosodiphenylamine	10
		N-Nitrosodipropylamine	10
		Phenanthrene	10
		Pyrene	10
		1,2,4-Trichlorobenzene	10
		Acid Extractables	
		4-Chloro-3-methylphenol	10
		2-Chlorophenol	10
		2,4-Dichlorophenol	10
		2,4-Dimethylphenol	10
		4,6-Dinitro-2-methylphenol	50
		2,4-Dinitrophenol	50
		2-Methylphenol	10
		4-Methylphenol	10
		2-Nitrophenol	10
		4-Nitrophenol	50
		Pentachlorophenol	50
		Phenol	10
		2,4,5-Trichlorophenol	50
		2,4,6-Trichlorophenol	10

TABLE C-2

PRACTICAL QUANTITATION LIMITS FOR METHOD TO-14

Parameter	Method	Analyte	PPBV
Volatile Organic Compounds	TO-14 (Air)	Dichlorodifluoromethane	2
		Chloromethane	4
		1,2-Dichloro-1,1,2,2-Tetrafluoroethane	2
		Vinyl Chloride	2
		Bromomethane	2
		Chloroethane	4
		Trichlorofluoromethane	2
		1,1-Dichloroethene	2
		Carbon Disulfide	10
		1,1,2-Trichloro-1,2,2-Trifluoroethane	2
		Acetone	10
		Methylene Chloride	2
		trans-1,2-Dichloroethene	2
		1,1-Dichloroethane	2
		Vinyl Acetate	10
		cis-1,2-Dichloroethene	2
		2-Butanone	10
		Chloroform	2
		1,1,1,-Trichloroethane	2
		Carbon Tetrachloride	2
		Benzene	2
		1,2-Dichloroethane	2
		Trichloroethene	2
		1,2-Dichloropropane	2
		Bromodichloromethane	2
		cis-1,3-Dichloropropene	2
		4-Methyl-2-pentanone	10
		Toluene	2
		trans-1,3-Dichloropropene	2
		1,1,2-Trichloroethane	2
		Tetrachloroethene	2
		2-Hexanone	30
		Dibromochloromethane	2
		1,2-Dibromoethane (EDB)	2
		Chlorobenzene	2
		Ethylbenzene	2
		Total Xylenes	2
		Styrene	2
		Bromoform	2
		1,1,2,2-Tetrachloroethane	2
		Benzyl Chloride	10
		4-Ethyl Toluene	2
		1,3,5-Trimethylbenzene	2
		1,2,4-Trimethylbenzene	2
		1,3-Dichlorobenzene	2
		1,4-Dichlorobenzene	2
		1,2-Dichlorobenzene	2
		1,2,4-Trichlorobenzene	20
		Hexachlorobutadiene	4

PPBV Parts by billion by volume

TABLE C-3

VOLATILE ORGANIC COMPOUNDS ANALYSIS REPORTING LIMITS

Parameter	Analytical Method	Analyte	Water (25 ml purge) MDL µg/l	Water (25 ml purge) PQL µg/l	Soil MDL µg/kg	Soil PQL µg/kg
Volatile Organic Compounds	SW8260A	Acetone	2.80	5	4.4	50
		Acrylonitrile	0.27	1	1.4	5
		Allyl chloride	0.20	1	2.3	5
		Benzene	0.04	1	1.9	5
		Bromobenzene	0.05	1	0.6	5
		Bromochloromethane	0.19	1	1.0	5
		Bromodichloromethane	0.12	1	0.9	5
		Bromoform	0.11	1	0.7	5
		Bromomethane	0.25	1	2.3	10
		2-Butanone	2.30	5	5.1	10
		n-Butylbenzene	0.07	1	0.8	5
		tert-Butylbenzene	0.12	1	0.9	5
		sec-Butylbenzene	0.10	1	0.9	5
		Carbon disulfide	0.15	1	1.1	5
		Carbon tetrachloride	0.12	1	1.2	5
		Chlorobenzene	0.04	1	1.2	5
		Chlorodibromomethane	0.12	1	0.6	5
		Chloroethane	0.24	1	6.4	10
		2-Chloroethylvinyl ether	0.21	1	2.6	10
		Chloroform	0.17	1	1.0	5
		Chloromethane	0.12	1	1.6	10
		2-Chlorotoluene	0.12	1	0.6	5
		4-Chlorotoluene	0.17	1	0.8	5
		1,2-Dibromo-3-chloropropane	0.35	1	2.1	5
		1,2-Dibromoethane	0.15	1	1.3	5
		Dibromomethane	0.15	1	1.0	5
		1,2-Dichlorobenzene	0.15	1	0.7	5
		1,3-Dichlorobenzene	0.12	1	0.6	5
		1,4-Dichlorobenzene	0.06	1	0.8	5
		Dichlorodifluoromethane	0.12	1	1.6	10
		1,1-Dichloroethane	0.12	1	1.0	5
		1,2-Dichloroethane	0.17	1	1.1	5
		1,1-Dichloroethene	0.06	1	1.6	5
		cis-1,2-Dichloroethene	0.12	1	1.0	5
		trans-1,2-Dichloroethene	0.12	1	1.2	5
		1,2-Dichloroethene (Total)	0.24	1	NA	NA
		1,3-Dichloropropane	0.15	1	1.7	5
		2,2-Dichloropropane	0.15	1	1.6	5
		1,1-Dichloropropene	0.12	1	1.1	5
		cis-1,3-Dichloropropene	0.15	1	0.8	5
		trans-1,3-Dichloropropene	0.12	1	1.4	5
		cis-1,4-Dichloro-2-butene	0.31	1	2.6	5
		trans-1,4-Dichloro-2-butene	0.63	1	2.0	5
		Ethyl benzene	0.04	1	1.0	5
		Ethyl methacrylate	0.24	1	1.9	5
		Hexachlorobutadiene	0.05	1	1.3	5
		2-Hexanone	2.59	5	1.7	10
		Iodomethane	0.13	1	0.6	5
		Isopropylbenzene	0.18	1	0.8	5
		p-Isopropyltoluene	0.11	1	0.9	5
		Methacrylonitrile	0.86	1	2.9	5
		Methylene chloride	0.43	1	6.6	10
		Methyl methacrylate	0.31	1	1.1	5
		4-Methyl-2-pentanone	3.06	5	1.9	10

TABLE C-3

VOLATILE ORGANIC COMPOUNDS ANALYSIS REPORTING LIMITS

Parameter	Analytical Method	Analyte	Water (25 ml purge) MDL µg/l	Water (25 ml purge) PQL µg/l	Soil MDL µg/kg	Soil PQL µg/kg
Volatile Organic Compounds (continued)	SW8260A	Methyl-tert-butyl ether	0.15	1	1.6	5
		Naphthalene	0.14	1	1.2	5
		Pentachloroethane	0.13	1	0.8	5
		n-Propylbenzene	0.08	1	0.9	5
		Styrene	0.17	1	1.2	5
		1,2,3-Trichlorobenzene	0.13	1	1.1	5
		1,2,4-Trichlorobenzene	0.05	1	0.4	5
		1,1,1,2-Tetrachloroethane	0.06	1	0.8	5
		1,1,2,2-Tetrachloroethane	0.11	1	1.5	5
		Tetrachloroethane	0.07	1	0.6	5
		Toluene	0.08	1	0.7	5
		1,1,1-Trichloroethane	0.12	1	0.8	5
		1,1,2-Trichloroethane	0.17	1	1.4	5
		Trichloroethene	0.12	1	1.5	5
		Trichlorofluoromethane	0.07	1	0.6	5
		1,2,3-Trichloropropane	0.40	1	1.4	5
		1,3,5-Trimethylbenzene	0.04	1	0.9	5
		Vinyl acetate	0.28	1	1.6	10
		Vinyl chloride	0.08	1	1.2	10
		o-Xylene	0.07	1	0.5	5
		m,p-Xylene	0.10	2	0.7	5
		Xylenes (Total)	0.16	5	1.1	5

NA Not available; MDL study not required by SW-846
µg/l Microgram per liter
µg/kg Microgram per kilogram

TABLE C-4

BASE/NEUTRAL AND ACID EXTRACTABLE ANALYSIS REPORTING LIMITS

Parameter	Analytical Method	Analyte	Water PQL (µg/l)	Water MDL (µg/l)	Soil PQL (µg/kg)	Soil MDL (µg/kg)
Base/neutral, and acid extractables (BNAE)	SW8270B	Acenaphthene	10	0.77	330	41.4
		Acenaphthylene	10	0.70	330	28.4
		Acetophenone	10	NA	330	82.4
		4-Aminobiphenyl	10	NA	660	44.5
		Aniline	10	NA	330	77.6
		Anthracene	10	0.48	330	34.2
		Benzidine	10	1.75	330	25.9
		Benzo (a) anthracene	10	0.50	330	39.6
		Benzo (b) fluoranthene	10	1.92	330	47.6
		Benzo (k) fluoranthene	10	2.18	330	53.0
		Benzo (g,h,i) perylene	10	0.65	330	34.2
		Benzo (a) pyrene	10	0.76	330	39.8
		Benzyl alcohol	20	1.87	660	39.9
		Bis (2-chloroethoxy) methane	10	0.53	330	30.2
		Bis(2-ethylhexyl) phthalate	10	2.10	330	48.6
		Bis(2-chloroethyl) ether	10	0.79	330	41.4
		Bis(2-chloroisopropyl) ether	10	2.63	330	33.5
		4-bromophenyl phenyl ether	10	0.41	330	36.5
		Butyl benzyl phthalate	10	5.71	330	231.1
		4-chloroaniline	20	0.67	660	47.6
		2-chloronaphthalene	10	0.83	330	36.1
		4-chlorophenyl phenyl ether	10	0.57	330	31.8
		Chrysene	10	0.47	330	37.0
		Dibenz (a,h) anthracene	10	0.74	330	43.2
		Dibenzofuran	10	0.66	330	31.8
		Di-n-butyl phthalate	10	3.20	330	223.1

TABLE C-4

BASE/NEUTRAL AND ACID EXTRACTABLE ANALYSIS REPORTING LIMITS

Parameter	Analytical Method	Analyte	Water PQL (µg/l)	Water MDL (µg/l)	Soil PQL (µg/kg)	Soil MDL (µg/kg)
Base/neutral, and acid extractables (BNAE) (continued)	SW8270B	1,2-dichlorobenzene	10	1.03	330	28.8
		1,3-dichlorobenzene	10	1.09	330	23.1
		1,4-dichlorobenzene	10	1.04	330	28.5
		3,3-dichlorobenzidine	20	0.62	660	37.2
		Diethyl phthalate	10	3.18	330	38.6
		p-Dimethylaminoazobenzene	10	NA	330	93.7
		7,12-Dimethbenz(a)anthracene	10	NA	330	82.2
		Dimethyl phthalate	10	2.16	330	37.1
		2,4-dinitrotoluene	10	0.97	330	37.0
		2,6-dinitrotoluene	10	0.43	330	32.7
		Di-n-octyl phthalate	10	0.49	330	44.2
		Azobenzene	10	0.43	330	28.8
		Ethyl methane sulfonate	10	NA	330	NA
		Fluoranthene	10	0.68	330	35.9
		Fluorene	10	0.68	330	34.4
		Hexachlorobenzene	10	0.57	330	27.4
		Hexachlorobutadiene	10	1.68	330	27.3
		Hexachlorocyclopentadiene	10	NA	330	36.4
		Hexachloroethane	10	1.31	330	33.7
		Indeno (1,2,3-cd) pyrene	10	0.54	330	33.1
		Isophorone	10	0.46	330	34.5
		2-methylnaphthalene	10	0.86	330	34.4
		Naphthalene	10	0.80	330	32.7
		1-Napthylamine	10	NA	330	135.1
		2-Napthylamine	10	NA	330	149.3

TABLE C-4

BASE/NEUTRAL AND ACID EXTRACTABLE ANALYSIS REPORTING LIMITS

Parameter	Analytical Method	Analyte	Water PQL (µg/l)	Water MDL (µg/l)	Soil PQL (µg/kg)	Soil MDL (µg/kg)
Base/neutral, and acid extractables (BNAE) (continued)	SW8270B	2-nitroaniline	50	1.07	1600	49.6
		3-nitroaniline	50	0.74	1600	29.4
		4-nitroaniline	20	0.50	1600	35.6
		Nitrobenzene	10	0.53	330	29.9
		N-nitrosodimethylamine	10	0.96	330	48.4
		N-nitroso-di-n-butylamine	10	NA	330	97.3
		N-nitrosodiphenylamine	10	0.63	330	21.4
		N-nitroso-di-n-propylamine	10	0.89	330	37.0
		N-nitrosopiperidine	20	NA	660	89.6
		Phenanthrene	10	0.42	330	35.1
		Pentachlorobenzene	10	NA	330	102.1
		Pentachloronitrobenzene	20	NA	660	96.8
		Phenacetin	20	NA	660	107.3
		2-Picoline	10	NA	330	60.5
		Pronamide	10	NA	330	107.5
		Pyrene	10	0.66	330	40.5
		1,2,4,5 Tetrachlorobenzene	10	NA	330	99.9
		1,2,4-trichlorobenzene	10	1.29	330	29.8
		<u>Acid Extractables</u>				
		Benzoic acid	50	NA	1600	NA
		4-chloro-3-methylphenol	20	0.66	660	30.4
		2-chlorophenol	10	0.69	330	31.4
		2,4-dichlorophenol	10	0.53	330	37.6
		2,6-dichlorophenol	10	NA	330	NA
		2,4-dimethylphenol	10	2.16	330	33.0

TABLE C-4

BASE/NEUTRAL AND ACID EXTRACTABLE ANALYSIS REPORTING LIMITS

Parameter	Analytical Method	Analyte	Water PQL (µg/l)	Water MDL (µg/l)	Soil PQL (µg/kg)	Soil MDL (µg/kg)
Base/neutral, and acid extractables (BNAE) (continued)	SW8270B	4,6-dinitro-2-methylphenol	50	0.81	1600	45.2
		2,4-dinitrophenol	50	2.38	1600	78.9
		2-methylphenol	10	0.93	330	31.0
		4-methylphenol	10	0.87	330	32.4
		2-nitrophenol	50	0.52	330	34.7
		4-nitrophenol	50	0.80	1600	45.9
		Pentachlorophenol	50	1.55	1600	45.1
		Phenol	10	0.71	330	42.3
		2,3,4,6-tetrachlorophenol	10	NA	330	107.8
		2,4,5-trichlorophenol	10	0.56	330	39.0
		2,4,6-trichlorophenol	10	0.74	330	29.4

NA Not available; MDL study not required by SW-846
µg/l Microgram per liter
µg/kg Microgram per kilogram

TABLE C-5

ORGANOCHLORINE PESTICIDES AND PCBS ANALYSIS REPORTING LIMITS

Parameter	Analytical Method	Analyte	Water PQL (µg/l)	Water MDL (µg/l)	Soil PQL (µg/kg)	Soil MDL (µg/kg)
Organochlorine Pesticides and PCBs	SW8081	Aldrin	0.050	0.012	8.0	5.37
		alpha-BHC	0.050	0.024	8.0	0.67
		beta-BHC	0.050	0.016	8.0	0.19
		delta-BHC	0.050	0.012	8.0	0.34
		Lindane	0.050	0.022	8.0	0.69
		Chlordane(technical chlordane)	0.50	0.024	80	NA
		4,4'-DDD	0.10	0.051	16	0.69
		4,4'-DDE	0.10	0.026	16	0.21
		4,4'-DDT	0.10	0.049	16	1.01
		Dieldrin	0.10	0.046	16	1.31
		Endosulfan I	0.050	0.018	8.0	0.86
		Endosulfan II	0.10	0.031	16	2.72
		Endosulfan sulfate	0.10	0.025	16	0.22
		Endrin	0.10	0.047	16	1.31
		Endrin aldehyde	0.10	0.042	16	1.10
		Heptachlor	0.050	0.030	8.0	0.82
		Heptachlor epoxide	0.050	0.012	8.0	0.42
		Methoxychlor	0.50	0.22	80	7.30
		Toxaphene	5.0	1.3	160	44.4
		PCB-1016	0.50	0.31	80	2.12
		PCB-1221	0.50	0.18	80	41.6
		PCB-1232	0.50	0.18	80	5.88
		PCB-1242	0.50	0.07	80	5.55
		PCB-1254	1.0	0.12	160	4.37
		PCB-1260	1.0	0.90	160	4.84

NA Not available; MDL study not required by SW-846
µg/l Microgram per liter
µg/kg Microgram per kilogram

TABLE C-6

CHLORINATED HERBICIDES AND PENTACHLOROPHENOL ANALYSIS REPORTING LIMITS

Parameter	Analytical Method	Analyte	Water (µg/l)	Soil (µg/kg)
Chlorinated Herbicides	SW 8151	Pentachlorophenol	1.0	NA

NA Not analyzed
µg/l Microgram per liter
µg/kg Microgram per kilogram

TABLE C-7

DIOXIN/FURAN REPORTING LIMITS

Parameter	Analytical Method	Analyte	Water[a] (pg/l)	Soil[b] (pg/g)
Dioxins/Furans	SW-8290	**DIOXINS**		
		TCDDs (Total)	0.80	0.21
		2,3,7,8-TCDD	0.80	0.21
		PeCDDs (Total)	1.3	2.4
		1,2,3,7,8-PeCDD	1.3	0.32
		HxCDDs (Total)	2.2	0.33
		1,2,3,4,7,8-HxCDD	2.0	0.33
		1,2,3,6,7,8-HxCDD	2.0	0.31
		1,2,3,7,8,9-HxCDD	2.3	0.33
		HpCDDs (Total)	2.0	0.28
		1,2,3,4,6,7,8-HpCDD	2.0	0.28
		OCDD	9.2	2.0
		FURANS		
		TCDFs (Total)	1.3	0.14
		2,3,7,8-TCDF	1.3	0.14
		PeCDFs (Total)	1.1	0.33
		1,2,3,7,8-PeCDF	1.1	0.33
		2,3,4,7,8-PeCDF	0.94	0.28
		HxCDFs (Total	2.0	0.25
		1,2,3,4,7,8-HxCDF	0.72	0.17
		1,2,3,6,7,8-HxCDF	0.57	0.21
		1,2,3,7,8,9-HxCDF	0.89	0.20
		2,3,4,6,7,8-HxCDF	2.6	0.25
		HpCDFs (Total)	3.9	0.20
		1,2,3,4,6,7,8-HpCDF	2.4	0.15
		1,2,3,4,7,8,9-HpCDF	3.9	0.20
		OCDF	6.1	0.27

pg/l Picogram per liter
pg/g Picogram per gram

(a) Based upon 1 liter sample aliquot. Sensitivity of the method depends on the level of interferences rather than instrument limitations. Typical waste samples may have higher reporting limits and may require additional cleanup techniques.

(b) Based upon 10 gram sample aliquot. Maximum CRQLs for samples "as received." Correction for moisture content may raise reporting limits above these levels. Typical waste samples may have higher reporting limits and may require additional cleanup techniques.

TABLE C-8

REPORTING LIMITS FOR TRACE METALS

Parameter	Analytical Method	Analyte	Water PQL (mg/l)	Water MDL (µg/l)	Soil PQL (mg/kg)	Soil MDL (mg/kg)
METALS	6010A	Antimony	.060	1.197	6.0	0.0006
	6010A	Arsenic	.010	1.335	1.0	0.0007
	6010A	Barium	0.200	.093	20	0.00005
	6010A	Beryllium	.005	0.112	0.5	0.0001
	6010A	Cadmium	.005	.171	0.5	0.0001
	6010A	Calcium	.500	161	500	16.2
	6010A	Chromium	.010	1.703	1	0.0002
	6010A	Iron	.100	9.71	10	0.0047
	6010A	Lead (Trace)	.003	1.064	0.3	0.001
	6010A	Magnesium	.500	3.71	500	0.0018
	6010A	Manganese	.015	0.238	1.5	0.0001
	7470/7471	Mercury	.002	0.06	0.1	0.00003
	6010A	Potassium	.500	17.2	500	0.0019
	6010A	Thallium	.010	2.074	1	0.001
	6010A	Selenium	.005	2.768	0.5	0.0007
	6010A	Silver	.010	.188	1	0.0002
	6010A	Sodium	5.0	561	500	0.28

mg/l Milligram per liter
µg/l Microgram per liter
mg/kg Milligram per kilogram

TABLE C-9

WET CHEMISTRY METHODS
REPORTING LIMITS

Analysis	Analytical[(a)] Method	Analyte	Water (mg/L)	Soil (mg/kg)
Common Anions	EPA 325.2	Chloride	0.4	10
	EPA 365.2	O-phosphorous	0.02	.2
	EPA 375.4	Sulfate	2.0	20
	EPA 365.2	T-phosphorous	0.02	0.2
	EPA 377.1	Sulfide	2.0	20
Nitrate/Nitrite	EPA 353.2	Nitrate	0.02	0.2
		Nitrite	0.02	0.2
Fluoride	EPA 340.2	Fluoride	0.1	1.0
Total Organic Carbon	EPA 415.1	TOC	1.0	100
Ammonia	EPA 350.3	Ammonia	0.1	10
Hexavalent chromium	SW846-7196	Hexavalent Chromium	0.010	2.0
Total Kjeldahl Nitrogen (TKN)	EPA 351.2	Total Kjeldahl Nitrogen (TKN)	0.1	100
Acidity	EPA 310.1	Acidity	10	10
pH	EPA 150.1/ SW846-9045	pH	0.1 (unit)	0.1 (unit)

mg/l Milligram per liter
mg/kg Milligram per kilogram

(a) The methods cited are from the following sources:

Methods for Chemical Analysis of Water and Wastes, EPA Manual, 600/4-79-020 (USEPA, 1983—with additions)

Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW-846, 3rd Edition (USEPA, 1986)

ATTACHMENT D

QUALITY CONTROL CRITERIA

TABLE D-1

SEMI-VOLATILE ORGANIC COMPOUND ANALYSIS FOR AIR QUALITY CONTROL CRITERIA FOR LABORATORY DATA EVALUATION

Analytical Method	Spiking Compounds	Laboratory-Established Control Limits		
		Spike Concentration (µg/sample) Air	Accuracy Recovery (%) Air	Precision (RPD %) Air
Laboratory Control Samples	Matrix			
VOC TO-13	Phenol	100	60-140	35
	2-Chlorophenol	100	60-140	35
	1,4-Dichlorobenzene	50	60-140	35
	N-Nitroso-di-n-propylamine	50	60-140	35
	1,2,4-Trichlorobenzene	50	60-140	35
	4-Chloro-3-methylphenol	100	60-140	35
	Acenaphthene	50	60-140	35
	4-Nitrophenol	100	60-140	35
	2,4-Dinitrotoluene	50	60-140	35
	Pentachlorophenol	100	60-140	35
	Pyrene	50	60-140	35
	Surrogate			
	Nitrobenzene-d5	50	46-121	NA
	2-Fluorobiphenyl	50	64-123	NA
	Terphenyl-d14	50	33-170	NA
	Phenol-d5	50	29-115	NA
	2-Fluorophenol	50	50-117	NA
	2,4,6-Tribromophenol	50	29-143	NA

NA Not applicable

TABLE D-2

VOLATILE ORGANIC COMPOUND ANALYSIS FOR AIR
QUALITY CONTROL CRITERIA FOR LABORATORY DATA EVALUATION

Analytical Method	Spiking Compounds	Laboratory-Established Control Limits		
		*Spike Concentration (ppbv) Air	Accuracy Recovery (%) Air	Precision (RPD %) Air
Laboratory Control Samples				
	Matrix			
VOC TO-14	Methylene chloride	50	86-116	10
	1,1-Dichloroethene	50	90-115	10
	Trichloroethene	50	85-114	10
	Toluene	50	92-114	10
	1,1,2,2-Tetrachloroethane	50	76-124	10

TABLE D-3

VOLATILE ORGANIC COMPOUND ANALYSIS
QUALITY CONTROL CRITERIA FOR LABORATORY DATA EVALUATION

Analytical Method	Spiking Compounds	Laboratory-Established Control Limits					
		Spike Concentration		Accuracy Percent Recovery (%)		Precision (RPD %)	
		Water (µg/l)	Soil/Sediments (µg/kg)	Water	Soil/Sediments	Water	Soil/Sediments
Matrix Spike/Matrix Spike Duplicate							
VOCs SW 8260A	Matrix						
	1,1-Dichloroethene	5.0	50	60-140	59-172	20	22
	Trichloroethene	5.0	50	60-140	62-137	20	24
	Benzene	5.0	50	60-140	66-142	20	21
	Toluene	5.0	50	60-140	59-139	20	21
	Chlorobenzene	5.0	50	60-140	60-133	20	21
	Surrogate Spikes						
	Toluene-d8	5.0	50	90-108	90-123	NA	NA
	Bromofluorobenzene	5.0	50	78-115	75-129	NA	NA
	1,2-Dichloroethane-d4	5.0	50	62-121	70-139	NA	NA
Laboratory Control Samples							
VOCs SW 8260A	Matrix						
	Bromomethane	5.0	20	10-200	10-200	NA	NA
	Vinyl chloride	5.0	20	10-200	10-200	NA	NA
	1,1-Dichloroethene	5.0	20	10-200	10-200	NA	NA
	trans-1,2-Dichloroethene	5.0	20	10-200	10-200	NA	NA
	cis-1,2-Dichloroethene	5.0	20	10-200	10-200	NA	NA
	1,1-Dichloroethane	5.0	20	10-200	10-200	NA	NA
	Chloroform	5.0	20	10-200	10-200	NA	NA
	1,2-Dichloroethane	5.0	20	10-200	10-200	NA	NA
	1,1,1-Trichloroethane	5.0	20	10-200	10-200	NA	NA
	Carbon tetrachloride	5.0	20	10-200	10-200	NA	NA
	Bromodichloromethane	5.0	20	10-200	10-200	NA	NA

TABLE D-3

VOLATILE ORGANIC COMPOUND ANALYSIS
QUALITY CONTROL CRITERIA FOR LABORATORY DATA EVALUATION

Analytical Method	Spiking Compounds	Laboratory-Established Control Limits					
		Spike Concentration		Accuracy Percent Recovery (%)		Precision (RPD %)	
		Water (µg/l)	Soil/Sediments (µg/kg)	Water	Soil/Sediments	Water	Soil/Sediments
	1,2-Dichloropane	5.0	20	10-200	10-200	NA	NA
	Trichloroethene	5.0	20	10-200	10-200	NA	NA
	1,1,2-Trichloroethane	5.0	20	10-200	10-200	NA	NA
	Benzene	5.0	20	10-200	10-200	NA	NA
	Tetrachloroethene	5.0	20	10-200	10-200	NA	NA
	Toluene	5.0	20	10-200	10-200	NA	NA
	Chlorobenzene	5.0	20	10-200	10-200	NA	NA
	Ethylbenzene	5.0	20	10-200	10-200	NA	NA
	m-and p-Xylenes	5.0	20	10-200	10-200	NA	NA
	o-Xylene	5.0	20	10-200	10-200	NA	NA
	1,3-Dichlorobenzene	5.0	20	10-200	10-200	NA	NA
	1,4-Dichlorobenzene	5.0	20	10-200	10-200	NA	NA
	1,2-Dichlorobenzene	5.0	20	10-200	10-200	NA	NA
	Surrogate Spikes						
	Toluene-d8	5.0	50	90-108	90-123	NA	NA
	Bromofluorobenzene	5.0	50	78-115	75-129	NA	NA
	1,2-Dichloroethane-d4	5.0	50	62-121	70-139	NA	NA

NA Not available

TABLE D-4

BASE/NEUTRAL AND ACID EXTRACTABLE ANALYSIS
QUALITY CONTROL CRITERIA FOR LABORATORY DATA EVALUATION

Analytical Method	Spiking Compounds	Spike Concentration		Laboratory-Established Control Limits			
				Accuracy Percent Recovery (%)		Precision (RPD %)	
		Water (µg/l)	Soil/Sediments (µg/kg)	Water	Soil/Sediments	Water	Soil/Sediments
Matrix Spike/Matrix Spike Duplicates							
BNAEs, SW 8270B	Matrix						
	Phenol	50	1,667	12-110	26-90	42	35
	2-Chlorophenol	50	1,667	27-123	25-102	40	50
	1,4-Dichlorobenzene	50	1,667	36-97	28-104	28	27
	n-Nitroso-di-n-propylamine	50	1,667	41-116	41-126	38	38
	1,2,4-Trichlorobenzene	50	1,667	39-98	38-107	28	23
	4-Chloro-3-methylphenol	50	1,667	23-97	26-103	42	33
	Acenaphthene	50	1,667	46-118	31-137	31	19
	4-Nitrophenol	50	1,667	10-80	11-114	50	50
	2,4-Dinitrotoluene	50	1,667	24-96	28-89	38	47
	Pentachlorophenol	50	1,667	9-103	17-109	50	47
	Pyrene	50	1,667	26-127	35-142	31	36
	Surrogate Spikes						
	Nitrobenzene-d5	50	1,667	35-114	23-120	NA	NA
	2-Fluorobiphenyl	50	1,667	43-116	30-115	NA	NA
	Terphenyl-d14	50	1,667	33-141	18-137	NA	NA
	Phenol-d5	75	2,500	10-94	24-113	NA	NA
	2-Fluorophenol	75	2,500	21-100	25-121	NA	NA
	2,4,6-Tribromophenol	75	2,500	10-123	19-122	NA	NA
Laboratory Control Samples							
BNAE SW 8270B	Phenol	50	1,700	30-112	10-112	NA	NA
	2-Chlorophenol	50	1,700	40-134	23-134	NA	NA
	1,4-Dichlorobenzene	50	1,700	40-124	61-90	NA	NA
	n-Nitroso-di-n-prop	50	1,700	40-138	52-93	NA	NA
	1,2,4-Trichlorobenzene	50	1,700	44-142	67-94	NA	NA
	4-Chloro-3-methylphenol	50	1,700	40-147	40-145	NA	NA
	Acenaphthene	50	1,700	47-145	63-92	NA	NA
	4-Nitrophenol	50	1,700	40-132	37-132	NA	NA

TABLE D-4

BASE/NEUTRAL AND ACID EXTRACTABLE ANALYSIS
QUALITY CONTROL CRITERIA FOR LABORATORY DATA EVALUATION

Analytical Method	Spiking Compounds	Spike Concentration		Laboratory-Established Control Limits			
				Accuracy Percent Recovery (%)		Precision (RPD %)	
		Water (µg/l)	Soil/Sediments (µg/kg)	Water	Soil/Sediments	Water	Soil/Sediments
Laboratory Control Samples (continued)							
BNAE SW 8270B							
	2,4-Dinitrotoluene	50	1,700	40-139	67-95	NA	NA
	Pentachlorophenol	50	1,700	40-150	29-152	NA	NA
	Pyrene	50	1,700	52-115	48-121	NA	NA
	2,4-Dinitrophenol	50	1,700	10-141	10-133	NA	NA
	2,4-Dichlorophenol	50	1,700	39-135	10-167	NA	NA
	2,4-Dimethyphenol	50	1,700	32-119	10-167	NA	NA
	2,4,6-Trichlorophenol	50	1,700	37-144	10-159	NA	NA
	Napthalene	50	1,700	21-133	10-145	NA	NA
	2-Methylnaphthalene	50	1,700	10-136	54-93	NA	NA
	Surrogate Spikes						
	Nitrobenzene-d5	50	1,700	35-114	23-120	NA	NA
	2-Fluorobiphenyl	50	1,700	43-116	30-115	NA	NA
	Terphenyl-d14	50	1,700	33-141	18-137	NA	NA
	Phenol-d5	75	2,500	10-94	24-113	NA	NA
	2-Fluorophenol	75	2,500	21-100	25-121	NA	NA
	2,4,6-Tribromophenol	75	2,500	10-123	19-122	NA	NA

NA Not applicable

TABLE D-5

PESTICIDE/PCB ANALYSIS
QUALITY CONTROL CRITERIA FOR LABORATORY DATA EVALUATION

Analytical Method	Spiking Compounds	Laboratory-Established Control Limits					
		Spike Concentration		Accuracy Percent Recovery (%)		Precision (RPD %)	
		Water (µg/l)	Soil/Sediments (µg/kg)	Water	Soil/Sediments	Water	Soil/Sediments
Matrix Spike/Matrix Spike Duplicates							
Pesticides/PCBs, SW 8081	Matrix						
	Lindane (Gamma -BHC)	0.20	16.0	32-127	32-127	15	50
	Heptachlor	0.20	16.0	34-111	34-111	20	31
	Aldrin	0.20	16.0	42-122	42-122	22	43
	Dieldrin	0.20	16.0	36-146	36-146	18	38
	Endrin	1.0	80.0	30-147	30-147	21	45
	4,4'-DDT	1.0	80.0	25-160	25-160	27	50
	Surrogate Spikes						
	TCMX	0.20	0.667	60-150	60-150	NA	NA
	DCB	0.20	0.667	60-150	60-150	NA	NA
Laboratory Control Samples							
Pesticides/PCB SW 8081	Matrix						
	Lindane (Gamma-BHC)	0.20	13	60-127	52-108	NA	NA
	Heptachlor	0.20	13	61-111	52-111	NA	NA
	Aldrin	0.20	13	64-113	42-122	NA	NA
	Dieldrin	0.20	13	75-146	57-146	NA	NA
	Endrin	1.0	13	58-133	55-126	NA	NA
	4,4-DDT	1.0	13	46-134	25-160	NA	NA
	Surrogate Spikes						
	TCMX	0.20	0.667	60-150	60-150	NA	NA
	DCB	0.20	0.667	60-150	60-150	NA	NA

NA Not applicable

TABLE D-6

SW-846 8151 ANALYSIS
QUALITY CONTROL CRITERIA FOR LABORATORY DATA EVALUATION

Analytical Method	Spiking Compounds	Laboratory-Established Control Limits		
		Spike Concentration (µg/l)	Accuracy (% Recovery)	Precision (RPD %)
Matrix Spike/Matrix Spike Duplicates				
	Pentachlorophenol	5	30-140	35
	Surrogate 2,4-DB	5	30-140	NA
Laboratory Control Samples				
	Pentachlorophenol	5	30-140	NA
	Surrogate 2,4-DB	5	30-140	NA

TABLE D-7

DIOXIN/FURAN QUALITY CONTROL CRITERIA FOR LABORATORY DATA EVALUATION

Analytical Method	Spiking Compounds	Spike Concentration		Laboratory-Established Control Limits			
				Accuracy Percent Recovery (%)		Precision (RPD %)	
		Water (mg/L)	Soil/Sediments (mg/kg)	Water	Soil/Sediments	Water	Soil/Sediments
Laboratory Control Samples							
SW-8290 (c)	2,3,7,8-TCDD	200	200	60-140	60-140	50	50
	2,3,7,8-TCDF	200	200	60-140	60-140	50	50
	1,2,3,7,8-PeCDD	500	500	60-140	60-140	50	50
	1,2,3,7,8-PeCDF	500	500	60-140	60-140	50	50
	2,3,6,7,8-PeCDF	500	500	60-140	60-140	50	50
	1,2,3,6,7,8-HxCDD	500	500	60-140	60-140	50	50
	1,2,3,6,7,8-HxCDD	500	500	60-140	60-140	50	50
	1,2,3,7,8,9-HxCDD	500	500	60-140	60-140	50	50
	1,2,3,6,7,8-HxCDF	500	500	60-140	60-140	50	50
	1,2,3,6,7,8-HxCDF	500	500	60-140	60-140	50	50
	1,2,3,7,8,9-HxCDF	500	500	60-140	60-140	50	50
	2,3,4,6,7,8-HxCDF	500	500	60-140	60-140	50	50
	1,2,3,4,6,7,8-HpCDD	500	500	60-140	60-140	50	50
	1,2,3,4,6,7,8-HpCDF	500	500	60-140	60-140	50	50
	1,2,3,4,7,8,9-HpCDF	500	500	60-140	60-140	50	50
	OCDD	1000	1000	60-140	60-140	50	50
	OCDF	1000	1000	60-140	60-140	50	50
(d)(f)	^{13}C-2,3,7,8-TCDD	1000	1000	25-150	25-150	50	50
(d)(f)	^{13}C-2,3,7,8-TCDF	1000	1000	25-150	25-150	50	50
(d)(f)	^{13}C-1,2,3,7,8-PeCDD	1000	1000	25-150	25-150	50	50
(d)(f)	^{13}C-1,2,3,7,8-PeCDF	1000	1000	25-150	25-150	50	50
(d)(f)	^{13}C-1,2,3,6,7,8-HxCDD	2500	2500	25-150	25-150	50	50
(d)(f)	^{13}C-1,2,3,4,6,7,8-HxCDF	2500	2500	25-150	25-150	50	50
(d)(f)	^{13}C-1,2,3,4,6,7,8-HpCDD	2500	2500	25-150	25-150	50	50
(d)(f)	^{13}C-1,2,3,4,6,7,8-HpCDF	2500	2500	25-150	25-150	50	50
(d)(f)	^{13}C-OCDD	5000	5000	25-150	25-150	50	50

(c) Native compound limits are COE default limits.
(d) Method default control limits. Signal-to-noise is also evaluated for (:eptability.
(f) These labeled analytes are spiked into all samples. RPD will be use :mpare sample/duplicate IS recoveries.

TABLE D-7

DIOXIN/FURAN QUALITY CONTROL CRITERIA FOR LABORATORY DATA EVALUATION (CONTINUED)

Analytical Method	Spiking Compounds	Spike Concentration		Laboratory-Established Control Limits			
				Accuracy Percent Recovery (%)		Precision (RPD %)	
		Water (mg/L)	Soil/Sediments (mg/kg)	Water	Soil/Sediments	Water	Soil/Sediments
Laboratory Control Samples (continued)							
SW-8290 (c)	2,3,7,8-TCDD	200	200	60-140	60-140	50	50
	2,3,7,8-TCDF	200	200	60-140	60-140	50	50
	1,2,3,7,8-PeCDD	500	500	60-140	60-140	50	50
	1,2,3,7,8-PeCDF	500	500	60-140	60-140	50	50
	2,3,6,7,8-PeCDF	500	500	60-140	60-140	50	50
	1,2,3,6,7,8-HxCDD	500	500	60-140	60-140	50	50
	1,2,3,6,7,8-HxCDD	500	500	60-140	60-140	50	50
	1,2,3,7,8,9-HxCDD	500	500	60-140	60-140	50	50
	1,2,3,6,7,8-HxCDF	500	500	60-140	60-140	50	50
	1,2,3,6,7,8-HxCDF	500	500	60-140	60-140	50	50
	1,2,3,7,8,9-HxCDF	500	500	60-140	60-140	50	50
	2,3,4,6,7,8-HxCDF	500	500	60-140	60-140	50	50
	1,2,3,4,6,7,8-HpCDD	500	500	60-140	60-140	50	50
	1,2,3,4,6,7,8-HpCDF	500	500	60-140	60-140	50	50
	1,2,3,4,7,8,9-HpCDF	500	500	60-140	60-140	50	50
	OCDD	1000	1000	60-140	60-140	50	50
	OCDF	1000	1000	60-140	60-140	50	50
(d)	^{13}C-2,3,7,8-TCDD	1000	1000	25-150	25-150	50	50
(d)	^{13}C-2,3,7,8-TCDF	1000	1000	25-150	25-150	50	50
(d)	^{13}C-1,2,3,7,8-PeCDD	1000	1000	25-150	25-150	50	50
(d)	^{13}C-1,2,3,7,8-PeCDF	1000	1000	25-150	25-150	50	50
(d)	^{13}C-1,2,3,6,7,8-HxCDD	2500	2500	25-150	25-150	50	50
(d)	^{13}C-1,2,3,4,6,7,8-HxCDF	2500	2500	25-150	25-150	50	50
(d)	^{13}C-1,2,3,4,6,7,8-HpCDD	2500	2500	25-150	25-150	50	50
(d)	^{13}C-1,2,3,4,6,7,8-HpCDF	2500	2500	25-150	25-150	50	50
(d)	^{13}C-OCDD	5000	5000	25-150	25-150	50	50

(c) Native compound limits are COE default limits.
(d) Method default limits. Signal-to-noise is also evaluated for data acceptability.
(f) These labeled analytes are spiked into all samples. RPD will be used to compare sample/duplicate IS recoveries.

TABLE D-8

ICP AND COLD VAPOR ATOMIC ABSORPTION METALS ANALYSES
QUALITY CONTROL CRITERIA FOR LABORATORY DATA EVALUATION

Analytical Method	Spiking Compounds	Spike Concentration		Control Limits			
				Accuracy Percent Recovery (%)		Precision (RPD %)	
		Water (µg/L)	Soil/Sediments (mg/kg)	Water	Soil/ Sediments	Water	Soil/ Sediments
Matrix Spike/Matrix Duplicates							
Metals ICP, 6010A	Antimony	500	50	80-120	95 ConfW	20	20
	Arsenic	2,000	200	80-120	95 ConfW	20	20
	Barium	2,000	200	80-120	95 ConfW	20	20
	Beryllium	50	5	80-120	95 ConfW	20	20
	Cadmium	50	5	80-120	95 ConfW	20	20
	Chromium	200	20	80-120	95 ConfW	20	20
	Lead (Trace)	500	50	80-120	95 ConfW	20	20
	Manganese	500	50	80-120	95 ConfW	20	20
	Selenium	2,000	200	80-120	95 ConfW	20	20
	Silver	50	5	80-120	95 ConfW	20	20
	Thallium	2,000	200	80-120	95 ConfW	20	20
	Calcium	2,000	10	80-120	95 ConfW	20	20
	Iron	2,000	10	80-120	95 ConfW	20	20
	Magnesium	2,000	10	80-120	95 ConfW	20	20
	Potassium	5,000	10	80-120	95 ConfW	20	20
	Sodium	10,000	10	80-120	95 ConfW	20	20
Metals, 7470/7471	Mercury	1.0	0.5	80-120	95 ConfW	20	20
Laboratory Control Samples							
Metals ICP, 6010A	Antimony	500	100	80-120	95 ConfW	20	20
	Arsenic	2,000	400	80-120	95 ConfW	20	20
	Barium	2,000	400	80-120	95 ConfW	20	20
	Beryllium	50	10	80-120	95 ConfW	20	20
	Cadmium	50	10	80-120	95 ConfW	20	20
	Chromium	200	40	80-120	95 ConfW	20	20
	Lead (Trace)	500	100	80-120	95 ConfW	20	20
	Manganese	500	100	80-120	95 ConfW	20	20
	Selenium	2,000	400	80-120	95 ConfW	20	20

TABLE D-8

ICP METALS ANALYSES
QUALITY CONTROL CRITERIA FOR LABORATORY DATA EVALUATION
(CONTINUED)

Analytical Method	Spiking Compounds	Spike Concentration		Control Limits			
				Accuracy Percent Recovery (%)		Precision (RPD %)	
		Water (µg/L)	Soil/Sediments (mg/kg)	Water	Soil/ Sediments	Water	Soil/ Sediments
Laboratory Control Samples (continued)							
Metals ICP, 6010A (continued)	Silver	50	10	80-120	95 ConfW	20	20
	Thallium	2,000	400	80-120	95 ConfW	20	20
	Calcium	5,000	1,300	80-120	95 ConfW	20	20
	Iron	5,000	9,000	80-120	95 ConfW	20	20
	Magnesium	5,000	1,100	80-120	95 ConfW	20	20
	Potassium	5,000	1,500	80-120	95 ConfW	20	20
	Sodium	5,000	300	80-120	95 ConfW	20	20
Metals, 7470/7471	Mercury	1.0	2.0	80-120	95 ConfW	20	20

95 ConfW 95% confidence window

TABLE D-9

WET CHEMISTRY ANALYSES
QUALITY CONTROL CRITERIA FOR LABORATORY DATA EVALUATION

Analytical Method	Spiking Compounds	Spike Concentration		Laboratory-Established Control Limits			
				Accuracy Percent Recovery (%)		Precision (RPD %)	
		Water (mg/L)	Soil/Sediments (mg/kg)	Water	Soil/Sediments	Water	Soil/Sediments
Matrix Spike/Matrix Duplicates							
TOC, EPA 415.1	Potassium hydrogen phosphorous	.20	40	80-120	80-120	20	20
Hexavalent chromnium, 7196	Hexavalent chromium	25	25	80-120	80-120	20	20
Common Anions							
EPA 325.2	Chloride	25	25	80-120	80-120	20	20
EPA 365.2	O-phosphate	0.25	25	75-125	75-125	20	20
EPA 365.2	Total phosphorous	0.25	25	75-125	75-125	20	20
EPA 375.4	Sulfate	10	100	75-125	75-125	20	20
EPA 353.2	Nitrate	0.02	0.2	75-125	75-125	20	10
	Nitrite	0.02	0.2	75-125	75-125	20	20
EPA 340.2	Fluoride	2.0	20	75-125	75-125	20	20
EPA 350.3	Ammonia	2.0	2.0	75-125	75-125	20	20
EPA 351.2	TKN	NA	7	NA	75-125	NA	20
EPA 377.1	Sulfite	10	100	80-120	80-120	20	20
EPA 150.1	pH	NA	NA	NA	NA	NA	NA
EPA 305.1	Acidity	20	200	NA	80-120	20	20
Laboratory Control Samples							
TOC, EPA 415.1	Potassium hydrogen phosphorous	10	NA	80-120	NA	20	NA
Hexavalent chromnium, 7196	Hexavalent chromium	.25	NA	80-120	NA	20	NA
Common Anions							
EPA 325.2	Chloride	50	NA	80-120	NA	20	NA
EPA 365.2	O-phosphate	0.4	NA	80-120	NA	20	NA
EPA 365.2	Total phosphorous	0.4	NA	80-120	NA	20	NA
EPA 375.4	Sulfate	NA	NA	NA	NA	NA	NA
EPA 353.2	Nitrate	0.8	NA	80-120	NA	20	NA
	Nitrite	0.8	NA	80-120	NA	20	NA
EPA 340.2	Fluoride	4.0	NA	80-120	NA	20	NA
EPA 350.3	Ammonia	2.0	NA	80-120	NA	20	NA
EPA 351.2	TKN	NA	NA	NA	NA	NA	NA
EPA 377.1	Sulfite	NA	NA	NA	NA	NA	NA
EPA 305.1	Acidity	NA	NA	NA	NA	NA	NA
EPA 150.1	pH	NA	NA	NA	NA	NA	NA

Not applicable

Appendices

APPENDIX A

ISVE PILOT STUDY ON BURIED WASTE IN THE ON-SITE AREA

FIELD SAMPLING PLAN AND TESTING PROCEDURES

APPENDIX A

ISVE PILOT STUDY ON BURIED WASTE IN THE ON-SITE AREA

FIELD SAMPLING PLAN AND TESTING PROCEDURES

IN-SITU VAPOR EXTRACTION PILOT STUDY ON BURIED WASTES IN THE ON-SITE AREA FIELD SAMPLING PLAN AND TESTING PROCEDURES

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APPENDIX A

IN-SITU VAPOR EXTRACTION PILOT STUDY ON BURIED WASTES IN THE ON-SITE AREA FIELD SAMPLING PLAN AND TESTING PROCEDURES

A.1.0. INTRODUCTION

The Statement of Work (SOW) for the Remedial Design and Remedial Action (RD/RA) for the American Chemical Service (ACS) Site allows the ACS Technical Committee the option, with approval by the U.S. EPA, to conduct an In-Situ Vapor Extraction (ISVE) pilot project in the On-Site Area. The purpose of this study will be to evaluate the effectiveness of ISVE on the buried waste materials found in the On-Site Area. If the study does not demonstrate that ISVE has the potential to reduce contaminant concentrations to the clean-up levels in the On-Site Area, then ISVE may be abandoned in favor of LTTT after approval by U.S. EPA. This Field Sampling Plan presents the scope and objectives of the pilot study, and the specifics of how the study will be conducted and evaluated. This Field Sampling Plan (FSP) is divided into the follow sections.

- Section A.2.0 Scope And Objectives Of The ISVE Pilot Study
- Section A.3.0 Pilot Study Design and Testing
- Section A.4.0 Sample Designation
- Section A.5.0 Decontamination Procedures
- Section A.6.0 Sample Handling And Analysis
- Section A.7.0 Sample Documentation
- Section A.8.0 Data Reduction And Evaluation And Reporting
- Section A.9.0 Compliance Requirements

A.2.0. SCOPE AND OBJECTIVES OF THE ISVE PILOT STUDY

A.2.1. Scope.

The scope of the ISVE pilot study includes:

- Installation of a test cell and dewatering wells in the pilot test area and dewatering of the test cell

- Installation of two vapor extraction wells and four multi-level vapor monitoring probe nests within the dewatered area

- Construction of a pilot-scale ISVE system, including a blower, vapor treatment unit, and associated piping and instrumentation

- Pre-test soil/waste and vapor sampling

- Start-up, operation, and monitoring of the pilot-scale ISVE system

- Post-test soil and vapor sampling

- Data Evaluation.

A.2.2. Objectives.

The specific objectives of the ISVE pilot study are to:

- Assess the effectiveness of ISVE on the buried waste materials in the On-Site area

- Demonstrate whether ISVE has the potential to reduce the contaminant levels in the buried waste to the performance standards outlined in the Record of Decision (ROD), the Unilateral Administrative Order (UAO), and SOW

- Determine if ISVE will enhance the natural degradation of contaminants

- Determine design parameters for full-scale implementation of ISVE in the On-Site Area.

A.3.0 PILOT STUDY DESIGN AND TESTING

A.3.1. Test Cell.

Because the wastes in the On-Site area are saturated, a test cell will be installed and dewatered to permit air flow during the ISVE pilot testing. The test cell size needs to be large enough to allow determination of most of the design parameters, but the size is limited by the quantity of purge water that can be handled and stored on site. A test cell with the minimum dimensions of 30 by 30 feet will be constructed within the On-Site Area shown in Figure A-1. The test cell walls will be constructed using either sheet piles, a soil bentonite slurry wall, or equivalent technology to minimize the quantity of groundwater generated during dewatering for the pilot study. The test cell will be tied into the underlying clay layer approximately 20 to 25 feet below the ground surface, as it is anticipated that a full-scale system would also be installed down to the underlying clay layer. This study area was selected based on a review of analytical results and soil boring logs generated during the Remedial Investigation Report (Warzyn, 1991) and the subsequent Technical Memorandum, Dewatering/Barrier Wall Alignment Investigation Report (Montgomery Watson, 1995). Based on this review, the study area represents the general contamination and stratigraphy found throughout the On-Site Area.

A.3.2. Test Cell Dewatering.

After the test cell is installed, one 2-inch diameter and three 1-inch diameter piezometers will be installed in the locations shown in Figure A-1. The piezometers will be constructed of stainless-steel well casing and screen. A conceptual design for the piezometers is shown in Figure A-2. Standard Operating Procedures (SOPs) for the installation of the piezometers are provided in Appendix G. The piezometers will be installed to a depth of approximately 20 feet below ground surface (bgs). The exact depths and screened intervals will be determined in the field. Any significant variance (greater than 2 feet) from the proposed depths will need approval by U.S. EPA and IDEM personnel. Soil samples from the boreholes will be collected for logging purposes only. No soil samples will be collected for analytical purposes from the piezometer soil borings. The 2-inch diameter piezometer will be installed in the center of the test cell and will be used to dewater the test cell. Assuming a test cell of 30 by 30 by 25 feet, an average soil porosity of 30 percent,

and an average groundwater column of 23 feet, approximately 46,500 gallons of groundwater will need to be extracted to dewater the cell. If the single extraction piezometer is capable of sustaining a three gallon per minute purge rate, it will take approximately 11 days to dewater the cell. All purge water will be stored on site in tanks (such as a Baker tank), sampled, and then disposed of at either an off-site disposal facility or at the on-site water treatment facility once it is operational. The purge water will be analyzed following the methods listed in Table A-1.

A.3.3. Installation of ISVE Wells and Vapor Probes.

After the cell has been dewatered, two ISVE wells (VEW1 and VEW2) and four vapor monitoring probe nests (VP1 through VP4) will be installed in the test cell as shown in Figures A-2 and A-3. A conceptual design for the ISVE wells is shown in Figure A-3, and a conceptual design for the nested vapor probes is provided in Figure A-2. The ISVE wells and vapor probes will be drilled and installed following the SOPs found in Appendix G. The soil boring for the shallow ISVE well will be drilled to a depth of approximately 11 feet bgs and the screen interval will be set from 5 to 10 feet bgs. One soil/waste sample will be collected from this boring at approximately 5 feet bgs and submitted for the analysis specified Table A-1. The soil boring for the deeper ISVE well will be drilled to a depth of approximately 21 feet bgs and the screen interval will be set from 10 to 20 feet bgs. One soil/waste samples will be collected from this boring at approximately 15 feet bgs and submitted for the analysis listed in Table A-1. The exact depths and screened intervals will be determined in the field. Any significant variance (greater than 2 feet) from the proposed depths will need approval by U.S. EPA and IDEM personnel.

Two soil/waste samples will be collected from the soil borings drilled to install the four vapor monitoring probe nests (four probe nests x 2 samples per boring = 8 total samples). These samples will be collected at approximately 5 and 15 feet bgs and submitted for the analyses listed in Table A-1. The boreholes for the vapor monitoring probes will be drilled to a depth of approximately 18 feet bgs. Each vapor monitoring probe nest will consist of three, 1-inch in diameter, 6-inch in length screen sections. The top of the screen sections will be placed at 7, 12, and 17 feet bgs at each nest, and will be labeled A, B, and C, respectively. The exact depths and screened intervals will be determined in the field.

Any significant variance (greater than 2 feet) from the proposed depths will need approval by U.S. EPA and IDEM personnel.

A.3.4. Pre-Test Vapor Sampling.

Before conducting any ISVE testing, the temperature and pressure at each vapor probe, temperature and pressure at both extraction wells, and the ambient air pressure, temperature, and relative humidity will be measured and recorded. Soil gas samples also will be collected from each extraction well and vapor monitoring probe and analyzed in the field for carbon dioxide (CO_2), oxygen (O_2), explosive atmospheres, and total volatile hydrocarbons as shown in Figure A-4. These measurements will be made using a GasTech Model 3250 OX infrared CO_2/O_2 gas analyzer connected to the soil vapor monitoring probes with quick connect/disconnect fittings. The total hydrocarbon measurements will be made using a flame ionization detector (FID). Additionally, the pressure in each vapor probe, the air temperature at the extraction well, and the ambient air pressure will be recorded prior to the system startup. SOPs describing the collection of the pre-test vapor samples are provided in Appendix F. SOPs describing the calibration of the field meters are provided in Appendix E. Measurements will be recorded using Figure A-5.

A.3.5. ISVE Equipment.

The pilot-scale ISVE system will consist of a blower, a condensate collection system, a vapor treatment unit, a flow meter located at the vapor extraction well head, a vacuum gauge at the vapor extraction well head, vapor sampling ports at the vapor extraction well head and at the outlet of the vapor treatment unit, and associated piping, valves, and fittings. The blower will be an intrinsically safe, explosion-proof regenerative blower capable of producing a minimum of 400 cubic feet per minute (cfm). Figure A-6 is a schematic of the ISVE system. Although the cell will be dewatered, the vacuum produced by the ISVE system will draw water into the system. To prevent water from flowing into the extraction system, an electric submersible pump will be installed in the deeper vapor extraction well. In addition, a depth to water probe will be placed through the ISVE well head to monitor the water depth in the extraction well.

A.3.6. ISVE System Start-Up.

Prior to conducting the ISVE testing, the equipment will be started and checked to confirm proper operation. Items to be checked before start-up system include: blower operation, vacuum and flow gauge calibration and operation, FID and Gastech sensor operation, and valves and pipes associated with the vacuum system. Any operational problems encountered during system start-up must be resolved before commencing pilot testing.

A.3.7. Initial Vapor Sampling.

Vapor sampling will be conducted at each vapor extraction well and at each vapor monitoring probe (14 total samples) at the beginning of the ISVE tests to determine the initial composition of the extracted vapors. Prior to collecting samples, one pore volume of air will be purged prior to collecting the samples using either the blower at the extraction wells or a vacuum pump at the vapor sampling ports. The vacuum pump will be connected to the vapor monitoring probe using quick connect/disconnect fittings. The vapor monitoring probes will be purged until soil gas concentrations stabilize as monitored using the GasTech meter. The soil vapor samples collected for laboratory analysis will be collected in one-liter Tedlar bags or Summa canisters. The bags will be purged with nitrogen gas before sample collection. Only one Tedlar bag or canister is necessary for the scheduled analyses. The samples of extracted vapor will also be stored in a cooler at ambient air temperature. Additional details of the vapor sampling are provided in the SOPs (Appendix F). The vapor samples will be sent to an off-site laboratory for analyses described in Table A-1.

A.3.8. Short-Term Variable Flow Rate.

Short-term variable flow rate tests will be conducted initially to determine the vacuum versus flow rate operating curve for each extraction well. These short-term tests also will be the air permeability tests. Five flow rates will be tested in ascending order for each extraction well (five flow rates for each well, ten tests total). During the short-term tests, vacuum measurements will be made at each monitoring probe and extraction well as follows:

Time from Start of Test	Measurement Frequency
0 to 15 minutes	1 minutes
15 to 30 minutes	3 minutes
30 to 60 minutes	10 minutes
1 to 2 hours	30 minutes
2 to 8 hours	60 minutes

Measurements will be recorded on the Air Permeability Test Data Sheet shown in Figure A-7. If the vacuum measurements stabilize before 8 hours, the flow rate will be changed and a new test will begin. Although actual flow rates will be determined in the field with U.S. EPA and IDEM input, it is anticipated that the five flow rates will be 10, 20, 30, 40, and 50 cubic feet per minute (cfm) for the shallow extraction well (5 feet of screen) and 20, 40, 60, 80, and 100 cfm for the deeper extraction well (10 feet of screen). If a short-term test is terminated due to equipment failure, the test will be rerun after the cell has stabilized (a minimum of 8 hours).

Results of the short-term test will be analyzed to determine air permeability and flow rate following the procedures outlined in Appendix F. The calculations, however, assume a study area of infinite areal extent. The presence of the test cell will impact the vapor flow paths and the time frame necessary for the vapor probes to reach steady state. Consequently, the permeability values may not be used for developing design parameters or estimating cleanup time. If necessary, permeability values obtained from sites with similar lithology (or values from the ISVE test in the ONCA) and values reported in the literature will used. The main purpose of this test, however, is to determine the appropriate flow rate to use in the long-term test. The information needed to select a flow rate is essentially a curve showing vacuum at the well head versus flow rate. As the flow rate increases, the vacuum will also increase up to a certain value. The optimum flow rate will be determined on each vapor extraction well.

In addition, the presence of subsurface heterogeneities can be identified during the short-term test by comparing response times and vacuum measurements in the individual vapor monitoring probes.

A.3.9. Long-Term Constant Flow Rate.

After the short-term tests, a long-term constant rate test will be conducted by extracting vapors from both extraction wells simultaneously. The extraction flow rate for the long-term test will be determined based on the results of the short-term test upon approval by the U.S. EPA and IDEM. The duration of the long-term test will be a minimum of two months but no longer than the time required to implement treatment for source materials in the Off-Site Area, as stated in the SOW. The actual duration of the test will be determined based on performance of the system and with input from U.S. EPA and IDEM personnel. If the long-term test is shut down due to equipment failure, the test will be restarted as soon as repairs can be performed. The system will need to operate at a minimum of 90 percent during the test duration.

As mentioned previously, the study area is in a cell, which is necessary for dewatering purposes. Consequently, a calculation of the radius of influence using the data generated during this test may not produce a result representative of the area once it is dewatered without a test cell. At a minimum, the data will be used to verify that the area of influence extends out to the vapor probes. Vacuum measurements collected during transient condition may also be used to evaluate the level of heterogeneity and possibly estimate the radius of influence and air permeability of the soils. Air permeability of the soils will be calculated using the SOP found in Appendix F. The calculated values will be compared to literature values , values obtained at sites with similar lithology, and values obtained from the ISVE test in the ONCA . Based on data from these sources, an air permeability value will be selected for use in developing design parameters and in estimating cleanup times. The primary information that will be obtained from the long-term study, however, is the contaminant loading data from the extracted vapors and the quantity and quality of the condensate.

Field monitoring data collection during the long-term test will be conducted according the following schedule.

- Every 8 hours for the first week
- Every 12 hours for the second week
- Daily thereafter.

The following field measurements will be collected according to the above schedule during each field monitoring data collection period:

- Vapor extraction flow rate at the extraction well
- Vacuum measurements at each vapor monitoring probe, well head, and the inlet to the blower
- Volume of condensate produced
- Field hydrocarbon measurements of extracted vapors at the vapor extraction well manifold
- Water level in piezometers
- Field hydrocarbon measurements in the vapor treatment unit offgas.

In addition to the field vapor analyses, vapor samples will be collected from the extraction well manifold and the vapor treatment unit offgas for analysis in an off-site laboratory. Samples will be collected for analysis according the following schedule.

- Every two days for the first week
- Twice the second week
- Weekly for the third through sixth weeks
- Monthly thereafter.

These samples will be collected as described in Section A.3.7. and submitted for analysis as described in Table A-1. The daily field measurements for total volatile hydrocarbons will be correlated with the samples collected for laboratory analysis. This will provide adequate monitoring of the vapor concentrations removed by the ISVE system and will determine the daily removal of contaminants.

Samples of the condensate will be sampled bi-weekly during the test and sent to an off-site laboratory for the analyses listed in Table A-1. This sampling schedule differs from that presented in the SOW, but it will provide sufficient data to meet the objectives of characterizing the condensate and estimating the mass of contaminants removed.

A.3.10. Post-Test Vapor Sampling.

After the long-term constant flow rate test is completed, a post-test vapor sampling program will begin. Vapor samples will be collected from the two extraction wells and the vapor monitoring probes and analyzed in the field for CO_2, O_2, and total hydrocarbons. The SOP for post-test vapor sampling is identical to the pre-test vapor sampling. Post-test measurements will be collected at system shut down, and at 0.5, 1, 2, 4, 8, 12, 24, and 48 hours after shut down.

Within 48 hours after completion of the long-term test, vapor samples also will be collected from the two vapor extraction wells and four vapor probe nests (14 total samples) and submitted to the laboratory for analysis. These samples will be collected as described in Section A.3.7. and submitted for analysis as described in Table A-1.

A.3.11. Post-Test Soil/Waste Sampling.

Within two week following shut down of the long-term test, one soil boring will be installed within 2 feet of the extraction wells and each of the four soil vapor monitoring probes. Soil samples will be collected from each boring at the approximate depths (within 1 foot) as those collected during the pre-test soil/waste sampling. These samples will be submitted for laboratory analysis as described in Table A-1. The soil borings will be installed using a hollow stem auger and following the SOP found in Appendix G. The soil borings will be backfilled with granular bentonite and hydrated.

A.3.12. Data Evaluation and Design Criteria.

The data generated from the pilot study will be used to:

- Evaluate the effectiveness of ISVE at removing contaminants from the buried waste materials in the On-Site Area and to assess the potential to meet the performance standards,

- Perform a mass balance

- Evaluate the effectiveness of ISVE for enhancing naturally-occurring biodegradation of the contaminants, and

- Determine design parameters for full-scale implementation of ISVE in the On-Site Area.

Effectiveness of Technology. The effectiveness of ISVE for removing contaminants and the potential for the technology to meet the performance standards will be evaluated based upon:

- The mass of contaminants removed during the ISVE pilot study, calculated based upon extracted vapor concentrations and vapor extraction flow rates (a mass balance will also be performed),

- Comparison between pre-test and post-test soil/waste and soil gas sampling results,

- Predicted cleanup time and ability to meet performance standards for buried wastes based on mathematical modeling (such as Hyperventilate or the MODFLOW-based AIR3D) and concentrations observed during the pilot testing.

Since the time frame of the test will not be long enough to reduce actual contaminant concentrations to the cleanup goals, the potential for the technology to meet the cleanup goals will be determined using the mathematical model. To accomplish this, the mass of a particular contaminant in the test area will be estimated using the available data. A mass removal rate will also be estimated based on the offgas concentrations observed in the latter part of the long term test. With these inputs, the model will predict the time to reach the

desired residual concentration for the respective contaminant. If the model generated cleanup time is greater than ten years, it is not likely that ISVE can effectively meet the cleanup goal. A cutoff point based on duration needs to be used because the mathematical models currently available assume that 100 percent of the mass can be removed. Practical experience demonstrates otherwise. Ten years was selected as a cutoff point for several reasons. First of all, the models generally predict a faster cleanup time than can actually be achieved in the field. Secondly, practical experience has shown that if the predictions do not indicate that the site can be remediated with ISVE within ten years, then it is unlikely that the cleanup goals can be met without some type of enhancement such as heating or biodegradation.

Bioremediation Enhancement. The effectiveness of ISVE for enhancing naturally-occurring biodegradation of the buried wastes will be determined by evaluating the pre- and post-test vapor sampling results. If oxygen is the limiting factor in biodegradation, the pre-test soil vapor samples in the contaminated area should exhibit elevated CO_2 concentrations (in the range of 5% to 15%), depressed O_2 concentrations (<5%), and elevated hydrocarbon concentrations. Background soil vapor concentrations should be similar to atmospheric concentrations for CO_2 and O_2, with hydrocarbon concentrations generally less than 100 ppm. During the ISVE test, the O_2 concentration in the soil vapor in the test area should increase to approximately 20%. Using the O_2 data from the 48-hour post-vapor sampling test, an oxygen utilization rate will be calculated. If the utilization rate is within the range of 0.05% to 1% O_2/hour, this will be sufficient evidence that some microbial activity is occurring and that the addition of O_2 in this area will enhance biodegradation.

Ideally, the utilization rate from the post-test vapor sampling should be compared to a utilization rate from a background vapor monitoring probe. Unfortunately, the probe would need to be very shallow (and therefore would reflect atmospheric conditions) and would need to be located off of the site. It is not possible to locate a vapor monitoring probe which will not be affected by the ISVE system within the dewatered test cell. In general, however, an uncontaminated background probe would have little or no oxygen utilization. Consequently, a background vapor probe for this purpose is not believed to be of significant value, and comparison to the baseline data will be used.

In addition to the above, microbial plate counts in pre-test and post-test soil samples will be compared to see if the microbial population has noticeably increased. Analytical data for

specific constituents required for biological growth will also be evaluated to determine if other factors could be limiting biodegradation.

Design Parameters. The design parameters for full-scale ISVE will be determined as follows:

- An ISVE blower operating curve will be generated for each extraction well by plotting vapor extraction flow rate per foot of well screen versus vacuum at the extraction well head. The vacuums generated during the pilot study will be greater than those experienced in a system without the test cell, which could result in a conservatively-sized (oversized) blower for the full-scale system. This will need to be considered when finalizing the blower selection if the technology is implemented at full scale.

- Extracted vapor concentrations at the vapor extraction wells, volumes of extracted condensate, and condensate composition will be used to design the offgas treatment system.

- The effective treatment radii of the vapor extraction wells and the air permeability of the soil/waste will be calculated based upon the vacuum measurements at the vapor monitoring probes and the vacuum and flow rate measurements at the vapor extraction wells. However, it is important to emphasize that the presence of the test cell walls will affect vapor flow paths which will impact the radius of influence generated from the pilot study as well as the vacuum measurements. Therefore, the effective radius of influence for the full-scale system may need to be selected based on engineering judgement and experience with sites of similar lithology (e.g., wells in silty sand formations are typically spaced on 40 foot centers).

A.4.0 SAMPLE DESIGNATION

All samples collected will have the study area prefix "ISVE" to define which pilot study the sample comes from. Location designators are as follows: vapor probes (VP) plus a probe number and letter designation, piezometers (PZ) plus a piezometer number, vapor

extraction well (VEW) plus a well number, extracted vapor (EV), treated vapor (TV), condensate (CD), purge water (WP), and confirmation soil borings (SB) plus a soil boring number. The media designations are as follows: soil samples (SO), groundwater (WG), and soil vapor (SV). A sequential number will be added to each sample collected for laboratory analysis. In addition, soil samples collected during the installation of the vapor extraction wells, monitoring probes, or confirmation soil sampling will have the sample interval in feet bgs (i.e., 5.0 for 5 feet bgs). Table A-2 provides a summary of sample identification.

A.5.0 DECONTAMINATION PROCEDURES

To prevent the introduction of contaminants into the subsurface during drilling, all downhole drilling and soil sampling equipment will be steam cleaned prior to use and after each borehole has been completed. After steam cleaning, all drilling equipment will be kept off the ground until it is used. Lubricants will not be used on any equipment sent downhole. The drill rig will be monitored for hydraulic leaks and for the presence of oil or grease.

To prevent cross contamination of the soil samples collected during drilling, the split-spoon soil sampler, catcher, brass sleeves, and stainless steel knives and trowels will be decontaminated between sample intervals using the following procedure:

- Wash with laboratory-grade detergent (Alconox)
- Rinse with approved water
- Rinse with isopropyl alcohol
- Air dry
- Triple rinse with double deionized water
- Air dry.

A.6.0 SAMPLE HANDLING AND ANALYSIS

A.6.0.1. Chain-of-Custody. A project specific Chain-of-Custody (COC) form will be completed and will accompany each sample cooler. The COC form includes project identification, project location, sample location, sample designation, sample container type,

analysis type, field lot control number, and cooler identification. In addition, there are spaces for entry of the sample collection date and time, sample collection technique, signature of the persons relinquishing and receiving samples, and the status of the samples upon receipt by the laboratory. A project specific COC is provided in the QAPP.

Additional sample handling and analytical procedures are provided in the QAPP

A.7.0 SAMPLE DOCUMENTATION

A.7.0.1. Documentation Procedures. The field analytical data generated the pilot studies will be recorded on the forms provided in Appendix E. In addition, on-Site personnel will maintain a field logbook. The field logbook will be a weather resistant, bound, survey-type book, with non-removable pages. Information that will be entered in the logbook includes the name and location of the job, personnel on Site, name and address of the field contact person, the date(s) sampling was started and completed, weather conditions, the calibration methods used for the organic vapor detectors, sampling location, sampling methodology, sample destination and how it was transported (e.g., name of shipping agent), any deviations from the FSP, and any other observations that may be relevant to the field program.

A.8.0 DATA REDUCTION AND EVALUATION AND REPORTING

All data generated during the pilot study will be reduced , evaluated, and reported following procedures outlined in the QAPP.

A.9.0 COMPLIANCE REQUIREMENTS

A.9.0.1 Drill Cuttings Handling Procedures. All soil cuttings produced during drilling will be containerized in Department of Transportation (DOT)-approved 55-gallon drums. The presence of VOCs will be monitored using an organic vapor detector. In addition, the on-Site personnel will document any visual signs of contamination (e.g., discolored or odoriferous soil). All drums will be labeled with the following information:

- American Chemical Service Site
- Borehole number
- Date
- Type of material
- Depth interval
- Maximum organic vapor reading.

All drums will be moved to a staging area. Drums containing soil with organic vapor detector readings that indicate the presence of contaminants or drums that contain soil that had other indications of contamination will be segregated. After the scheduled analyses for the soil samples are completed, the data will be reviewed to determine the types of contaminants that may be present and the proper disposal method for the cutting.

A.9.0.2. Purge and Decontamination Water Handling Procedures. All purge and decontamination water generated during the pilot testing will be placed in containers (e.g. Baker or Frac tanks) and stored on site. The purge and decontamination water will be sent to the on-Site water treatment plant once it is operational.

A.9.0.3. Vapor Treatment Unit Off-Gas. Based on the concentration of contaminants in the subsurface, ISVE operation will generate air emissions that will need to be treated. A catalytic oxidizer treatment unit will be installed to treat air emissions during both the short and long-term studies. The vapor treatment unit will consist of a catalytic oxidizer capable of treating the extracted vapors to the applicable air discharge limits (i.e., IDEM air quality standards and the 1×10^{-4} to 1×10^{-6} cumulative cancer risk range for nearby residence and site workers as stated on page 30 of the ROD). The need for, and design details of, additional emissions control, such as scrubbers, will be finalized prior to mobilization based upon input from system suppliers (regarding removal efficiencies) and based upon review of the proposed approach by the IDEM Office of Air Management. Air samples will be collected from the vapor treatment unit and analyzed in the field for total volatile hydrocarbons using an FID organic vapor meter. Air samples also will be collected and submitted for analysis on a bi-weekly basis. The samples will be analyzed for the parameters detailed in Table A-1. Should the results of the vapor treatment unit off-gas indicate failure in reducing air emissions to below the IDEM standards, the long-term testing will be terminated until the vapor treatment unit is replaced with a unit capable of

meeting the discharge limits. In addition, if the vapor treatment unit should fail during the testing, the test will be interrupted until the unit is functional.

A.9.0.4. ISVE System Condensate. The volume of condensate produced during the ISVE test will be measured on a daily basis. Samples of the condensate will be collected on a bi-weekly basis and submitted for the analysis specified in Table A-1. The condensate volume will be measured on a daily basis. The condensate will be placed with the purge and decontamination water and ultimately treated.

TABLE A-1

ISVE PILOT STUDY ON BURIED WASTES IN THE ON-SITE AREA
SUMMARY OF ANALYTICAL SAMPLES

Task	Total Number of Samples	Frequency	Matrix	Required Analysis
Pre-Test Sampling				
Install two ISVE well borings (1 sample per boring)	2	Once	Soil	VOCs, SVOCs, Pest/PCB, metals, Cr^{+6}, TOC, TKN, o-phosphate, plate count, grain size distribution, moisture content
Install four vapor probe borings (2 samples per boring; 5' and 15' bgs)	8	Once	Soil	VOCs, SVOCs, Pest/PCB, metals, Cr^{+6}, TOC, TKN, o-phosphate, plate count, grain size distribution, moisture content
Pre-test vapor samples collected from each VEW and vapor probe	14	Once	Vapor	CO_2, O_2, and TVH (field analyses)
Short-Term ISVE Testing				
Initial vapor samples collected from each VEW and vapor probe	14	Once	Vapor	VOCs, SVOCs, methane and ethane
Long-Term ISVE Testing				
Vapor samples collected from VEW manifold and vapor treatment unit offgas	224*	Every 8 hours for the first week Every 12 hours for the second week Daily thereafter	Vapor	TVH (field analysis)
Vapor samples collected from VEW manifold and vapor treatment unit offgas	14	Every two days for the first week Twice the second week Weekly for 3 - 6 weeks Monthly thereafter	Vapor	VOCs, SVOCs
Condensate samples collected from ISVE system	13	Bi-weekly	Water	VOCs, SVOCs, Pest/PCB, PCP, metals
Post-Test Sampling				
Post-test vapor samples collected from each VEW and vapor probe	14	Once	Vapor	VOCs, SVOCs, methane and ethane
Post-test vapor samples collected from each VEW and vapor probe	14 monitoring points 8 measurements/period 112 total measurements	At 0.5, 1, 2, 4, 8, 12, 24, and 48 hours after shut down	Vapor	TVH (field analysis)
Install five soil borings adjacent to the two VEWs and four vapor probes	10	Once	Soil	VOCs, SVOCs, Pest/PCB, metals, Cr^{+6}, TOC, TKN, o-phosphate, plate count, grain size distribution, moisture content
Purge water from test cell	1	Once	Water	VOCs, SVOCs, Pest/PCB, PCP, metals

See Table 1-2 in the QAPP for analytical methods
See Table 3-4 in the QAPP for QA/QC samples
* Based on a 6 month ISVE test

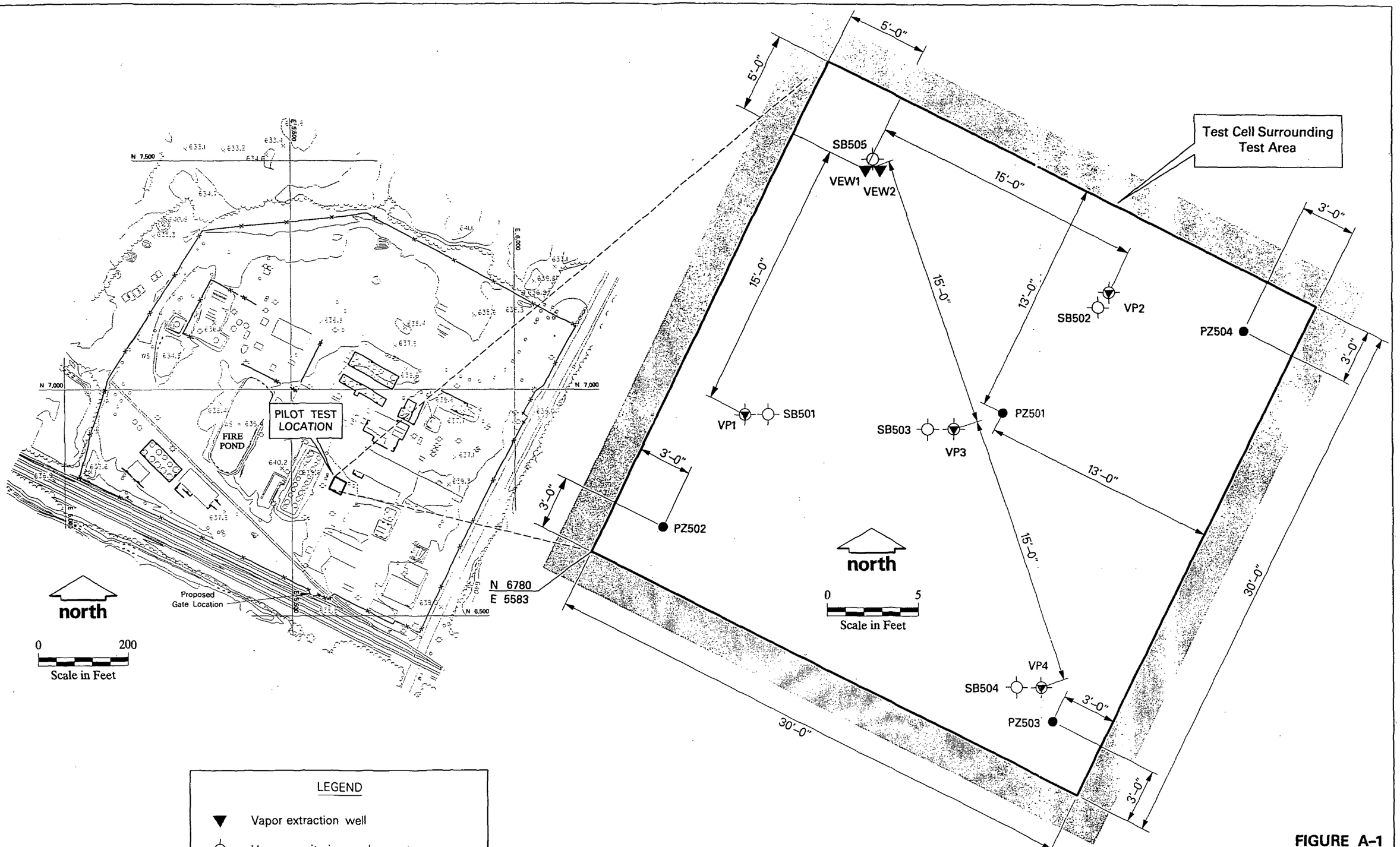
TABLE A-2

ISVE PILOT STUDY ON BURIED WASTE IN THE ON-SITE AREA
SUMMARY OF SAMPLE IDENTIFIERS

Pilot Study	Sampling Location	Well/Probe/Boring Identification	Sample Medium	Sample Depth (feet)	Sequential Identifier
In-Situ Vapor Extraction (ISVE)	Vapor Probe (VP)	1 to 4 and a to c	Soil (SO), Soil Vapor (SV)	5 &15 feet bgs	101-200
	Vapor Extraction Well (VEW)	1 and 2	Soil (SO), Soil Vapor (SV)	5 &15 feet bgs	201-300
	Extraction Vapor (EV)	NA	Soil Vapor (SV)	NA	301-400
	Treated Vapor (TV)	NA	Soil Vapor (SV)	NA	401-500
	Confirmation Soil Boring (SB)	501 to 505	Soil (SO)	5 &15 feet bgs	501-600
	Condensate (CD)	NA	Groundwater (WG)	NA	601-700
	Purge Water (WP)	NA	Groundwater (WG)	NA	701-800
	Piezometer (PZ)	501-504	NA	NA	NA

NA Not Applicable

Management Review
Other
Technical Review
Project Manager
Graphic Standards
Lead Professional
QUALITY CONTROL



LEGEND

- Vapor extraction well
- Vapor monitoring probe nest
- Post-test soil borings
- Piezometer–groundwater extraction well

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FIGURE A-1

Developed By	Drawn By
Approved By	Date
Reference	
Revisions	

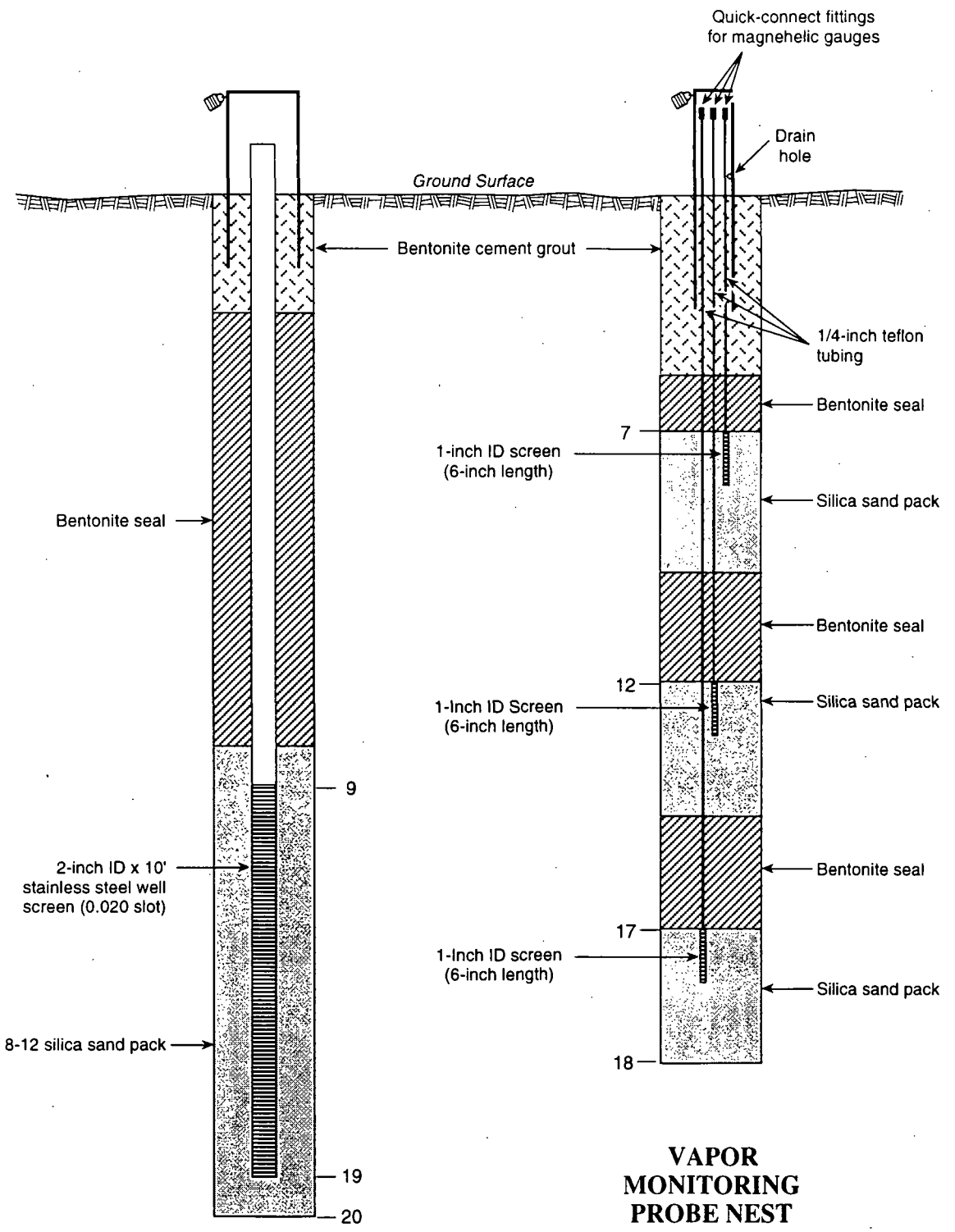
LOCATION MAP AND LAYOUT
ISVE PILOT STUDY ON BURIED WASTE IN THE ON-SITE AREA

TREATABILITY STUDY
AMERICAN CHEMICAL SERVICE, INC.
NPL SITE
GRIFFITH, INDIANA

Drawing Number
4077.0191

MONTGOMERY WATSON

QUALITY CONTROL | Graphic Standards / Lead Professional | Technical Review / Project Manager | Management Review / Other



PIEZOMETER DETAIL

VAPOR MONITORING PROBE NEST DETAIL

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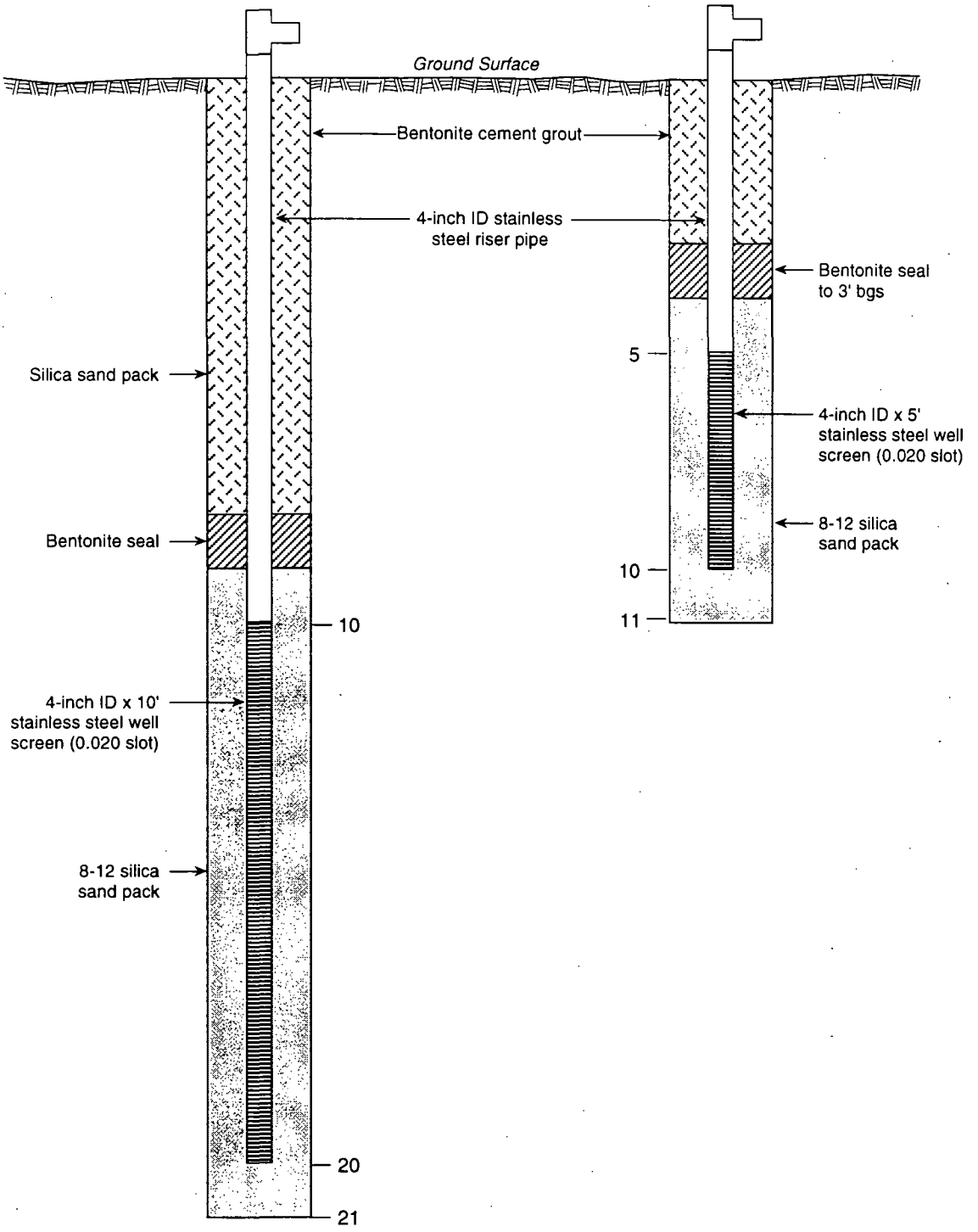
FIGURE A-2

Developed By	Drawn By	**CONCEPTUAL PIEZOMETER AND VAPOR MONITORING PROBE DESIGNS**	Drawing Number 4077.0191
Approved By	Date *8/12/96*		
Reference		TREATABILITY STUDY AMERICAN CHEMICAL SERVICE, INC. NPL SITE GRIFFITH, INDIANA	MONTGOMERY WATSON
Revisions			

QUALITY CONTROL | Graphic Standards Lead Professional | Technical Review Project Manager | Management Review Other

This document has been developed for a specific application and may not be used without the written approval of Montgomery Watson.

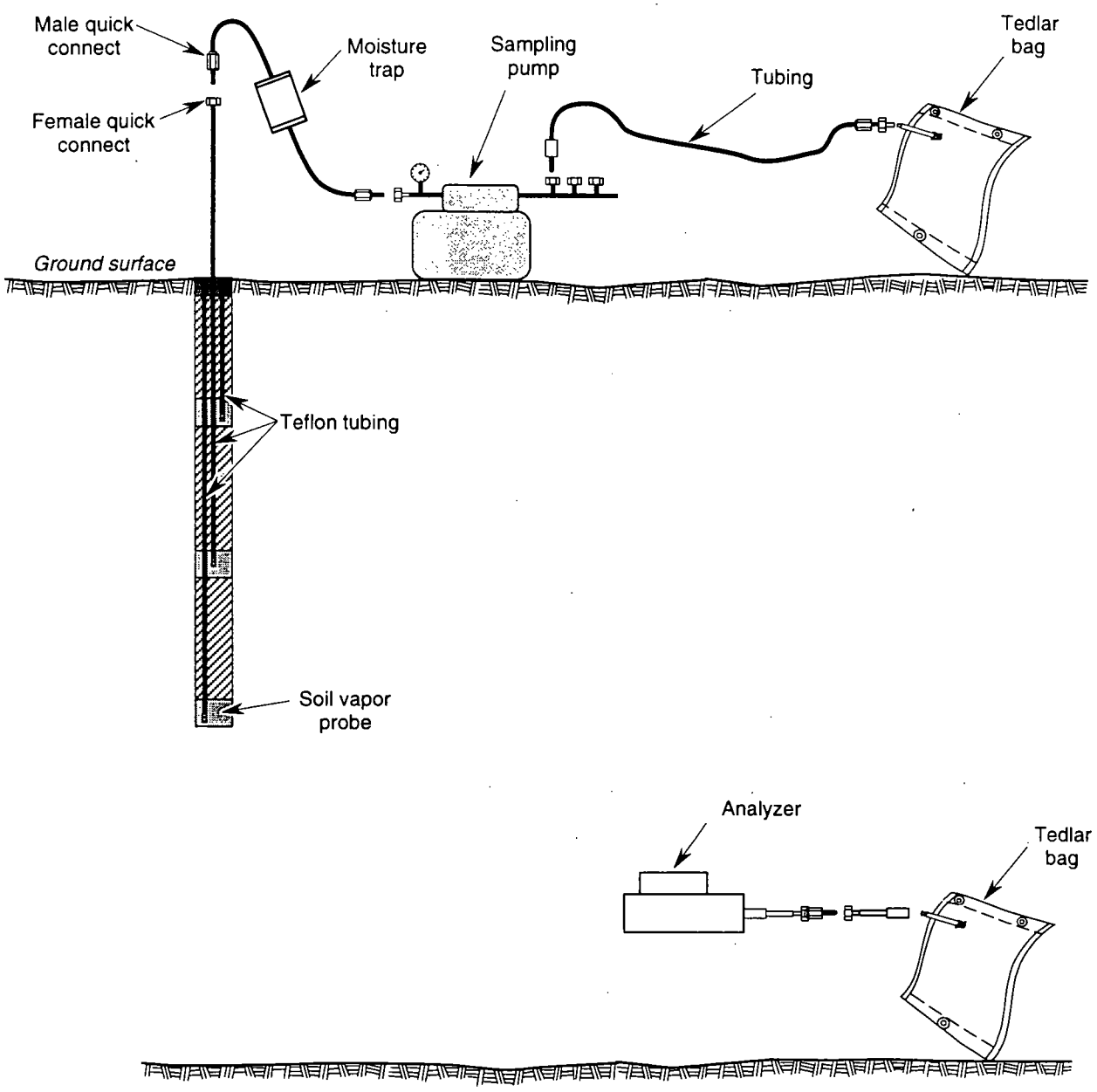


VAPOR EXTRACTION WELL NEST

FIGURE A-3

Developed By	Drawn By	**CONCEPTUAL ISVE WELL DESIGNS**	Drawing Number 4077.0191
Approved By	Date 8/12/96		
Reference		TREATABILITY STUDY AMERICAN CHEMICAL SERVICE, INC. NPL SITE GRIFFITH, INDIANA	MONTGOMERY WATSON
Revisions			

QUALITY CONTROL | Graphic Standards Lead Professional | Technical Review Project Manager | Management Review Other



Not to Scale

Modified from Hinchee, et al., 1992

FIGURE A-4

Developed By	Drawn By	**SCHEMATIC DIAGRAM OF VAPOR SAMPLING FROM VAPOR MONITORING PROBE NESTS**	Drawing Number 4077.0191
Approved By	Date *8/12/96*		
Reference		TREATABILITY STUDY AMERICAN CHEMICAL SERVICE, INC. NPL SITE GRIFFITH, INDIANA	**MONTGOMERY WATSON**
Revisions			

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QUALITY CONTROL			
	Graphic Standards _____	Technical Review _____	Management Review _____
	Lead Professional _____	Project Manager _____	Other _____

ISVE TEST DATA SHEET

Date	Time	Barometric Pressure	Dew Point	Humidity	Temp	VP1			VP2		
						a	b	c	a	b	c
						O_2	CO_2	Total Hydrocarbons	O_2	CO_2	Total Hydrocarbons

Developed By

Drawn By

Approved By

Date 8/12/96

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ISVE TEST DATA SHEET

TREATABILITY STUDY
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FIGURE A-5

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QUALITY CONTROL	Graphic Standards	Technical Review	Management Review
	Lead Professional	Project Manager	Other

ISVE TEST DATA SHEET

Date	Time	Barometric Pressure	Dew Point	Humidity	Temp	VP3			VP4		
						a	b	c	a	b	c
						O_2	CO_2	Total Hydrocarbons	O_2	CO_2	Total Hydrocarbons

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Approved By

Date 8/12/96

Reference

Revisions

ISVE TEST DATA SHEET

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FIGURE A-5

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QUALITY CONTROL	Graphic Standards	Technical Review	Management Review
	Lead Professional	Project Manager	Other

FIGURE A-6

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VAPOR EXTRACTION SYSTEM SCHEMATIC

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QUALITY CONTROL			
	Graphic Standards _____	Technical Review _____	Management Review _____
	Lead Professional _____	Project Manager _____	Other _____

TEST START

Time _____

Date _____

Time	Air Flow Rate (cfm)	Vapor Temp (°F)	Depth to GW (ft)	Extraction Well	Barometric Pressure	Relative Humidity	Dew Point	Temp	Pressures (in-H_2O)											
									VP1			VP2			VP3			VP4		
									a	b	c	a	b	c	a	b	c	a	b	c

a Shallow Probe
b Intermediate Probe
c Deep Probe

% O_2 prior to test
% O_2 after test
% CO_2 prior test
% CO_2 after test
ppm Total Hydrocarbons prior to test
ppm Total Hydrocarbons after test

FIGURE A-7

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Drawn By
Approved By
Date 8/12/96
Reference
Revisions

AIR PERMEABILITY TEST DATA SHEET

TREATABILITY STUDY
AMERICAN CHEMICAL SERVICE, INC.
NPL SITE
GRIFFITH, INDIANA

MONTGOMERY WATSON

Drawing Number
4077.0191

APPENDIX B

ISVE/AIR SPARGING PILOT STUDY OF CONTAMINATED SOILS IN THE ON-SITE CONTAINMENT AREA

FIELD SAMPLING PLAN AND TESTING PROCEDURES

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FIELD SAMPLING PLAN AND TESTING PROCEDURES

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APPENDIX B

ISVE/AIR SPARGING PILOT STUDY OF CONTAMINATED SOILS IN THE ON-SITE CONTAINMENT AREA FIELD SAMPLING PLAN AND TESTING PROCEDURES

B.1.0 INTRODUCTION

The Statement of Work (SOW) for the Remedial Design and Remedial Action (RD/RA) for the American Chemical Service (ACS) Site requires the ACS Technical Committee to conduct an In-Situ Vapor Extraction (ISVE) pilot study on contaminated soils in the On-site Containment Area (ONCA). Because the water table in the ONCA is approximately 2 to 3 feet bgs, and contamination is present in saturated soils, the area would need to be dewatered to perform the ISVE tests. Dewatering of an area of sufficient size and depth to conduct the ISVE tests would require at least six months and would generate a significant amount of contaminated water which would have to be treated. A sheet pile test cell similar to the one proposed for the ISVE tests for the On-site buried waste area (Appendix A) could be installed to reduce the time period and volume of water extracted, but as discussed in Appendix A, this would impact the usefulness of the data for establishing full-scale system design parameters. As an alternative to dewatering, air sparging is proposed to volatilize contaminants from the saturated soil into the vadose zone where the contaminants will be captured by the ISVE system.

The purpose of this study will be to evaluate the implementability and effectiveness of ISVE/air sparging on the contaminated soils found in the ONCA. This Field Sampling Plan (FSP) describes the procedures for the collection and analysis of field samples, and for the installation and operation of equipment during the ISVE/air sparging pilot study. As discussed in the Pre-Design Work Plan, samples of soil, soil vapor, and groundwater will be collected before, during, and after the pilot study to assess the performance of the treatment system and evaluate the effectiveness of the process in removing contaminants from the soil in the ONCA. If the results of the ISVE/air sparging pilot study show that ISVE/air sparging is not viable, the Respondents will submit documentation of this finding to the U.S. EPA and may request from the U.S. EPA an alternate course of action.

The ISVE/air sparging pilot study will be conducted in the ONCA in the location shown in Figure B-1. This study area was selected based on a review of analytical results and soil boring logs presented in the Remedial Investigation Report (Warzyn, 1991). Based on this review, the study area represents the general soils contamination and lithology found throughout the ONCA portion of the site. This FSP is divided into the follow sections:

- Section B.2.0—Scope And Objectives Of The ISVE/Air Sparging Pilot Study
- Section B.3.0—Pilot Study Design and Testing
- Section B.4.0—Sample Designation
- Section B.5.0—Decontamination Procedures
- Section B.6.0—Sample Handling and Analysis
- Section B.7.0—Sample Documentation
- Section B.8.0—Data Reduction and Evaluation and Reporting
- Section B.9.0—Compliance Requirements

B.2.0 SCOPE AND OBJECTIVES OF THE ISVE/AIR SPARGING PILOT STUDY

B.2.1. Scope.

The scope of the ISVE/air sparging pilot study includes:

- Installation of two air injection wells, three vapor extraction trenches, four groundwater/vapor monitoring wells and three piezometers within the pilot study area.

- Construction of a pilot-scale ISVE/air sparging system, including an air injection blower, vacuum extraction blower, vapor treatment unit, extraction trench sump pumps and associated piping and instrumentation

- Pre-test soil, groundwater, and vapor sampling

- Start-up, operation, and monitoring of the pilot-scale ISVE/air sparging system

- Post-test soil, groundwater, and vapor sampling
- Data Evaluation

B.2.2. Objectives

The specific objectives of the ISVE/air sparging study are to:

- Determine the feasibility of using ISVE/air sparging to remove contaminants from saturated and vadose zone soils in the ONCA
- Evaluate the effects of water table mounding on the implementability of an ISVE/air sparging system
- Generate data to determine design parameters for full-scale implementation of the technology at the site (i.e., air flow rates, sparging frequency)
- Assess the effectiveness of the technology in achieving the performance standard for contaminants in the ONCA soils
- Assess whether or not ISVE/air sparging will enhance the natural degradation of contaminants

B.3.0 PILOT STUDY DESIGN AND TESTING

B.3.1. Test Location

The ISVE/air sparging study location will be delineated and staked in the ONCA at the approximate location shown in Figure B-1. The final location and stake out of the site will be made in the field by Montgomery Watson and U.S. EPA personnel. Two air injection wells, three vapor extraction trenches, four groundwater/vapor monitoring wells, and three piezometers will be installed to conduct the ISVE/air sparging study in the ONCA. All wells, trenches and other systems that are necessary to conduct the pilot study will be located relative to the test site boundaries. The air injection wells, vapor

extraction trenches, groundwater/vapor monitoring wells, and piezometers will be installed in the relative locations shown in Figure B-1.

B.3.2. Installation of the Air Injection Wells, Groundwater/Vapor Monitoring Wells, and Piezometers

B.3.2.1. Drilling and Sampling Procedures. The air injection wells, groundwater/vapor monitoring wells, and piezometers will be drilled and installed following the SOPs for Drilling and Sampling found in Appendix G. Hollow stem auger drilling methods will be used with 5-foot lengths of 7 3/4-inch OD augers. The boreholes will have an effective diameter of approximately 8 inches. All boreholes will be continuously sampled at two-foot intervals using split spoon samplers and logged for lithologies. The soil collected from the split-spoon sampler will be visually inspected and screened with a Sensidyne FID organic vapor analyzer (OVA) for signs of contamination. After reaching the required depth, construction of the wells and piezometers will commence. All screens and riser pipe for air injection wells, monitoring wells, and piezometers will be constructed of threaded type 304 stainless steel. The screens will be 0.020 slotted screen with threaded stainless steel end caps.

B.3.2.2. Air Injection Well Construction. Borings for the air injection wells will be drilled to a depth of 21 feet bgs. Two soil samples will be collected and sent for analyses from each air injection well boring (two wells x 2 samples per boring = 4 total samples). These samples will be collected at approximately one foot bgs (vadose zone) and ten feet bgs (saturated zone) and sent to the appropriate laboratory for the analyses listed in Table B-1. Air injection wells will consist of a single 2-inch ID well. They will be screened over an interval of 15 to 20 feet bgs. The sand pack for the air injection wells will consist of two intervals of different size sand. The lower interval, from 21 feet to 17.5 feet bgs, will consist of size 8-12 silica sand. The remaining sand pack interval, from 17.5 to 14 feet bgs, will consist of size 16-30 silica sand. The purpose of the two sand sizes is to compensate for a decreasing pressure gradient towards the bottom of the well. A minimum 2 feet thick bentonite seal will be placed over the sand pack. The remainder of the borehole will be filled with cement bentonite grout. Figure B-2 illustrates the injection well design.

B.3.2.3. Groundwater/Vapor Monitoring Well Construction. Each boring for the groundwater/vapor monitoring wells will be drilled to a depth of 16 feet bgs. Two soil samples will be collected and sent for analyses from each soil boring drilled to install the four groundwater/vapor monitoring wells (four wells x 2 samples per boring = 8 total samples) These samples will be collected at approximately one foot bgs (vadose zone) and ten feet bgs (saturated zone) and sent to the appropriate laboratory for the analyses listed in Table B-1. The groundwater/vapor monitoring wells will contain a vapor monitoring probe and a groundwater sampling well as shown in Figure B-2. The soil vapor monitoring probe will be installed in the vadose zone at a depth of approximately 18 inches bgs. Vapor probes are installed using the same procedures as outlined in Appendix G for monitoring well installation. Vapor probes will be installed such that the vapor probe screen is vertically aligned in the center of the borehole. The groundwater monitoring well will be screened across the saturated zone over a depth of approximately 5 to 15 feet. Each vapor probe will be constructed with 1/4-inch Teflon tubing connected to a 6-inch long, 1-inch diameter stainless steel screen. The Teflon tubing will extend approximately 3 feet above the ground surface and will be fitted with air-tight, quick connect/disconnect couplings that will be used in conjunction with magnehelic™ gauges, which will also be equipped with air-tight, quick connect/disconnect fittings. The sand pack surrounding the vapor probe will begin at 2 feet bgs and extend to 1 foot bgs. The sand pack will consist of size 8-12 silica sand. The remainder of the borehole will be filled with a bentonite seal. Each monitoring well will be constructed of 2-inch ID casing with 10 feet of screen. The sand pack for the monitoring well will also consist of size 8-12 silica sand followed by a minimum 2 feet thick bentonite seal.

B.3.2.4. Piezometer Construction. Soil borings for the piezometers will be drilled and sampled for lithologies to a depth of 11 feet bgs. No laboratory samples will be collected from these borings. Piezometers will be constructed of 2-inch ID casing with 5 feet of screen. All piezometers will be installed to a depth of 10 feet. The sand pack for all piezometers will consist of size 8-12 silica sand and will be followed with a minimum 2 feet thick bentonite seal. The exact depths and screened intervals will be determined in the field. Any significant variance (greater than two feet) from the proposed depths will need approval by U.S. EPA and IDEM personnel. Construction details for the piezometers are shown in Figure B-3.

B.3.2.5. Well and Piezometer Development. Each air injection well, monitoring well, and piezometer will be developed using the procedures described in the Well Development SOP in Appendix F. Bailing and surging will be conducted using a stainless steel bailer decontaminated before and after each use. Decontamination procedures are outlined in Section B.5.0. A stainless steel submersible pump may be used to evacuate the remaining calculated purge column as per the Well Development SOP.

B.3.3. Installation of the Vapor Extraction Trenches

B.3.3.1. Trenching Procedures. The vapor extraction trenches will be excavated to a depth of approximately 2 feet using a standard 18-inch wide backhoe bucket. The trenches will have an effective width of 18 inches and will be 60 feet in length. The trenches will slope gradually from each end towards the center (3 inches over 30 feet). The excavation will be enlarged at the center of each trench to house a 24 x 24 x 48-inch concrete vault which will collect any groundwater that enters the trench.

B.3.3.2. Design and Construction. After excavation of the trench, construction of the horizontal vapor extraction well will commence. The vapor extraction well will consist of a single 4-inch PVC pipe which will be slotted on one side and which will lay slotted side down in the trench. Prior to installation of the extraction piping in the trench, the 24 x 24 x 48 inch deep concrete vault will be installed in the center of each trench. The vault will extend from the ground surface to approximately 24-inches below the bottom of the trench. Following installation of the vault, 12-inches of 1/4 inch pea gravel will be placed in the trench. The PVC vapor extraction pipe will be placed on top of the pea gravel and will extend into the concrete vault in the center of each trench. The trench will then be backfilled to the surface with pea gravel and the piping penetrations into the concrete vault will be sealed with grout. Figure B-4 presents details of the extraction trench completion.

B.3.3.3. Surface Completion. The vapor extraction trenches will be completed at the ground surface by sealing the entire ground surface in the ISVE/air sparging pilot study area with 6 mil visqueen sheeting. The above-ground completion of the concrete vault will consist of a removable airtight cover that can be secured with a lock. Figure B-4 presents the above-ground completion detail for the vapor extraction trenches.

B.3.4. ISVE/Air Sparging Equipment

B.3.4.1. Extraction Blower System. The pilot-scale ISVE system will consist of a trailer-mounted, regenerative blower piped to the vapor extraction trenches. The blower will be capable of producing up to 400 cubic feet per minute (cfm) of vapor flow at 6 inches of mercury (in-Hg) vacuum. The unit will be equipped with explosion-proof combination motor starter/disconnects and will require a three-phase, 230-volt power source. Power will be provided by connecting to existing power at the site. The blower system and the vapor extraction trenches will be connected using 4-inch ID Schedule 40 PVC pipe and fittings with glued connections. Figure B-5 depicts a schematic of the vapor extraction system design. To protect the blower during winter months, the blower system will be housed in a portable shed. Additional equipment included on the extraction test unit includes:

- A condensate collection system consisting of a 55-gallon air/water separator (demister) with particulate filter, high water shut down and vacuum relief valve
- A vacuum gauge, pressure gauge, flow meter and temperature probe on the vacuum line from each trench and on the blower inlet line
- A pressure gauge on the blower discharge line
- A sampling port on the blower inlet
- A vapor treatment unit with a sampling port on the outlet
- An intrinsically safe pneumatic submersible pump installed in the bottom of each trench vault to prevent water from flowing into the vapor extraction system
- A piezometer located adjacent to each extraction trench to monitor the groundwater level at each trench

- Piping, valves and fittings necessary to connect all equipment

- A weather station equipped with gauges for temperature, barometric pressure, relative humidity, and dew point.

B.3.4.2. Air Injection Blower System. The flow rate tests and the air sparging test will be conducted using a trailer-mounted, regenerative blower piped to the air injection wells. The blower will be capable of producing up to 280 cubic feet per minute (cfm) of air flow at a discharge pressure of 10 psi. The unit will be equipped with explosion-proof combination motor starter/disconnects and will require a three-phase, 230-volt power source. Power will be provided by connecting to existing power at the site. The blower piping and the air injection wells will be connected using 2-inch ID Schedule 40 PVC pipe and fittings with glued connections. Figure B-5 depicts a schematic of the air injection system design. To protect the blower during winter months, the blower system will be housed in a portable shed. Additional equipment included on the injection blower unit includes:

- Particulate filters on the blower inlet and discharge lines

- Pressure relief valve, pressure gauge, air flow meter and temperature probe on the blower discharge line

- A vacuum gauge on the blower inlet line

- Piping, valves, and fittings necessary to connect all equipment

B.3.5. Pre-Test

B.3.5.1. Pre-Test Soil Sampling During installation of the ISVE/air sparging system, soil samples will be collected from the well boreholes as described in section B.3.2 according to the procedures contained in the Drilling and Soil Sampling SOP contained in Appendix G. All samples will be collected using split spoon samplers. The scheduled analyses are listed in Table B-1.

B.3.5.2. Pre-Test Groundwater Sampling Before conducting any ISVE/air sparging system testing, groundwater samples will be collected from the four groundwater monitoring wells according to the procedures contained in the Groundwater Sampling and Field Measurement SOP in Appendix G. Three purge volumes, as calculated using the procedures outlined in the SOP for groundwater sampling, will be evacuated prior to sampling. Stainless steel bailers will be used for all groundwater sampling. The scheduled analyses are listed in Table B-1.

B.3.5.3. Pre-Test Vapor Sampling Before conducting any ISVE/air sparging testing, the temperature and pressure at each vapor probe and the ambient air pressure, temperature, and relative humidity will be measured and recorded. Soil vapor samples also will be collected from each vapor monitoring probe and analyzed in the field for carbon dioxide (CO_2), oxygen (O_2), and total volatile hydrocarbons. These measurements will be made using a GasTech Model 3250 OX infrared CO_2/O_2 gas analyzer and a Trace-Techtor portable hydrocarbon vapor tester connected to the soil vapor monitoring probes with quick connect/disconnect fittings. Procedures describing the collection of the pre-test vapor samples are provided in Appendix F in the SOP for Sampling and Monitoring Procedures for ISVE System Operation and Data Collection. SOPs describing the calibration of the field meters are provided in Appendix E.

B.3.6. ISVE/Air Sparging System Start-Up

B.3.6.1. System Start-up Prior to conducting the ISVE/air sparging testing, the equipment will be started and checked to confirm proper operation. Items to be checked before start-up of the system include: Blower operation, vacuum and flow gauge calibration and operation, FID and Gastech sensor operation, and valves and pipes associated with the vacuum system. Any operational problems encountered during system start-up must be resolved before commencing any pilot testing.

B.3.7. ISVE Test (without air sparging)

The ISVE system will be operated alone without the air sparging system to determine the optimum vapor extraction flow rates for the vapor extraction system and to evaluate the effectiveness of ISVE without air sparging.

B.3.7.1. Initial Vapor Sampling Vapor sampling will be conducted at the vapor extraction trench manifold and at each vapor monitoring probe (5 total samples) at the beginning of the test to determine the initial composition of the extracted vapors. Prior to collecting samples, one pore volume of air will be purged using either the extraction blower or a vacuum pump at the vapor sampling ports. The vacuum pump will be connected to the vapor monitoring probe using quick connect/disconnect fittings. The vapor monitoring probes will be purged until soil gas concentrations stabilize as monitored using the GasTech meter. The soil vapor samples collected for laboratory analysis will be collected in one-liter Tedlar bags (or Summa canisters). Only one Tedlar bag or Summa canister is necessary for the scheduled analyses. The samples of extracted vapor will be stored in a cooler at ambient air temperature. Additional details of the vapor sampling are provided in the SOPs for Sampling and Monitoring Procedures for ISVE System Operation and Data Collection (Appendix F). The vapor samples will be sent to an off-site laboratory for the analyses scheduled in Table B-1.

B.3.7.2. Flow Rate Tests. Flow rate tests will be initially conducted without air sparging to determine the vacuum versus flow rate operating curve for the three trenches together. These short-term tests also will be the air permeability tests. Short-term variable flow rate tests will be conducted until steady state is reached for each flow rate. Based upon the results of the short-term tests, a two week constant flow rate test will be conducted using the optimum vapor extraction flow rate. The information needed to select a flow rate is essentially a curve showing vacuum at the well head versus flow rate. As the flow rate increases, the vacuum will also increase up to a certain value. From that point on, increasing the flow rate will not increase the vacuum. The lowest flow rate achieving the maximum vacuum will be selected for use in the long term test. The short-term and two week constant flow rate tests will provide design data for a full-scale ISVE/air sparging system.

B.3.7.3. Short-Term Variable Flow Rate Tests. Five flow rates will be tested in ascending order for the vapor extraction system alone in the short-term tests. During the vacuum extraction short-term tests, vacuum measurements will be made at each vapor probe as follows:

Time from Start of Test	Measurement Frequency
0 to 15 minutes	1 minute
15 to 30 minutes	3 minutes
30 to 60 minutes	10 minutes
1 to 2 hours	30 minutes
2 to 8 hours	60 minutes

The duration of each test will depend on the length of time required to achieve steady state vacuum measurements at the vapor monitoring probes. Although actual flow rates will be determined in the field with U.S. EPA and IDEM input, it is anticipated that the five flow rates will be 10, 20, 30, 40, and 50 cubic feet per minute for the three extraction trenches. Measurements will be recorded on the Air Permeability Test Data Sheet shown in Figure A-7 in Appendix A. Results of the short-term test will be analyzed to determine air permeability and flow rate following the procedures outlined in Appendix F.

B.3.7.4. Two Week Constant Flow Rate Test. At the conclusion of the short-term tests, a two week constant flow rate test will be conducted. The vapor extraction flow rate for the two week test will be determined in the field based on the results of the short-term tests upon approval by the U.S. EPA and IDEM.

Field monitoring data collection during the two week constant flow rate test will be conducted according the following schedule.

- Every 8 hours for the first week
- Every 12 hours for the second week

The following field measurements will be collected during each field monitoring data collection period:

- Vapor extraction flow rate at the extraction trench manifold
- Vacuum measurements at each vapor monitoring probe, trench manifold, and the inlet to the blower
- Volume of condensate produced
- Field hydrocarbon measurements of extracted vapors at the vapor extraction trench manifold
- Water levels in piezometers and monitoring wells
- Field hydrocarbon measurements in the vapor treatment unit off-gas
- Explosimeter readings at the extraction trench manifold and blower inlet.

The field hydrocarbon measurements at the vapor extraction trench manifold will monitor daily contaminant removal, as required by U.S. EPA.

In addition to the field vapor analyses, vapor samples will be collected at the vapor extraction trench manifold for VOC and SVOC analyses in an off-site laboratory. These samples will be collected every two days the first week and twice the second week. The off-gas from the vapor treatment unit will be sampled once during the test and submitted to an off-site laboratory for VOC and SVOC analyses.

Condensate samples will be collected once a week and submitted to an off-site laboratory for VOC and SVOC analyses.

These samples will be collected as described in the SOPs for Sampling and Monitoring Procedures for ISVE System Operation and Data Collection (Appendix F).

Due to the shallow depth to water in the ONCA, extracted groundwater may accumulate in the concrete vaults at each extraction trench. If appreciable amounts of water begin to accumulate, then the pumps will be turned on and the extracted groundwater will be discharged to the groundwater holding tanks staged on site. The volume of water pumped from the trenches will be measured with a flow meter attached to the discharge hose on the pump and the volume of water discharged to the tanks will be recorded in the field logbook. The effectiveness of this system will be evaluated prior to beginning the combined ISVE/air sparging test. Provisions will be made for any necessary upgrades.

B.3.8. Air Sparging Test (without the ISVE system)

Upon completion of the two week constant flow rate test for the ISVE system, the air sparging system will be started up and tested without the ISVE system in operation. The data collected from the air sparging test will be used to evaluate the effectiveness of air sparging without ISVE.

B.3.8.1. Initial Vapor Sampling Vapor samples will be collected at the extraction trench manifold and at each vapor monitoring probe (5 total samples). Prior to collecting samples, the soil vapor monitoring probes will be purged using a vacuum pump at the vapor sampling ports. The vacuum pump will be connected to the vapor monitoring probe using quick connect/disconnect fittings. The vapor monitoring probes will be purged until soil gas concentrations stabilize on the GasTech meter. The soil vapor samples collected for laboratory analyses will be collected in one-liter Tedlar bags or Summa canisters. Only one Tedlar bag or Summa canister is necessary for the scheduled analyses at each monitoring point. The samples of extracted vapor will be stored in a cooler at ambient air temperature. Additional details of the vapor sampling are provided in the SOPs for Sampling and Monitoring Procedures for ISVE System Operation and Data Collection (Appendix F). The vapor samples will be sent to an off-site laboratory for VOC and SVOC analyses as listed in Table B-1.

B.3.8.2. Flow Rate Tests. Flow rate tests will be conducted without the ISVE system in operation to determine the vapor pressure versus flow rate operating curve for the air injection wells. Short-term variable flow rate tests will be conducted until steady state is reached for each flow rate. Based upon the results of the short-term tests, a two week constant rate test will be conducted using the optimum air injection flow rate. The short-

term and two week constant flow rate tests will provide design data for a full-scale ISVE/air sparging system.

B.3.8.3. Short-Term Variable Flow Rate Tests. Five flow rates will be tested for the air injection system in the short-term tests. The duration of each test will depend on the length of time required to achieve steady state vapor pressure measurements at the vapor monitoring probes. Although actual flow rates will be determined in the field with U.S. EPA and IDEM input, it is anticipated that the five flow rates will be 3, 5, 10, 15, and 20 cubic feet per minute for the two injection wells. The optimal flow rate will be selected based on field observations of pressure at the well heads and mounding of the water table. The well head pressure at each monitoring well and piezometer will be monitored for each flow rate. It is expected that the pressure at the well heads will increase as the flow rate is increased. Additionally, as the flow rate increases the monitoring points located at a greater radial distance from the air injection wells will be impacted. The radius of influence is considered to be at a maximum when the water table begins to show a considerable increase in elevation or mounding. At this point, the air being injected into the subsurface has reached the maximum lateral extent and begins to vertically displace formation water. The flow rate at which mounding begins to occur will be considered the maximum flow rate for this system. The optimal flow rate selected will be the maximum flow rate that can be maintained while keeping mounding of the water table less than one foot.

B.3.8.4. Two Week Constant Flow Rate Air Sparging Test. At the conclusion of the short-term tests, a two week constant flow rate test using air sparging will be conducted. The air injection flow rate for the two week test will be determined in the field based on the results of the short-term tests upon approval by the U.S. EPA and IDEM.

Field monitoring data collection during the two week constant flow rate test will be conducted according the following schedule.

- Every 8 hours for the first week
- Every 12 hours for the second week

The following field measurements will be collected during each field monitoring data collection period:

- Air injection flow rate and temperature at the blower
- Vapor pressure measurements at each vapor monitoring probe and at the air injection blower outlet
- Water levels and pressure measurements at piezometers and monitoring wells

The above field measurements will be collected as described in the SOPs for Sampling and Monitoring Procedures for ISVE System Operation and Data Collection (Appendix F).

B.3.9. Constant Rate Combined ISVE/Air Sparging Test

The combined ISVE/air sparging constant rate test will be conducted using operating parameters selected from the results of the individual two week tests conducted for the ISVE and air sparging systems, and as agreed upon by U.S. EPA and IDEM.

The duration of the ISVE/air sparging test will be a minimum of two weeks, but no longer than the time required to implement treatment for source materials in the Off-site Area. The actual duration of the test will be determined based on performance of the system and with input from U.S. EPA and IDEM personnel. The duration of the test may be modified based upon the results and the schedule of the remaining RD/RA activities at the site.

Field monitoring data collection during the combined ISVE/air sparging test will be conducted according the following schedule.

- Every 8 hours for the first week
- Every 12 hours for the second week.

The following field measurements will be collected during each field monitoring data collection period:

- Vapor extraction flow rate at the vapor extraction trench manifold
- Air injection flow rate at the blower
- Vacuum measurements at each vapor monitoring probe, trench manifold, and the inlets and outlets to the blower
- Volume of condensate produced
- Field hydrocarbon measurements at the extraction trench manifold
- Explosimeter readings at the extraction trench manifold and the inlet to the blower
- Water levels and pressure measurements at piezometers and monitoring wells
- Field hydrocarbon measurements in the off-gas from the vapor treatment unit.

In addition to the field vapor analyses which will provide daily contaminant removal data, vapor samples will be collected at the vapor extraction trench manifold for VOC and SVOC analyses at an off-site laboratory. Samples will be collected for analysis according the following schedule.

- Every two days for the first week
- Twice the second week
- Weekly for the third through eighth weeks
- Monthly thereafter.

The vapor treatment unit off-gas samples will be collected on a bi-weekly basis and submitted to an off-site laboratory for VOC and SVOC analyses for emissions evaluation.

Condensate samples will also be collected bi-weekly from the air/water separator and submitted to an off-site laboratory for analyses listed in Table B-1.

All samples will be collected as described in the SOPs for Sampling and Monitoring Procedures for ISVE System Operation and Data Collection (Appendix F).

B.3.10. Post Test

B.3.10.1. Post-Test Vapor Samples. After the combined ISVE/air sparging test is completed, post test soil vapor samples will be collected. Vapor samples will be collected from the extraction trench manifold and each vapor monitoring probe and analyzed in the field for CO_2, O_2, and total volatile hydrocarbons. The SOP for post-test sampling is identical to the pre-test vapor sampling. Post-test vapor measurements will be collected at system shut down, and at 0.5, 1, 2, 4, 8, 12, 24, and 48 hours after shut down.

Additionally, within 48 hours after completion of the combined ISVE/air sparging test, post-test vapor samples will be collected from the extraction trench manifold and from each vapor monitoring probe and submitted to an off-site laboratory for VOC and SVOC analyses.

B.3.10.2. Post-Test Groundwater Samples. Within 48 hours after the completion of the ISVE/air sparging test, groundwater samples will be collected from the four groundwater monitoring wells for technology evaluation. The samples will be sent to an off-site laboratory for the analyses listed in Table B-1.

B.3.10.3. Post-Test Soil Samples. Within two weeks of the end of the combined ISVE/air sparging test, post-test soil samples will be collected. The sample locations will be from the same depth interval of the corresponding pre-test soil samples and from soil borings drilled within two feet of the borings drilled for pre-test soil sampling. The borings will be drilled and sampled according to the SOPs in Appendix G. The soil samples will be submitted to an off-site laboratory for the analyses listed in Table B-1.

B.3.11. Data Evaluation and Design Criteria

The data generated from the pilot study will be used to:

- Evaluate the effectiveness of ISVE/air sparging at removing contaminants from the contaminated soils in the ONCA and assess its potential to meet the performance standards,

- Evaluate ISVE/air sparging for enhancing naturally-occurring biodegradation of the contaminants, and

- Determine design parameters for full-scale implementation of ISVE/air sparging in the ONCA.

B.3.11.1 Effectiveness of Technology. The effectiveness of ISVE/air sparging for removing contaminants and the potential for the technology to meet the performance standards will be evaluated based upon:

- The mass of contaminants removed during the ISVE/air sparging pilot study, calculated based upon extracted vapor concentrations and vapor extraction flow rates and mass balance calculations,

- Comparison between pre-test and post-test soil, groundwater, and soil vapor sampling results,

- Predicted cleanup time and ability to meet performance standards as determined using an estimate of the mass of a particular contaminant in the area and a mass removal rate calculated from the pilot study data. The mass removal rate will be estimated based on extracted vapor concentrations observed during the test. The potential cleanup time will then be calculated by dividing the estimated mass by the estimated removal rate. If the cleanup time calculated by this method is greater than ten years, it is not likely that ISVE/air sparging will effectively meet the cleanup goals. The concentration reductions observed during the pilot testing will also be used to assess whether or not the technology can meet the performance standards.

B.3.11.2. Bioremediation Enhancement. The effectiveness of ISVE/air sparging for enhancing naturally-occurring biodegradation of the contaminated soil will be determined by evaluating the pre- and post-test vapor sampling results. If oxygen is the limiting factor in biodegradation, the pre-test soil vapor samples in the contaminated area should exhibit elevated CO_2 concentrations (in the range of 5% to 15%), depressed O_2 concentrations (<5%), and elevated hydrocarbon concentrations. Background soil vapor concentrations should be similar to atmospheric concentrations for CO_2 and O_2, with hydrocarbon concentrations generally less than 100 ppm. During the ISVE/air sparging test, the O_2 concentration in the soil vapor in the test area should increase to approximately 20%. Using the O_2 data from the 48-hour post-vapor sampling test, an oxygen utilization rate will be calculated. If the utilization rate is within the range of 0.05% to 1% O_2/hour, this will be sufficient evidence that some microbial activity is occurring and that the addition of O_2 in this area will enhance biodegradation.

Ideally, the utilization rate from the post-test vapor sampling should be compared to a utilization rate from a background vapor monitoring probe. Unfortunately, the probe would need to be very shallow (and therefore would reflect atmospheric conditions) and would need to be located off of the site. In general, little or no oxygen utilization is present in uncontaminated background areas. Consequently, a background vapor probe for this purpose is not believed to be of significant value.

In addition to the above, microbial plate counts in pre-test and post-test soil and groundwater samples will be compared to see if the microbial population has noticeably increased. Analytical data for specific constituents required for biological growth will also be evaluated to determine if other factors could be limiting biodegradation.

B.3.11.3. Design Parameters. The design parameters for full-scale ISVE/air sparging will be determined as follows:

- An ISVE blower operating curve will be generated for the extraction trenches by plotting vapor extraction flow rate per foot of trench screen versus vacuum at the extraction trench manifold. The operating curve will be used during full-scale ISVE system design to determine the vapor extraction flow rate and vacuum to be used for the full-scale system.

- Extracted vapor concentrations at the vapor extraction trenches, volumes of extracted condensate, and condensate composition will be used to design the offgas treatment system.

- Extracted vapor concentrations at the exhaust of the blower will be used to verify removal efficiencies.

- The effective treatment influence of the vapor extraction trenches and the air permeability of the soil will be calculated based upon the vacuum measurements at the vapor monitoring probes and the vacuum and flow rate measurements at the vapor extraction trenches. Air permeability calculations are included in Appendix F.

- The radius of influence of the air injection system will be determined from field observations and calculations included in Appendix F. Field observations of well head pressure and groundwater elevation changes at each monitoring well and piezometer will be used in the calculations. Additionally, mounding of the water table within the testing area will delineate the radius of influence of the air sparging wells. Radius of influence calculations will be compared to the field observations before finalizing full-scale radius and well spacing.

- The optimum air sparging flow rate and influence of air sparging on the groundwater table elevation will be determined. These parameters will be determined from field observations during the air sparging flow rate tests as described in Section B.3.8.3. The optimal flow rate will be selected based on field observations of pressure at the well heads and mounding of the water table. Mounding of the water table occurs when the air being injected into the subsurface has reached the maximum lateral extent and begins to vertically displace formation water. The flow rate at which mounding begins to occur will be considered the maximum flow rate for this system. Due to the shallow nature of the water table in this area, mounding of the water table must be kept at less than one foot from the static groundwater elevation. The optimum flow rate will be selected as the maximum flow rate that can be maintained while keeping mounding less than one foot. Ideally, the flow rate selected will have

minimal effects on mounding of the water table. If mounding of the water table occurs at very low flow rates, then the possibility of pulsed sparging will be explored. This would allow a full-scale system to operate at a flow rate that is effective at stripping contaminants from the saturated zone over short periods of time, depending on the length of time it takes for mounding to occur. The system would then shut down to relieve the mounding effects and then start up again, thus having a pulsing effect. This would alleviate problems associated with mounding of the shallow water table.

B.4.0 SAMPLE DESIGNATION

All samples collected will have the study area prefix "VEAS" to define which pilot study the sample comes from. Location designators are as follows: air sparging wells (AS) plus a well number, monitoring well (MW) plus a well number, vapor probes (VP) plus a probe number, piezometers (PZ) plus a piezometer number, vapor extraction trench (VET) plus a trench number, extracted vapor (EV), treated vapor (TV), condensate (CD), purge water (WP), and confirmation soil borings (SB) plus a soil boring number. The media designations are as follows: soil samples (SO), groundwater (WG), and soil vapor (SV). A sequential number will be added to each sample collected for laboratory analysis. In addition, soil samples collected during the installation of the air sparging wells, monitoring probes, or confirmation soil sampling will have the sample interval in feet bgs (i.e., 5.0 for 5 feet bgs). Table B-2 provides a summary of sample identification.

B.5.0 DECONTAMINATION PROCEDURES

To prevent the introduction of contaminants into the subsurface during drilling or sampling, all downhole drilling and soil (or groundwater) sampling equipment will be steam cleaned prior to use. All excavation equipment used in trenching will also be steam cleaned prior to use. After steam cleaning, all drilling and excavation equipment will be kept off the ground until it is used. Lubricants will not be used on any equipment sent downhole or placed in trenches. The drill rigs and backhoes used during the pilot study will be monitored for hydraulic leaks and for the presence of oil or grease.

To prevent cross contamination of the soil samples collected during drilling, the split-spoon soil sampler, catcher, brass sleeves, and stainless steel knives and trowels will be decontaminated between sample intervals using the following procedure:

- Wash with laboratory-grade detergent (Alconox)
- Rinse with approved water
- Rinse with isopropyl alcohol
- Air dry
- Triple rinse with double deionized water
- Air dry.

B.6.0 SAMPLE HANDLING AND ANALYSIS

B.6.0.1. Chain-of-Custody. A project specific Chain-of-Custody (COC) form will be completed and will accompany each sample cooler. The COC form includes project identification, project location, sample location, sample designation, sample container type, analysis type, field lot control number, and cooler identification. In addition, there are spaces for entry of the sample collection date and time, sample collection technique, signature of the persons relinquishing and receiving samples, and the status of the samples upon receipt by the laboratory. A project specific COC is provided in Section 3.0 of the QAPP.

Additional sample handling and analytical procedures are provided in the QAPP

B.7.0 SAMPLE DOCUMENTATION

B.7.0.1. Documentation Procedures. The field analytical data generated from the pilot studies will be recorded on the forms provided in Appendix F. In addition, on-site personnel will maintain a field logbook. The field logbook will be a weather resistant, bound, survey-type book, with non-removable pages. Information that will be entered in the logbook includes the name and location of the job, personnel on site, name and address of the field contact person, the date(s) sampling was started and completed, weather conditions, the calibration methods used for the organic vapor detectors, sampling location, sampling methodology, sample destination and how it was transported

(e.g., name of shipping agent), any deviations from the FSP, and any other observations that may be relevant to the field program.

B.8.0 DATA REDUCTION AND EVALUATION AND REPORTING

All data generated during the pilot study will be reduced, evaluated, and reported following procedures outlined in the QAPP.

B.9.0 COMPLIANCE REQUIREMENTS

B.9.0.1. Drill Cutting Handling Procedures. All soil cuttings produced during drilling of wells and piezometers will be containerized in Department of Transportation (DOT)-approved 55-gallon drums. The presence of VOCs will be monitored using an organic vapor detector. In addition, the on-site personnel will document any visual signs of contamination (e.g., discolored or odoriferous soil). All drums will be labeled with the following information:

- American Chemical Service Site
- Borehole number
- Date
- Type of material
- Depth interval
- Maximum organic vapor reading

All drums will be moved to a staging area. Drums containing soil with organic vapor detector readings that indicate the presence of contaminants or drums that contain soil that had other indications of contamination will be segregated. After the scheduled analyses for the soil samples are completed, the data will be reviewed to determine the types of contaminants that may be present and the proper disposal method for the cuttings.

B.9.0.2. Excavation Spoils Handling Procedures. All excavated materials produced during trenching will be stockpiled on site on polyethylene sheets. The material will be covered with polyethylene sheeting and kept on site until implementation of the remedy.

B.9.0.3. Purge and Decontamination Water Handling Procedures. All purge and decontamination water generated during the pilot testing will be placed in containers (e.g. Baker or Frac tanks) and stored on-site. The water will then be sent to the on-site water treatment plant once it is operational.

B.9.0.4. Vapor Treatment Unit Off-gas. Based on the concentration of contaminants in the subsurface, ISVE/air sparging operation will generate air emissions that will need to be treated. Either a carbon treatment unit or a catalytic oxidizer treatment unit will be installed to treat air emissions during both the short and long-term studies. The vapor treatment unit will be capable of treating the extracted vapors to the applicable air discharge limits (i.e., IDEM air quality standards and the 1×10^{-4} to 1×10^{-6} cumulative cancer risk range as stated on page 30 of the ROD). For this study, activated carbon canisters will most likely be used for off-gas treatment. The need for and design details of additional emissions control will be finalized prior to mobilization based upon input from system suppliers (regarding removal efficiencies) and based upon review of the proposed approach by the IDEM Office of Air Management. Air samples will be collected from the vapor treatment unit and analyzed in the field for total hydrocarbons using an FID. Air samples also will be collected and submitted for analysis on a bi-weekly basis. The samples will be analyzed for the parameters detailed in Table B-1. Should the results of the vapor treatment unit off-gas indicate failure in reducing air emissions to below the IDEM standards, the long-term testing will be terminated until the vapor treatment unit is replaced with a unit capable of meeting the discharge limits. In addition, if the vapor treatment unit should fail during the testing, the test will be interrupted until the unit is functional.

B.9.0.5. ISVE/Air Sparging System Condensate. The volume of condensate produced during the ISVE test will be measured on a daily basis. Samples of the condensate will be collected on a bi-weekly basis and submitted for the analyses specified in Table B-1. The condensate volume will be measured on a daily basis. The condensate will be placed with the purge and decontamination water and ultimately treated.

TABLE B-1

ISVE/AIR SPARGING PILOT STUDY ON CONTAMINATED SOIL IN THE ONCA
SUMMARY OF ANALYTICAL SAMPLES

Task (Duration)	Total Number of Samples	Frequency	Matrix	Required Analyses
Pre-Test Sampling				
Install two air injection well borings (2 samples per boring; 1' and 10' bgs.)	4	Once	Soil	VOCs, SVOCs, Pest/PCB, metals, Cr^{+6}, TOC, TKN, o-phosphate, plate count, grain size distribution, moisture content
Install four monitoring well/vapor probe borings (2 samples per boring; 1' and 10' bgs.)	8	Once	Soil	VOCs, SVOCs, Pest/PCB, metals, Cr^{+6}, TOC, TKN, o-phosphate, plate count, grain size distribution, moisture content
Pre-test sampling of vapor probes and extraction trench manifold	5	Once	Vapor	CO_2, O_2, and TVH (field analyses)
Pre-test sampling of monitoring wells	4	Once	Water	VOCs, SVOCs, Pest/PCB, PCP, metals, cations, chloride, fluoride, sulfate, sulfide, ammonia, nitrite/nitrate, total organic carbon,
ISVE Testing (2 weeks)				
Initial sampling of vapor probes and extraction trench manifold	5	Once	Vapor	VOCs, SVOCs, methane and ethane
Extraction trench manifold and vapor treatment unit offgas samples collected	70	Every 8 hours the first week Every 12 hours the second week	Vapor	TVH (field analysis)
Vapor samples collected from extraction trench manifold	5	Every 2 days the first week Twice the second week	Vapor	VOCs, SVOCs
Vapor samples collected from vapor treatment unit offgas	1	Once	Vapor	VOCs, SVOCs
Sampling of condensate from ISVE system	2	Once/week	Water	VOCs, SVOCs, Pest/PCBs, PCP, metals
Air Sparging Testing (2 weeks)				
None				

TABLE B-1

ISVE/AIR SPARGING PILOT STUDY ON CONTAMINATED SOIL IN THE ONCA
SUMMARY OF ANALYTICAL SAMPLES

Task (Duration)	Total Number of Samples	Frequency	Matrix	Required Analyses
Combined ISVE/Air Sparging Testing (2 weeks)				
Extraction trench manifold and vapor treatment unit offgas sampling	70	Every 8 hours the first week Every 12 hours the second week	Vapor	TVH (field analysis)
Vapor samples collected from extraction trench manifold	5*	Every 2 days the first week Twice the second week	Vapor	VOCs, SVOCs
Vapor samples collected from vapor treatment unit offgas	2*	Once/week	Vapor	VOCs, SVOCs
Sampling of condensate from ISVE system	2*	Once/week	Water	VOCs, SVOCs, Pest/PCBs, PCP, metals
Post-Test Sampling				
Post-test vapor measurements at each vapor monitoring probe and the extraction trench manifold (48 hours immediately after test completion)	104	Immediately after shut down, and 0.5, 1, 2, 4, 8, 12, 24, and 48 hours after shut down	Vapor	CO_2, O_2, and TVH (field analyses)
Post test-vapor samples collected from each vapor probe and the extraction trench manifold (within 48 hours of test completion)	5	Once	Vapor	VOCs, SVOCs, methane and ethane
Post-test groundwater samples collected from each monitoring well (within 48 hours of test completion)	4	Once	Water	VOCs, SVOCs, Pest/PCB, PCP, metals, cations, chloride, fluoride, sulfate, sulfide, ammonia nitrite/nitrate, total organic carbon,
Install six soil borings in the vicinity of the two air injection wells and the four monitoring well/vapor probes. Samples at 1' and 10' bgs in each boring (within 2 weeks of test completion)	12	Once	Soil	VOCs, SVOCs, Pest/PCB, metals, Cr^{+6}, TOC, TKN, o-phosphate, plate count, grain size distribution, moisture content

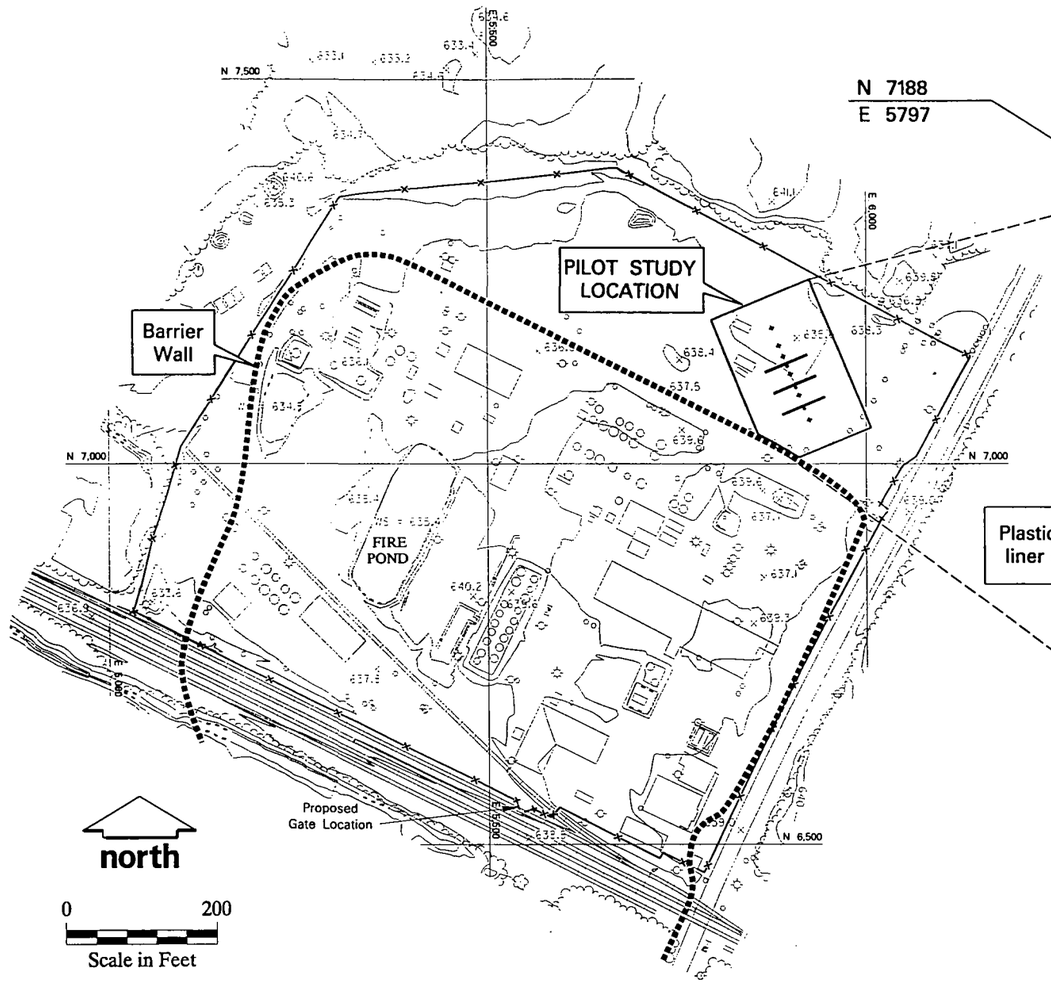
See Table 1-2 in the QAPP for analytical methods
See Table 3-5 in the QAPP for QA/QC samples
* Based on a 2 week test duration

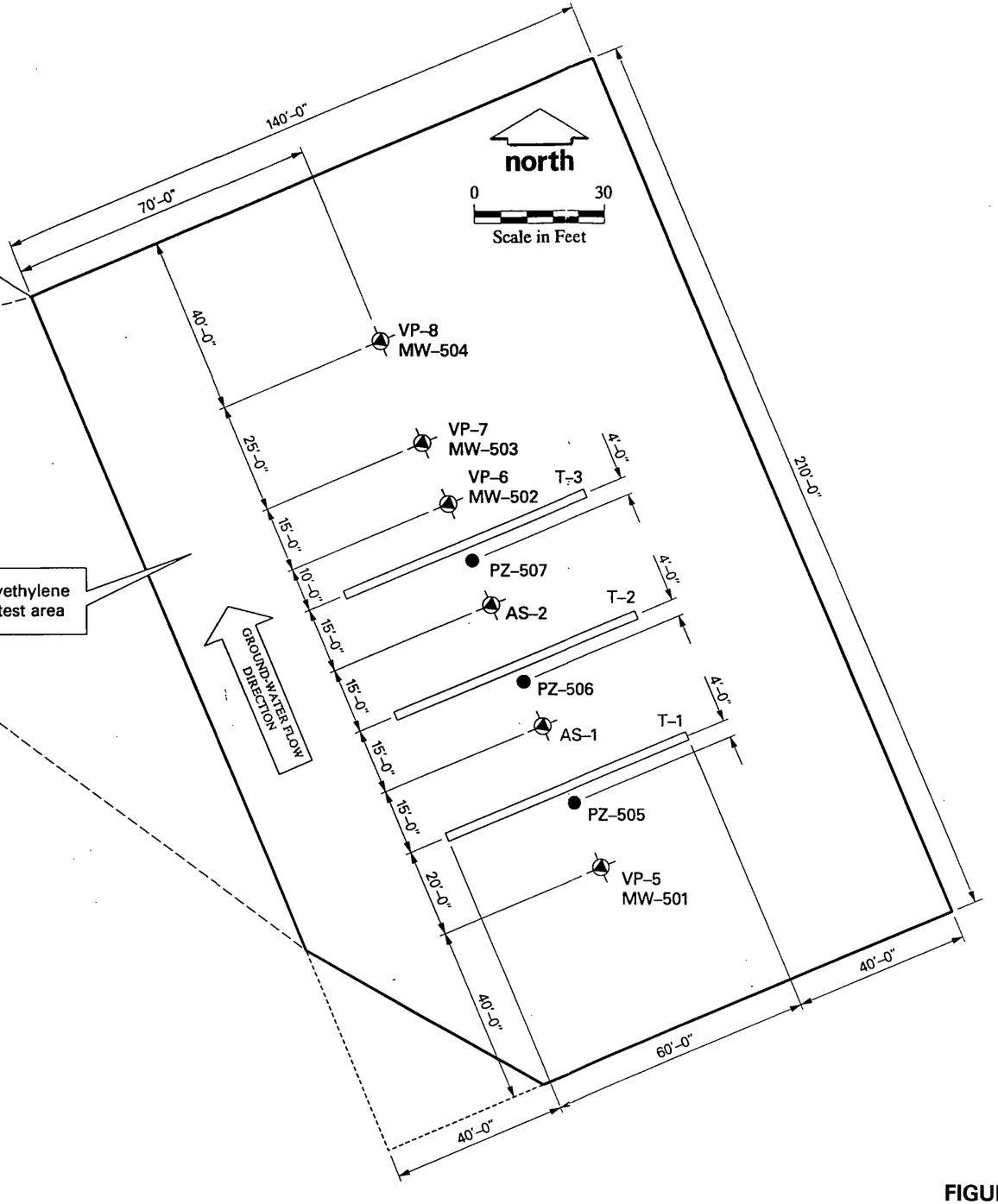
TABLE B-2

ISVE/AIR SPARGING PILOT STUDY ON CONTAMINATED SOIL IN THE ONCA
SUMMARY OF SAMPLE IDENTIFIERS

Pilot Study Area	Sampling Location	Well Identification	Sample Medium	Sample Depth (feet)	Sequential Identifier
In-Situ Vapor Extraction (ISVE)/Air Sparging	Air Sparging Well (AS)	1 and 2	Soil (SO), Soil Vapor (SV)	5 & 15 feet bgs	1001-1200
	Monitoring Well (MW)	501 to 504	Soil (SO), Groundwater (WG)	15 feet bgs	1201-1300
	Vapor Probe (VP)	5 to 8	Soil (SO), Soil Vapor (SV)	5 feet bgs	1301-1400
	Extraction Vapor (EV)	NA	Soil Vapor (SV)	NA	1401-1500
	Treated Vapor (TV)	NA	Soil Vapor (SV)	NA	1501-1600
	Confirmation Soil Boring (SB)	506 to 511	Soil (SO)	5 &15 feet bgs	1601-1700
	Condensate (CD)	NA	Groundwater (WG)	NA	1701-1800
	Purge Water (WP)	NA	Groundwater (WG)	NA	1801-1900
	Piezometer (PZ)	505-507	NA	NA	NA
	Vapor Extraction Trench (VET)	501-503	NA	NA	NA

NA Not Applicable





LEGEND

- Groundwater/vapor monitoring well
- Piezometer–groundwater extraction well

FIGURE B–1

Developed By	Drawn By
Approved By	Date 8/26/96
Reference	
Revisions	

ISVE/AIR SPARGING PILOT STUDY LOCATION MAP AND LAYOUT ON SOILS IN THE ONCA

TREATABILITY STUDY
AMERICAN CHEMICAL SERVICE, INC.
NPL SITE
GRIFFITH, INDIANA

Drawing Number 4077.0191

MONTGOMERY WATSON

Management Review — Other
Technical Review — Project Manager
QUALITY CONTROL — Graphic Standards — Lead Professional

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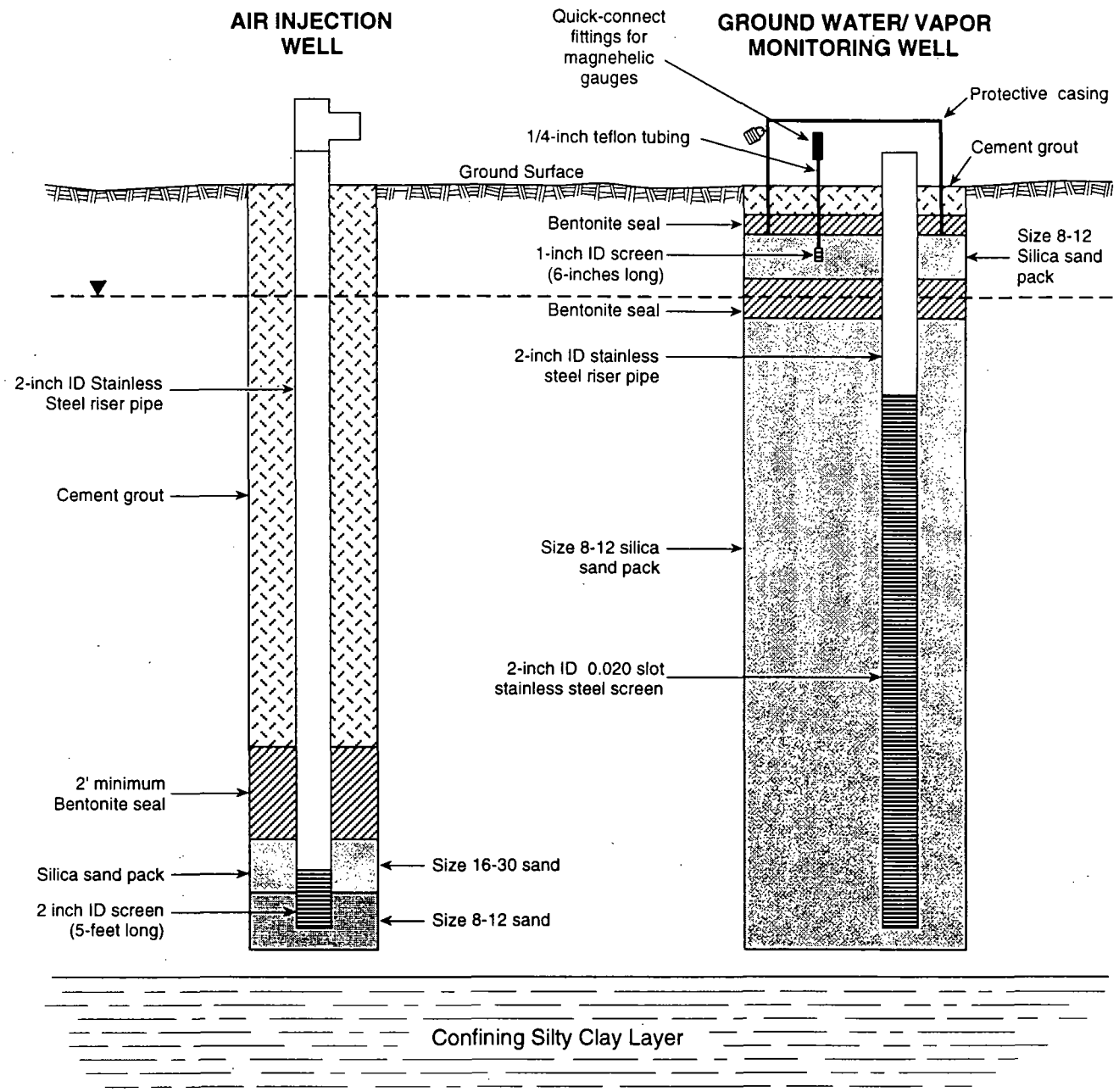
QUALITY CONTROL
Graphic Standards Lead Professional
Technical Review Project Manager
Management Review Other



Note: This drawing is not to scale.

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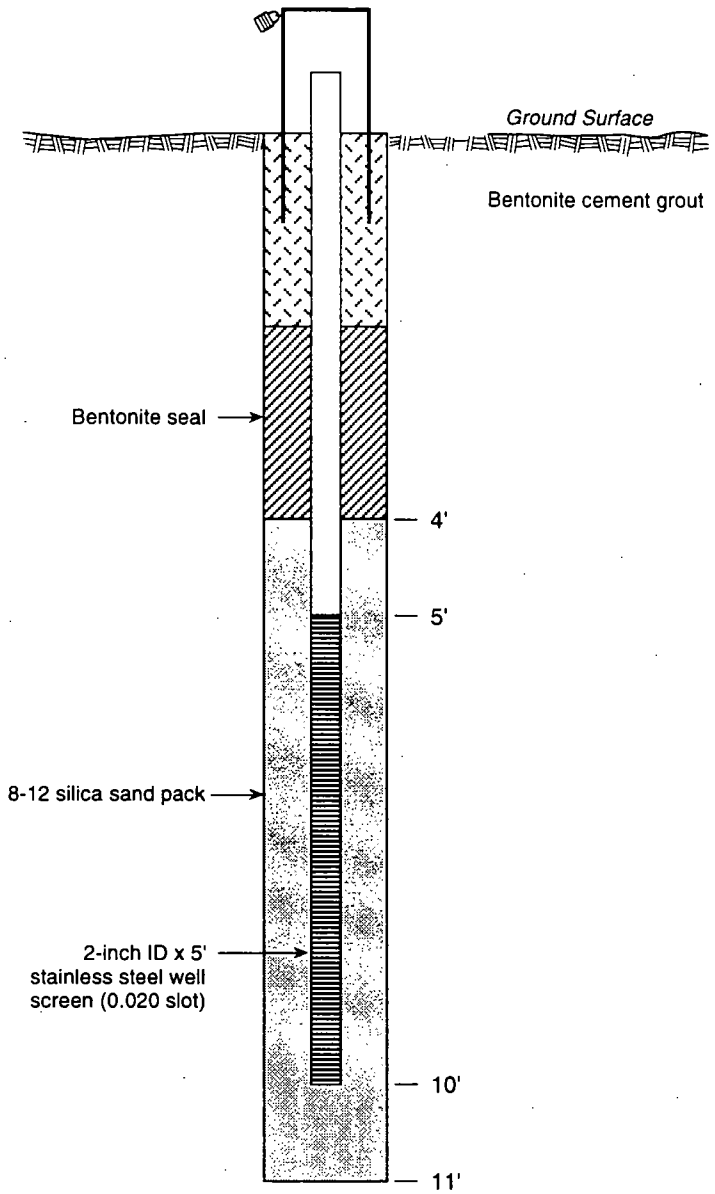
FIGURE B-2

Developed By	Drawn By	**AIR INJECTION AND MONITORING WELL DESIGN**	Drawing Number 4077.0191
Approved By	Date *8/12/96*		**MONTGOMERY WATSON**
Reference		TREATABILITY STUDY AMERICAN CHEMICAL SERVICE, INC. NPL SITE GRIFFITH, INDIANA	
Revisions			

QUALITY CONTROL | Graphic Standards Lead Professional | Technical Review Project Manager | Management Review Other



PIEZOMETER DETAIL

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FIGURE B-3

Developed By	Drawn By	**PIEZOMETER DESIGN**	Drawing Number 4077.0191
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	Graphic Standards _____	Technical Review _____	Management Review _____
	Lead Professional _____	Project Manager _____	Other _____

Side View

Cross-Section

Plan View

FIGURE B-4

VAPOR EXTRACTION TRENCH DESIGN

TREATABILITY STUDY
AMERICAN CHEMICAL SERVICE, INC.
NPL SITE
GRIFFITH, INDIANA

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QUALITY CONTROL	Graphic Standards	Technical Review	Management Review
	Lead Professional	Project Manager	Other

FIGURE B-5

ISVE/AIR SPARGING SYSTEM
PROCESS SCHEMATIC

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APPENDIX C

PRETREATMENT/MATERIAL HANDLING PILOT STUDY ON BURIED WASTE IN THE OFF-SITE CONTAINMENT AREA

FIELD SAMPLING PLAN AND TESTING PROCEDURES

APPENDIX C

PRETREATMENT/MATERIAL HANDLING PILOT STUDY ON BURIED WASTE IN THE OFF-SITE CONTAINMENT AREA

FIELD SAMPLING PLAN AND TESTING PROCEDURES

APPENDIX C - FIELD SAMPLING PLAN

PRETREATMENT/MATERIALS HANDLING STUDY
AMERICAN CHEMICAL SERVICES SITE

SUBMITTED TO:
MONTGOMERY WATSON

Revision 1
December 5, 1996
Focus Project No. 119406

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A Soil Boring Log Sheets Relevant to PMHS Test Pits

B Auger Probe Data Relevant to PMHS Test Pit Selection

C.1.0 INTRODUCTION

C.1.1 OVERVIEW

The U.S. EPA, Region V, has determined that thermal desorption is the remedial technology of choice for the American Chemical Service Inc. NPL site, (ACS site) located in Griffith, Indiana. Previous investigations have indicated that significant amounts of debris present are at the site which could impact technology application. The purpose of the Pretreatment/Material Handling Study (PMHS), is to investigate the ability to separate debris from site soils and collect soil samples for treatability testing. This plan details the requirements of the Pretreatment/Material Handling Study.

C.1.2 OBJECTIVES

The primary objectives of the Pretreatment/Materials Handling Study (PMHS), include :

- Assess the type and quantity (percentage) of debris in representative soils to be remediated
- Assess the ability of conventional materials handling equipment to effectively separate debris from soils to be thermally treated
- Determine the magnitude of the VOC fugitive emissions during excavation activities
- Collect soil samples for thermal treatability testing

C.1.3 PROJECT ORGANIZATION AND RESPONSIBILITY

Focus Environmental will provide PMHS oversight and ambient air monitoring associated with fugitive emissions. The site work, which will involve full-scale excavation and screening equipment and all associated health and safety activities, will be subcontracted to a remediation contractor. Health and Safety oversight will be provided by Montgomery Watson personnel. All operations during the PHMS will be documented by Focus Environmental using cameras, video equipment, and written data logs.

C.2.0 STUDY DESIGN

C.2.1 GENERAL APPROACH

The Offsite Containment Area is the only area of the ACS site that was chosen for this treatability test. This area was chosen due to the large quantity of materials anticipated to require remediation and the contaminant variety and concentrations found in this area. Soil borings from previous site investigations conducted by Montgomery Watson (formerly Warzyn), indicated contaminant concentrations frequently in the percent level in the Off-site Containment Area. With the use of the analytical data obtained from the earlier site investigation, five test pit locations were identified. These test pits will provide representative soils from different locations throughout the Offsite Containment Area which will serve as the test soils for full-scale material screening and the soil samples needed for the thermal desorption treatability test. During the active on-site portion of the treatability test, ambient air monitoring will be used to determine VOC fugitive emissions from the material handling activities.

C.2.2 EQUIPMENT DESCRIPTION

Excavation of soil during the PMHS will require a back hoe, and a high lift front end loader for moving the soils to the screening location. Physical size screening of all excavated materials will be used to segregate debris which may not be effectively treated using thermal desorption technology. The screening operations will require a vibratory screen with a coarse screen (6" grizzly), and a medium screen (2 inch). A ¾ inch screen will also be available if it is determined by visual observation that the excavated soils contain organic debris smaller than 2 inches that could cause problems in a thermal desorption system. It is anticipated that the ¾ screen will not be necessary. The screen will be powered by a portable generator supplied by the equipment contractor. Also a truck scale will be needed for weighing soils and debris.

Air monitoring will be conducted using Summa canister technology. Section C.5.1 gives a more detailed description of equipment involved in this technology.

C.2.3 RATIONALE FOR TEST PIT LOCATIONS

The soils excavated from the Offsite Containment Area for screening and treatability testing should be as representative of site soils as possible, and representative of the contaminant types and concentrations present at the site. Therefore, using the soil boring analyses and auger probe descriptions from earlier testing by Montgomery Watson, five test pits were identified. Figure C-1 locates the five test pits on a map of the Off-site Containment Area. Exact pit locations will be adjusted in the field to avoid excessive vegetation that would present excavation difficulties at the planned locations.

Test Pit 1 is in the exact location of soil boring (SB) 37. This location was selected particularly for the higher levels of PCB's shown in the SB37 analysis. Test Pit 2 is in the exact location of SB36. No relatively high concentration of contaminants are evident from the soil boring analysis. However, boring logs for AP12, which is in close proximity to SB36, indicate evidence of debris and waste at ten feet below the surface. Test Pit 3 is in the exact location of SB06. This location was selected because it appears to have high concentrations of nearly all of the contaminants under consideration. Of particular interest is the high level of lead relative to other soil borings. Test Pit 4 is in the vicinity of SB07. This location was selected for its very high VOC concentrations. Test Pit 5 is in the vicinity of SB39. This location was selected because of the relatively high concentrations of phthalates. VOC's were also detected at high concentrations in this area.

Table C-1 lists approximate site coordinates of test pit locations which will aid in the physical location of these areas in the field, and includes analytical results which were considered when the proposed test pit locations were determined (i.e. SB analysis results). Attachments A and B contain copies of boring logs, and auger probe observations, respectively.

C.3.0 STUDY EXECUTION

C.3.1 CONTRACTING AND MOBILIZATION

After approval has been obtained from the US EPA and the PRP Group to proceed, Focus will negotiate a contract with a remedial contractor to conduct the full-scale screening tests. After a contract has been signed and the tests have been scheduled, mobilization will begin.

Mobilization will involve location and staging of test pits to be excavated and setup of screening equipment and weigh scale. If appropriate power is not available to operate the vibrating screen, a portable generator will be mobilized to the site to power the screen. Screening equipment will be field located in a central area with convenient access from any of the five test pits.

C.3.2 LOCATION OF TEST PITS

Test pits will be located in the field with the use of the test pit location figure (Figure C-1), and the reference coordinates corresponding to those locations shown in Table C-1. The 20 foot square dimensions will be staked and appropriate safety zones established as needed.

C.3.3 EXCAVATION

C.3.3.1 Excavation Guidelines

All test pits will have approximate areal dimensions of 20 feet by 20 feet by 10 - 15 feet deep, with approximately 100 - 400 tons of soil being removed per test pit. The actual tonnage removed from each pit is not a critical parameter. Therefore, flexibility will be exercised in relation to the actual size and total tonnage removed from each pit in order to obtain pertinent data in support of the test objectives.

The primary factors that will be evaluated to determine the quantity of materials to be excavated from each pit will be safety, debris content, and previous samples indicating contamination. Excavation will continue in each pit until one or more of the following conditions has occurred: 1) debris is no longer being encountered, 2) excavated depth is below the deepest depth where contamination was previously identified, 3) side walls of pit become unstable from action of water in the pit. The greater the quantity of material removed from the contaminated area, the more representative the results of the study will be of the total site.

Because the soil is primarily sand or silty sand the contractor will be required to pay special attention to the sloping of the excavation pits or trenches to minimize the potential for cave-ins. No personnel will be allowed in the test pits.

C.3.3.2 Stockpiling Guidelines at the Test Pits

Groundwater at the site was found to be 4 to 6 feet below ground surface during the RI. Since the test pits are going to be excavated to depths lower than 5 feet, the excavated material will need to be drained prior to screening. Therefore, prior to the start of excavation at each test pit, the high side of each pit will be determined and preparations made for stockpiling the newly excavated material on the high side. A minimum eight inch berm will be created around the stockpile area using site surface soils from the stockpile area. The berm will encircle an area large enough to hold the excavated soil from the pit, with the natural gravity drainage route directed back into the excavation. Plastic sheeting with a minimum thickness of 6 mil will be placed on this area prior to excavation and placement of contaminated soils. Soil from the test pit deemed to be clean fill material will be staged in an area adjacent to the pit to provide cover material when the excavations are refilled prior to demobilization. Plastic sheeting and hold downs will be available to cover the exposed excavated soil in case of rain, or if odors become a significant problem off-site.

C.3.3.3 Fugitive Emissions

To assess the extent of VOC emissions, air sampling will be conducted by Focus during the testing. Refer to Section C.5.0 of this plan for air sampling details. The short duration of the project (2 or 3 weeks) will minimize the VOC emissions from the site. To further minimize the potential for VOC emissions, no more than three pits will be open at any one time. Also, plastic sheeting will be available to cover stockpiles to minimize emissions if off-site odors become a problem.

C.3.4 SCREENING

C.3.4.1 Location of Screen

The screening equipment will be located as close to all five test pits as possible, allowing easy access without impeding other operations at the site.

C.3.4.2 Screening Protocol

Material processed through the screen from each test cell will generate two streams: the oversized reject and the screened soil. The oversized reject (debris) will be weighed, and an estimate of the

percentage of the debris which is metal, wood, paper, plastic, etc. will be recorded. The screened soil will be inspected for organic debris that may have passed the 2 inch screen. If by visual observation it appears that the screened soil has greater than 1 wt% organic debris, then the screen will be fitted with the ¾ inch screen and the screened soil will be rescreened to determine separate the small debris from the screen soils. The small debris reject will be collected and composited to form one sample to send to the laboratory for testing as defined in Appendix D. The screened soil from each pit will be mixed with the bucket of the back hoe, sampled, and weighed.

The screening rate will be calculated and recorded in the field logbook by keeping track of the total time required to process a grid's excavated soils. After the total weight of this soil/debris is determined, a rate of tons/hour can be determined.

Screened soil piles will be covered with plastic sheeting to minimize odors if fugitive emissions become a problem during screening.

C.4.0 SOIL SAMPLING

C.4.1 SAMPLING PROCEDURE

Each test pit will have one five gallon composited sample of screened, mixed soil taken and shipped to the treatability test laboratory. An identical amount will be packaged and stored on-site in a secure location as an archive sample. Samples will be taken as follows:

Obtain three grab samples with a clean shovel from eight different locations within the screened, mixed, soil tailings pile. Place the grab samples on a plastic sheet and mix thoroughly breaking clods of soil or debris as much as possible. Quarter the sample and move it to the edges of the sheet. Mix each quarter. Roll the quarters back to the middle of the sheet and mix the entire sample again. By taking several grab samples from the screened soil pile to form the composite and by mixing the grabs thoroughly, the resulting composite will be as representative of the test pit area as possible. Finally, scoop samples into two 5 gallon hazardous waste shipping containers. Fill each sample container completely to the top, leaving no head space. Seal the container to insure airtightness, label appropriately. Affix tamper-proofing tape to the lid and container. The procedure for compositing of the soils from each test pit for purposes of thermal treatability testing is described in Appendix D of the Treatability Test QAPP.

C.4.2 SAMPLE NOMENCLATURE

Sample numbers will have the following format:

AA-P-MMDDYY

Where:

AA = Sample type (SP = soil primary, SA = soil archive)

P = Pit number (1 = Pit #1, 2 = Pit #2, etc.)

MM = Month sample was taken

DD = Day Sample was taken

YY = Year Sample was taken

Example 1: Primary soil sample taken from Pit 5 on August 10, 1996 --- SP-5-081096

Example 2: Archive soil sample taken from Pit 1 on 08/11/96 --- SA-1-081196

C.4.3 SAMPLE CUSTODY

All samples taken will be logged on a Chain of Custody form by the sampler. The chain-of-custody form contains information on the date and time of sample collection, the sampler, the project name and

number, the number of containers of each sample being shipped, and any remarks about the sample, including matrix type and preservatives used, prior to shipment. The chain-of-custody form is enclosed with the samples after it has been signed by the Sampler. The form is then signed each time sample possession changes, with the signature of the person relinquishing and receiving the sample. The time of the exchange will be indicated on the form.

The chain-of-custody form will serve as the formal custody record. This form may be revised to meet field conditions, as necessary. Chain-of-custody forms will be executed and retained as follows:

- One copy retained by the Sampler
- Original sent with samples to the treatability test laboratory

(See the thermal treatability study plan, Appendix D, for sample receipt and handling at the treatability test laboratory.)

C.4.4 SAMPLE PACKAGING AND TRANSPORTATION

Samples are to be shipped for overnight delivery to the treatability testing laboratory. Sample packaging and shipping procedures are as follows:

- The soil samples will be placed in 5 gallon plastic containers with sealable lids.
- Each container will be checked for a properly completed sample identification label.
- Each label will include at a minimum, sampler's name, address and phone number, sampling date, sample number, and sample description.
- Each container will be packaged, labeled and shipped to the treatabilility testing laboratory, in compliance with regulations for hazardous waste shipment.
- The treatability test samples will be shipped to International Technology, Technology Development Laboratory in Knoxville, TN.

C.4.5 FIELD SCREENING FOR PCB's

PCBs are present in the Off-site Containment Area as shown by several soil boring analyses. Therefore it is desirable to have PCBs present in at least one soil sample that will undergo treatability testing. In order to insure that this occurs, on-site field tests for the presence of PCBs in the test pit soils will be conducted using immunoassay technology. An aliquot of the composite soil sample from each test pit will be subjected to immunoassay testing for PCBs per the SOP provided in Attachment A of the QAPP. A 10 mg/kg or greater concentration of PCBs in the soil is desired for treatability testing. If the immunoassay testing indicates that none of the five test pit soil samples have PCB concentrations equal

to or greater than 10 mg/kg, a maximum of two additional pits will be excavated in order to obtain at least one sample containing the desired minimum PCB concentration.

C.4.6 ARCHIVED SAMPLES

Archived samples will be packaged in 5 gallon plastic containers. These samples will be labeled and kept in a secure location at the site, and will be available in the event that the treatability lab requires them. After it is certain that these samples will not be needed as archived samples, they will be available for inspection by potential remedial action contractors and/or regulatory agency personnel later in the project, if needed.

C.5.0 AIR MONITORING

C.5.1 OVERVIEW

Focus Environmental will be responsible for the ambient air monitoring activities on-site with respect to measuring fugitive emissions. During on-site excavation and screening activities, daily eight hour ambient air samples will be collected upwind and downwind of the material handling activities. In addition, an eight hour sample will be taken prior to the start of these activities, in order to establish a background reading. Air samples will be collected using Summa canisters with a flow control system for the collection of daily samples. The canisters will be sent by overnight delivery to the analytical lab for analysis.

Meteorological data will be recorded concurrently with sample collection activities. Such data will include ambient air temperatures, humidity, barometric pressure, and wind speed and direction, and will be obtained through the use of an electronic weather station.

A summary of the results of the ambient air testing, and corresponding meteorological data will be included in the PMHS Report.

C.5.2 SAMPLING PROCEDURE

Ambient air monitoring will be accomplished using Summa canister technology. Samples will be collected by setting the flow controller attached to the Summa canister to intake a constant 500 ml/hr flow. The total sampling time will be eight hours. The sample collected in the canister will therefore be a composite sample of air for that eight hour span. The sampling locations will be approximately 100 feet upwind and 100 feet downwind of each excavation pit.

Samples will be taken as follows:

- Prior to ground breaking activities two eight hour samples will be collected to establish a background VOC measurement. The location for the canisters during this sampling will be approximately 100 feet upwind and 100 feet downwind of the planned excavations.
- During the actual PMHS activities each sampling will include two eight hour Summa canister samples, one upwind, and one down wind of excavation or screening activities.

C.5.3 SAMPLE NOMENCLATURE

Sample numbers will have the following format:

AA-S-MMDDYY

Where:

AA = Sample type (AU = Air Upwind, AD = Air Downwind)

S = Sampling day (0 = Background day, 1 = 1st Sampling day, 2 = 2nd Sampling day, etc.)

MM = Month sample was taken

DD = Day Sample was taken

YY = Year Sample was taken

Example 1: Upwind air sample taken on the second air sampling day, August 6, 1996 --- AU-2-080696

Example 2: Background downwind sample taken August 5, 1996 --- AD-0-080596

A complete list of air samples anticipated to be taken during the PMHS is included in Table C-2.

C.5.4 SAMPLE CUSTODY

Sample custody procedures will follow the same procedures outlined in Section 4.3 for soil sample custody.

C.5.5 SAMPLE PACKAGING AND TRANSPORTATION

After sample collection is complete, an identification tag will be attached to the canister. The canister will be prepared for overnight shipment to the following address:

Quanterra Environmental Services
Air Toxics Laboratory
1721 South Grand Avenue
Santa Ana, California 92705

C.5.6 SAMPLE ANALYSIS

Analytical Method T014 will be used for the analysis of the samples taken. The analysis will be conducted by Quanterra Analytical Laboratories.

C.6.0 DEMOBILIZATION

C.6.1 REPLACEMENT OF SOILS/DEBRIS

All excavated soils and debris will be returned to the test pit from which it originated. The original soil cover will be replaced and compacted with the excavation equipment. The archived samples from each pit will be available for inspection if necessary, by potential remedial action contractors and/or regulatory agency personnel later in the project after it is certain that the samples will not be needed for treatability test, or analysis purposes.

C.6.2 EQUIPMENT DECONTAMINATION

All equipment used in the contaminated zones of the waste site will be decontaminated to prevent movement of contaminants off-site. Decontamination will be required for all vehicles, heavy equipment, and tools that might have been contaminated during work activities.

Equipment that has gross visible contamination will be cleaned using a pick, shovel, or scraper to remove contaminated soil. A high pressure washer will then be used to remove residuals. Chemical additives, such as surface-active detergent will be used to accomplish removal of contaminated material from equipment surfaces, as necessary. Plastic sheeting which has actually come into contact with contaminated soils will be decontaminated by pressure washing and will be disposed of in a Subtitle D landfill. Waste water generated in this process will be collected and stored on-site for future processing in the on-site treatment plant constructed for treating the extracted groundwater.

C.7.0 HEALTH AND SAFETY PROCEDURES

The Health and Safety Plan (HSP) for the on-site remedial design activities is being prepared by Montgomery Watson. On-site activities conducted as part of the PMHS will comply with the requirements established in the approved HSP.

Focus will provide health and safety oversight during excavation and material screening tests, as required. Focus personnel and subcontractors will have to meet the minimum health and safety requirements established in the HSP developed by Montgomery Watson for site characterization activities. Focus does not anticipate any additional requirements necessary for activities conducted by Focus personnel. Any additional health and safety requirements specific to Focus subcontractor activities will have to be defined by the subcontractor during the contracting phase and be submitted to Focus and Montgomery Watson as an addendum to the Montgomery Watson HSP.

During the demobilization phase of the PMHS, all discarded PPE, and other generated wastes will be properly bagged, labeled and disposed of in a Subtitle D landfill.

C.8.0 DATA MANAGEMENT

C.8.1 DOCUMENTATION/RECORDING

A field logbook will be maintained by Focus personnel during all activities conducted on-site. Narratives and pertinent information for documentation of PMHS test activities will be recorded in the logbook. A summary of pit soil/debris characteristics, percents, etc. will be included in the field logbook.

In addition to the logbook and relevant data sheets, VHS video tape and photographs of the excavation, screening and sampling will be taken to provide a visual documentation of the written data.

C.8.2 DATA REDUCTION AND EVALUATION AND REPORTING

Raw data obtained from the activities of the PMHS will be reduced and evaluated by Focus to include calculated data such as the following:

- Total tonnage per pit
- Total tonnage of soil per pit
- Total tonnage of debris per pit
- Percent debris per total pit tonnage
- Percent debris type (i.e. wood, plastic, paper, etc.)
- Screening throughput in tons/hour
- Summary of air sample analyses
- Summary of meteorological data corresponding to air sampling.

A test report will be prepared summarizing the results and observations from the PMHS. The report will contain, at a minimum, the following:

- Description of test equipment
- Description of test pits
- Test procedure description
- Summary of material screening results
- Estimates of types and quantities of debris per test pit
- Summary of ambient air monitoring results
- Discussion of screening feasibility based on test results
- Copy of logbook notes
- Copies of photographs and video tape from tests

It is anticipated that the draft PMHS report will be submitted to the EPA and IDEM within 12 weeks of field mobilization.

TABLES AND FIGURES

Table C-1. PMHS Test Pit Selection and Related Details

Test Pit ID	Approximate Test Pit Locations	Corresponding Boring/Probe Locations (a,b)	Boring Number	Boring Elevations	Concentrations From Soil Borings (µg/kg)			
					VOC's	PCB's	Phthalates	Lead
1	6050N, 5240E	SB37 (c), AP8	SB37	**10 ft**	293,500	**84,000**	---	---
				17 ft	3,865,000	71,000	---	---
2	5980N, 5300E	SB36 (c), AP12	SB36	**10 ft**	87,665	---	---	---
				17 ft	424,300	9,200	---	---
3	6110N, 5325E	SB06 (c), AP16	SB06	**11.5 ft**	15,193,000	---	1,741,000	**955**
				15 ft	3,100,000	30,000	358,900	38
4	6260N, 5360E	SB07, AP18	SB07	**14 ft**	**194,630,000**	---	---	---
				19 ft	8,230,000	44,000	135,690	21
5	6210N, 5150E	SB39, AP71	SB39	**10 ft**	5,274,700	---	**3,444,000**	---
				17 ft	4,219,880	---	3,260	---
				23.5ft	42,945	---	347	---

Notes:

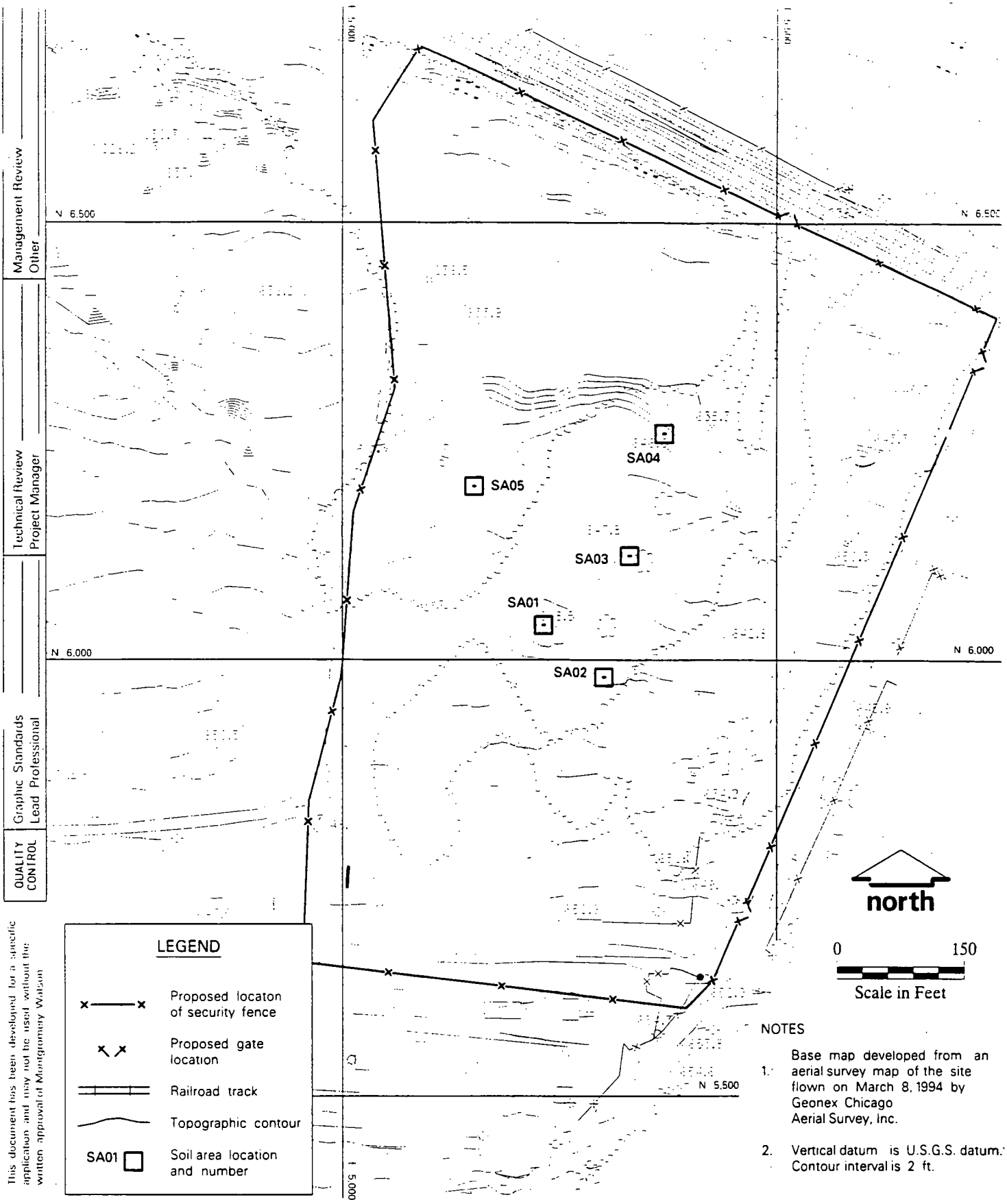
a. Copies of relevant soil boring logs are presented in Attachment A.
b. Relevant auger probe data is presented in Attachment B.
c. The proposed test pits match these previously investigated borings exactly. All other borings or probes are the closest to the proposed test pit location.
d. Data was obtained from Remedial Investigation Report, ACS NPL Site, Griffith, Indiana. June 1991.

Table C-2. Ambient Air Monitoring Samples List

Pretreatment/Material Handling Study
Ambient Air Monitoring Samples (a,b)

Sample Names	Sample Description
AU-0-"Date"	Background air sample taken upwind of the site
AD-0-"Date"	Background air sample taken downwind of the site
AU-1-"Date"	Upwind air sample for Day 1
AD-1-"Date"	Downwind air sample for Day 1
AD-"Date"-R1	Range finding sample #1 - downwind, Day 1
AD-"Date"-R2	Range finding sample #2 - downwind, Day 1
AU-2-"Date"	Upwind air sample for Day 2
AD-2-"Date"	Downwind air sample for Day 2
AU-3-"Date"	Upwind air sample for Day 3
AD-3-"Date"	Downwind air sample for Day 3
AU-4-"Date"	Upwind air sample for Day 4
AD-4-"Date"	Downwind air sample for Day 4
AU-5-"Date"	Upwind air sample for Day 5
AD-5-"Date"	Downwind air sample for Day 5
AD-5-"Date"-D	Duplicate downwind air sample for Day 5

a) All samples will be collected as part of the Pretreatment/Materials Handling Study.
b) All samples will be collected in Summa canisters and analyzed for VOC's using Method TO-14.



Management Review — Other

Technical Review — Project Manager

QUALITY CONTROL — Graphic Standards — Lead Professional

This document has been developed for a specific application and may not be used without the written approval of Montgomery Watson

NOTES

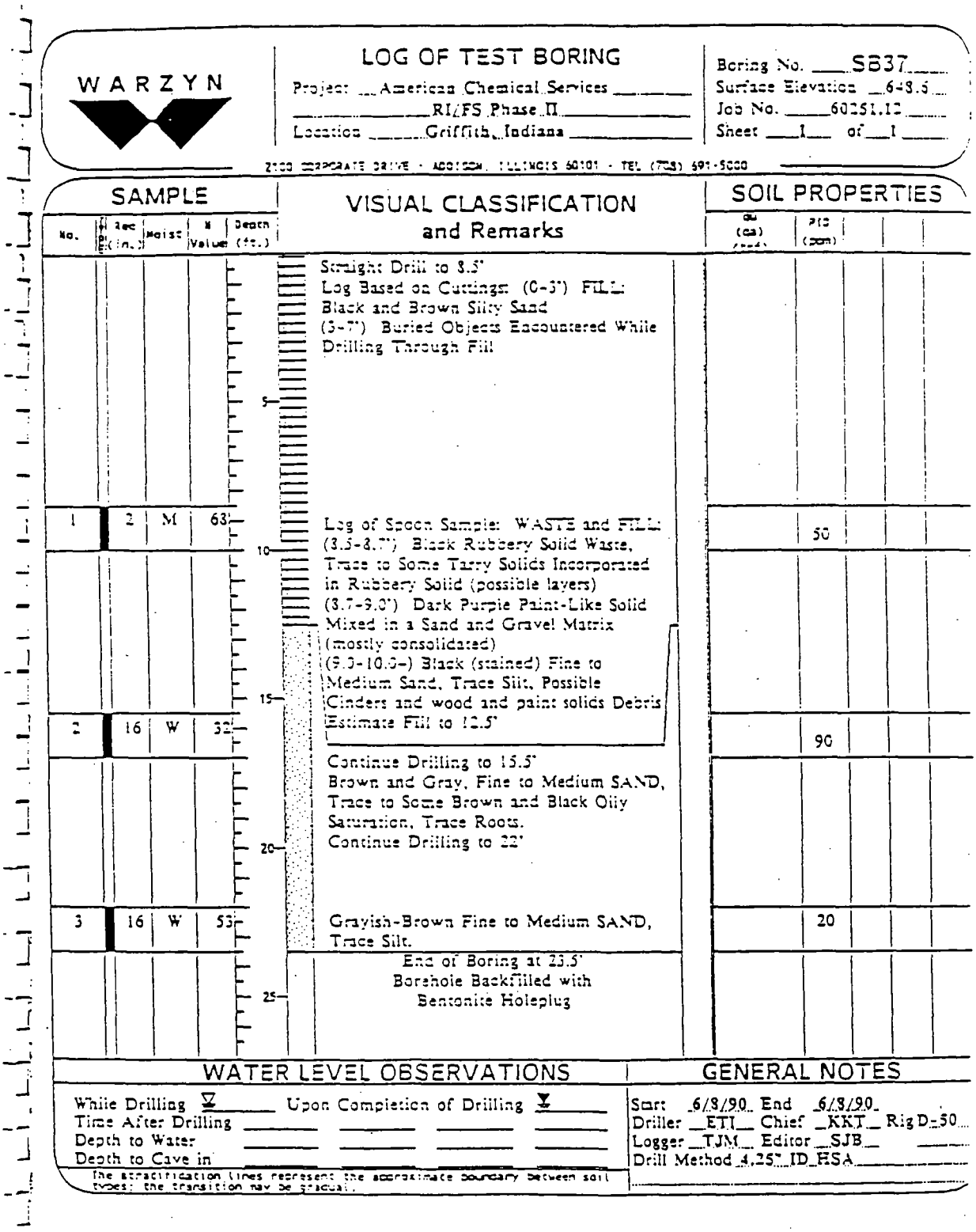
1. Base map developed from an aerial survey map of the site flown on March 8, 1994 by Geonex Chicago Aerial Survey, Inc.
2. Vertical datum is U.S.G.S. datum. Contour interval is 2 ft.

FIGURE C-1

Developed By	Drawn By	**LOCATION MAP PRETREATMENT/MATERIAL HANDLING STUDY IN THE OFF-SITE AREA TEST PIT**	Drawing Number 4077.0060 A12
Approved By	Date		**MONTGOMERY WATSON**
Reference		PRE-DESIGN WORK PLAN AMERICAN CHEMICAL SERVICE, INC. NPL SITE GRIFFITH, INDIANA	
Revisions			

ATTACHMENT A

Soil Boring Log Sheet Relevant to Test Grid #1



WARZYN

2300 CORPORATE DRIVE · ADDISON, ILLINOIS 60101 · TEL (708) 691-5000

LOG OF TEST BORING

Project: American Chemical Services
RI/FS Phase II
Location: Griffith, Indiana

Boring No. SB37
Surface Elevation 648.6
Job No. 60251.12
Sheet 1 of 1

SAMPLE No.	Rec (in.)	Moist	N Value	Depth (ft.)	VISUAL CLASSIFICATION and Remarks	SOIL PROPERTIES OVA (ca)	PID (ppm)
				0	Straight Drill to 8.5' Log Based on Cuttings: (0-3') FILL: Black and Brown Silty Sand (3-7') Buried Objects Encountered While Drilling Through Fill		
				5			
1	2	M	63	10	Log of Spoon Sample: WASTE and FILL: (8.5-8.7') Black Rubbery Solid Waste, Trace to Some Tarry Solids Incorporated in Rubbery Solid (possible layers) (8.7-9.0') Dark Purple Paint-Like Solid Mixed in a Sand and Gravel Matrix (mostly consolidated) (9.0-10.0-) Black (stained) Fine to Medium Sand, Trace Silt, Possible Cinders and wood and paint solids Debris Estimate Fill to 12.5'		50
				15			
2	16	W	32		Continue Drilling to 15.5' Brown and Gray, Fine to Medium SAND, Trace to Some Brown and Black Oily Saturation, Trace Roots. Continue Drilling to 22'		90
				20			
3	16	W	53		Grayish-Brown Fine to Medium SAND, Trace Silt.		20
				25	End of Boring at 23.5' Borehole Backfilled with Bentonite Holeplug		

WATER LEVEL OBSERVATIONS

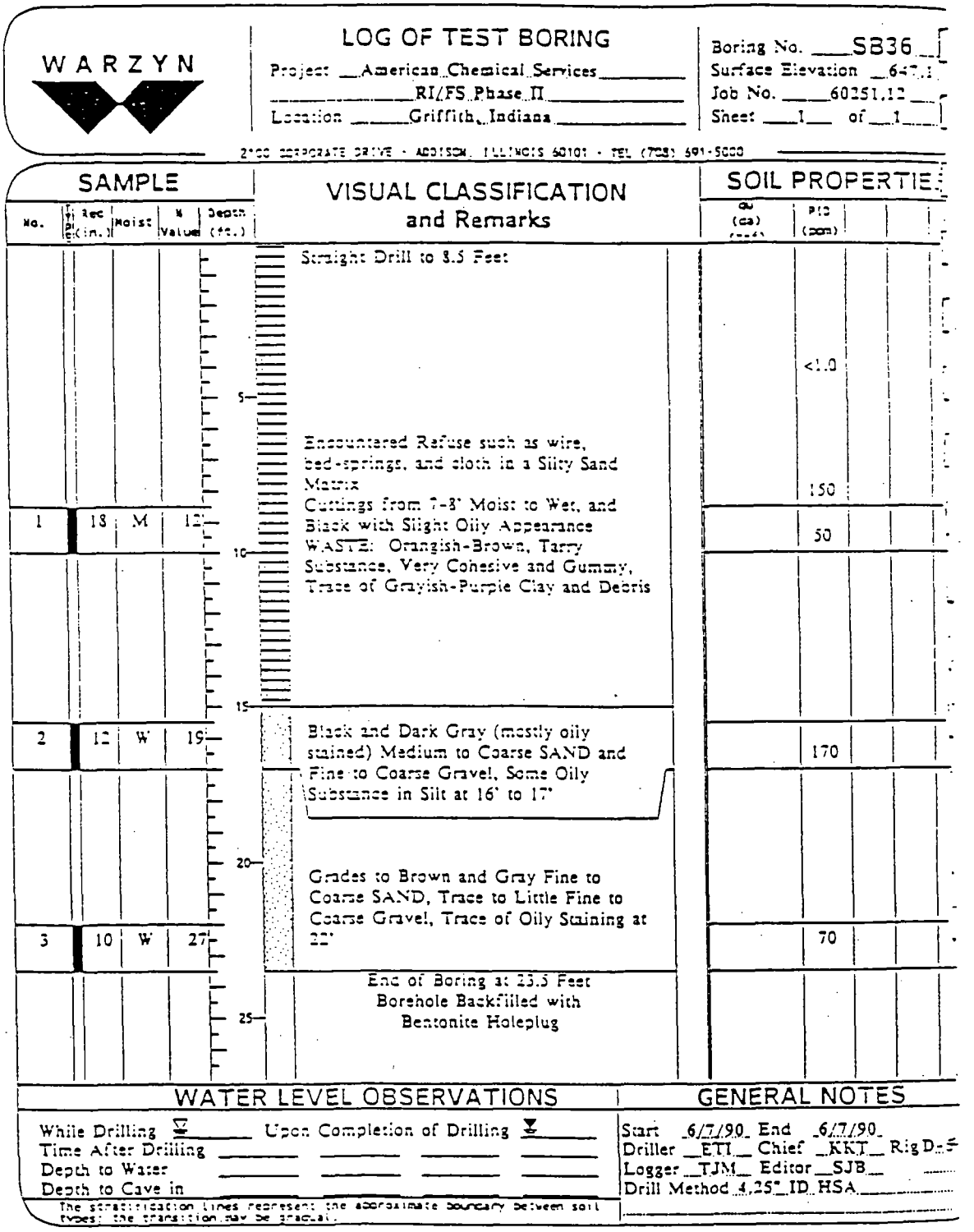
While Drilling ▽ Upon Completion of Drilling ▼
Time After Drilling
Depth to Water
Depth to Cave in

The stratification lines represent the approximate boundary between soil types: the transition may be gradual.

GENERAL NOTES

Start 6/8/90 End 6/8/90
Driller ETI Chief KKT Rig D-50
Logger TJM Editor SJB
Drill Method 4.25" ID HSA

Soil Boring Log Sheet Relevant to Test Grid #2



WARZYN

LOG OF TEST BORING

Project: American Chemical Services
RI/FS Phase II
Location: Griffith, Indiana

Boring No. SB36
Surface Elevation 647.1
Job No. 60251.12
Sheet 1 of 1

2400 SERVICE DRIVE · ADDISON, ILLINOIS 60101 · TEL (708) 691-5000

SAMPLE No.	Rec (in.)	Moist	N Value	Depth (ft.)	VISUAL CLASSIFICATION and Remarks	OVA (ca) (ppm)	PID (ppm)
					Straight Drill to 8.5 Feet		
				5			<1.0
					Encountered Refuse such as wire, bed-springs, and cloth in a Silty Sand Matrix		150
1	18	M	12		Cuttings from 7-8' Moist to Wet, and Black with Slight Oily Appearance		50
				10	WASTE: Orangish-Brown, Tarry Substance, Very Cohesive and Gummy, Trace of Grayish-Purple Clay and Debris		
				15			
2	12	W	19		Black and Dark Gray (mostly oily stained) Medium to Coarse SAND and Fine to Coarse Gravel, Some Oily Substance in Silt at 16' to 17'		170
				20	Grades to Brown and Gray Fine to Coarse SAND, Trace to Little Fine to Coarse Gravel, Trace of Oily Staining at 22'		
3	10	W	27				70
				25	End of Boring at 23.5 Feet Borehole Backfilled with Bentonite Holeplug		

WATER LEVEL OBSERVATIONS

While Drilling ▽ Upon Completion of Drilling ▼
Time After Drilling
Depth to Water
Depth to Cave in

The stratification lines represent the approximate boundary between soil types; the transition may be gradual.

GENERAL NOTES

Start 6/7/90 End 6/7/90
Driller ETI Chief KKT Rig Dat
Logger TJM Editor SJB
Drill Method 4.25" ID HSA

Soil Boring Log Sheet Relevant to Test Grid #3

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LOG OF TEST BORING

Project: American Chemical Services — Phase I RI/FS
Location: Griffith, Indiana

Boring No. SB-6
Surface Elevation
Job No. 60251.03
Sheet 1 of 1

2300 CORPORATE DRIVE ADDISON, IL 60101 TEL:(312) 691-5000

SAMPLE No.	Rec (in.)	Moist	N	Depth	VISUAL CLASSIFICATION and Remarks	SOIL PROPERTIES HNU
					FILL: Black to Brown Fine to Medium Sand	
1	18	D	26			3.0
2	12	D/M	78	5	Spoon refusal at 5.5', sample reveals Brown Fine to Medium Sand to 5', then distinctive color change to black at 5.7', strong solvent/glue-like odor, moist	210.0
3	2	W	100/5"		Poor Recovery, strong solvent odor with black heavy oily sheen in water, trace of solid paint pigment and cardboard waste to 11.5'	225.0
				10	Black wet silty sandy fill with trace of paint, oil, foam, wood from 11.5' to 15'	
4	22	W	44			150.0
					Brown-Gray Silty Fine to Coarse SAND, Trace to Little Fine to Coarse Gravel	
5	10	W	23	15		80.0
					End Boring at 15'	
				20		

WATER LEVEL OBSERVATIONS

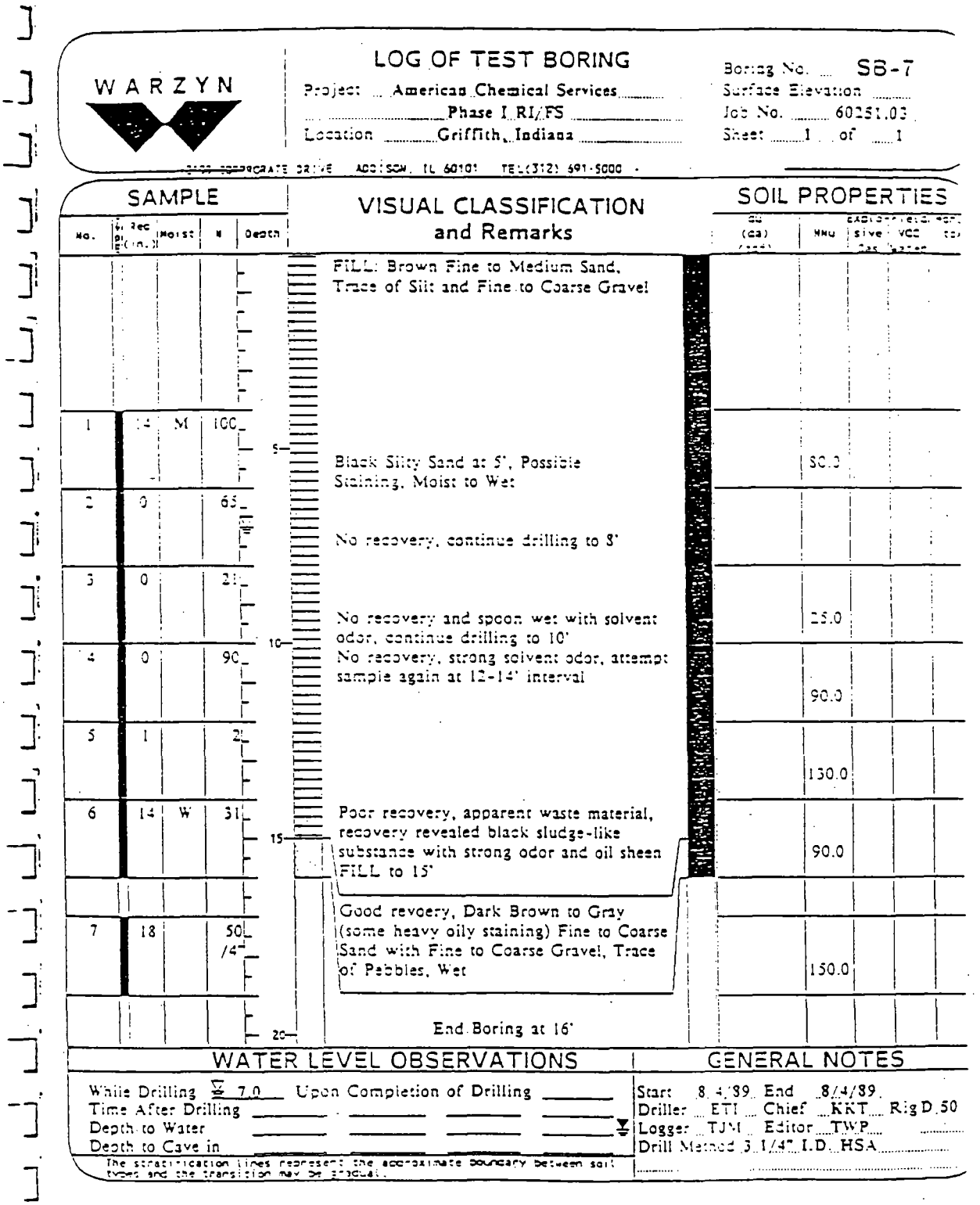
While Drilling ▽ 6.0 Upon Completion of Drilling
Time After Drilling
Depth to Water ▽
Depth to Cave in

The stratification lines represent the approximate boundary between soil types and the transition may be gradual.

GENERAL NOTES

Start 8/3/89 End 8/3/89
Driller ETI Chief KKT Rig D 5
Logger TJM Editor TWP
Drill Method 3 1/4" I.D. HSA

Soil Boring Log Sheet Relevant to Test Grid #4



WARZYN

LOG OF TEST BORING

Project: American Chemical Services
Phase I RI/FS
Location: Griffith, Indiana

Boring No. SB-7
Surface Elevation
Job No. 60251.03
Sheet 1 of 1

[illegible] CORPORATE DRIVE ADDISON, IL 60101 TEL(312) 691-5000

SAMPLE No.	Rec (in.)	Moist	N	Depth	VISUAL CLASSIFICATION and Remarks	SOIL PROPERTIES qu (qa) (ppm)	HNu	Explosive Gas	VOC	Hor. to water
					FILL: Brown Fine to Medium Sand, Trace of Silt and Fine to Coarse Gravel					
1	14	M	100	5	Black Silty Sand at 5', Possible Staining, Moist to Wet		80.0			
2	0		65		No recovery, continue drilling to 8'					
3	0		21	10	No recovery and spoon wet with solvent odor, continue drilling to 10'		25.0			
4	0		90		No recovery, strong solvent odor, attempt sample again at 12-14' interval		90.0			
5	1		2				130.0			
6	14	W	31	15	Poor recovery, apparent waste material, recovery revealed black sludge-like substance with strong odor and oil sheen FILL to 15'		90.0			
7	18		50/4"		Good revoery, Dark Brown to Gray (some heavy oily staining) Fine to Coarse Sand with Fine to Coarse Gravel, Trace of Pebbles, Wet		150.0			
				20	End Boring at 16'					

WATER LEVEL OBSERVATIONS

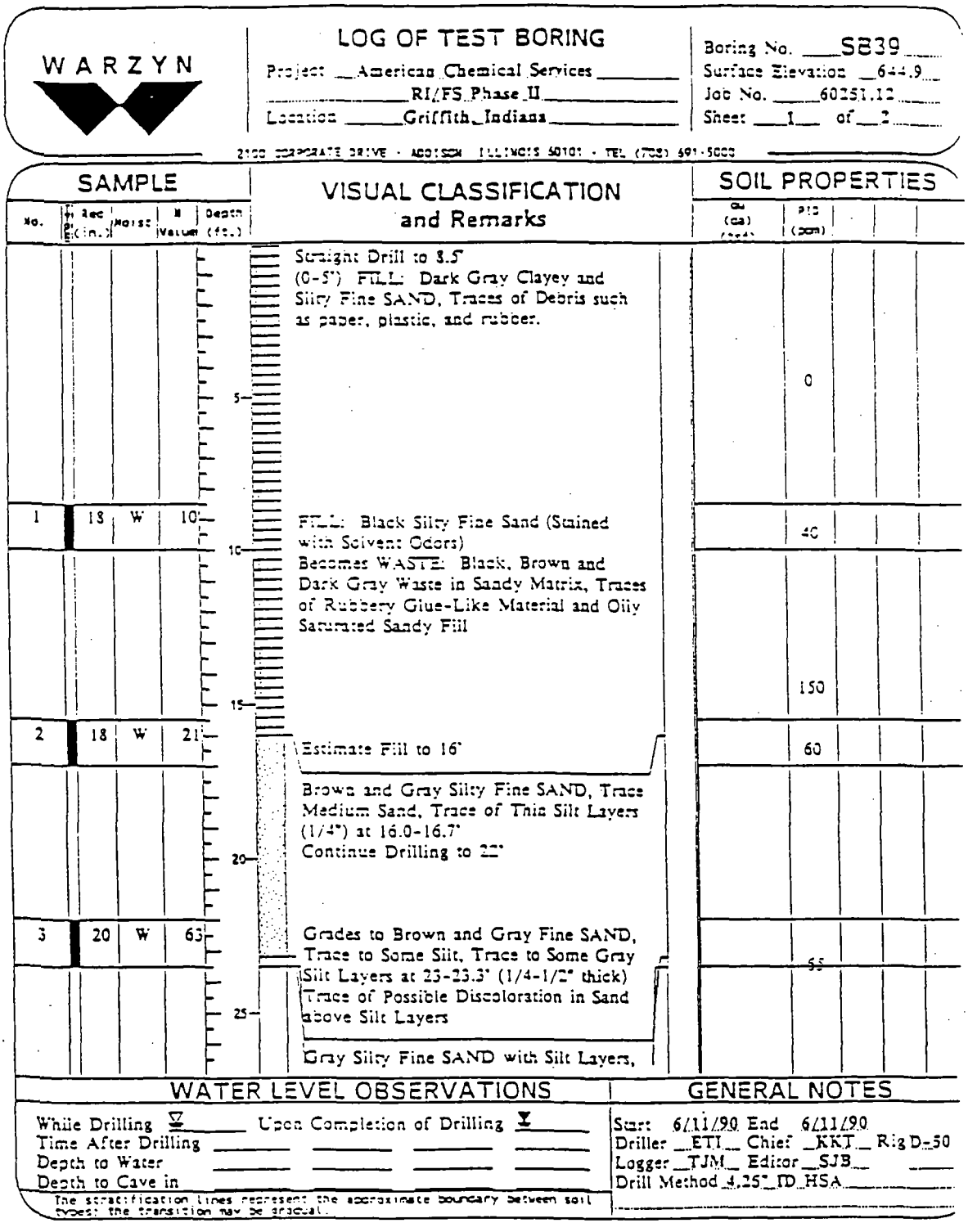
While Drilling ▽ 7.0 Upon Completion of Drilling
Time After Drilling
Depth to Water
Depth to Cave in

The stratification lines represent the approximate boundary between soil types and the transition may be gradual.

GENERAL NOTES

Start 8/4/89 End 8/4/89
Driller ETI Chief KKT Rig D 50
Logger TJM Editor TWP
Drill Method 3 1/4" I.D. HSA

Soil Boring Log Sheet Relevant to Test Grid #5



WARZYN

LOG OF TEST BORING

Project: American Chemical Services
RI/FS Phase II
Location: Griffith, Indiana

Boring No. SB39
Surface Elevation 644.9
Job No. 60251.12
Sheet 1 of 2

2100 CORPORATE DRIVE · ADDISON, ILLINOIS 60101 · TEL (708) 691-5000

SAMPLE No.	Rec (in.)	Moist	N Value	Depth (ft.)	VISUAL CLASSIFICATION and Remarks	SOIL PROPERTIES Qu (ea)	PID (ppm)
					Straight Drill to 8.5' (0-5') FILL: Dark Gray Clayey and Silty Fine SAND, Traces of Debris such as paper, plastic, and rubber.		0
1	18	W	10	10	FILL: Black Silty Fine Sand (Stained with Solvent Odors) Becomes WASTE: Black, Brown and Dark Gray Waste in Sandy Matrix, Traces of Rubbery Glue-Like Material and Oily Saturated Sandy Fill		40
				15			150
2	18	W	21		Estimate Fill to 16'		60
				20	Brown and Gray Silty Fine SAND, Trace Medium Sand, Trace of Thin Silt Layers (1/4") at 16.0-16.7' Continue Drilling to 22'		
3	20	W	63		Grades to Brown and Gray Fine SAND, Trace to Some Silt, Trace to Some Gray Silt Layers at 23-23.3' (1/4-1/2" thick) Trace of Possible Discoloration in Sand above Silt Layers		55
				25	Gray Silty Fine SAND with Silt Layers,		

WATER LEVEL OBSERVATIONS

While Drilling ▽ Upon Completion of Drilling ▼
Time After Drilling
Depth to Water
Depth to Cave in

The stratification lines represent the approximate boundary between soil types: the transition may be gradual.

GENERAL NOTES

Start 6/11/90 End 6/11/90
Driller ETI Chief KKT Rig D-50
Logger TJM Editor SJB
Drill Method 4.25" ID HSA

KEY TO BORING LOGS

Sample Type

Unsampled interval

Auger cuttings sample

Drilled by hollow stem augers; not sampled; logged by cuttings

Hand sample from surface

4" outside diameter core barrel sampler

Drilled by rotary wash bore; not sampled; logged by cuttings

5' continuous sampler

3" outside diameter split spoon sampler

3" shelby tube

2" outside diameter split spoon sampler

Lithologic Description

Unknown lithology

Lean clay

Silty clay or clayey silt

Sandy clay

Concrete

Construction debris-laden fill material

Gravel

Clayey gravel

Silty gravel

Sandy gravel

Limestone or dolomitic limestone

Silt

Gravelly silt

Silty sand

Peat

Sand

Gravelly sand

Sandy silt

Topsoil

Borehole Material Graphics

Concrete around flush-mounted protective casing

Concrete surface seal around well casing

Bentonite slurry or cement-bentonite grout around well casing

Bentonite pellet seal around well casing

Bentonite pellet seal around well screen

Sand backfill around well casing

Sand filter pack around well screen

Sand backfill or natural soil collapse in borehole

Concrete surface seal over borehole

Bentonite slurry or bentonite-cement grout backfill in borehole

Cuttings backfill in borehole

Bentonite seal in borehole

WARZYN

ATTACHMENT B

Attachment B. Auger Probe Data Relevant to PMHS Test Pit Selection

Test Pit ID	AP Numbers Identified With Test Pits	Auger Probe Depths	Visual Descriptions of Soils Observed during Auger Probes	Max HNu (ppm)	Minimum Excavation Depth for Test Pits During PMHS
1	AP8, close proximity	0-10 ft	Brown to gray sand with solvent odor	70	10 ft
2	AP12, close proximity	0-7 ft	Sand fill	20	10 ft
		7-10 ft	Black tar-like waste	70	
3	AP16, close proximity	0-8 ft	Brown to dark gray sand fill	60	12 ft
		8-10 ft	Sandy fill with landfill refuse and drum lid band	30	
4	AP18, moderate proximity	0-4 ft	Sandy fill with landfill refuse	5	14 ft
		4-6 ft	Dark gray sand	70	
		6-10 ft	Dark gray sand - trace of landfill refuse with oily stain	80	
5	AP71, moderate proximity	0-1 ft	Brown and black silty fine sand fill	NM	10 ft
		1-12 ft	Black silty fine sand fill with debris	1	
		12-13.5 ft	Black to dark gray fine sand	0	

Note:

Data was obtained from Appendix G "Auger Probe Descriptions", Remedial Investigation Report ACS NPL Site, Griffith, Indiana. June 1991.

APPENDIX D

LOW TEMPERATURE THERMAL TREATMENT TREATABILITY STUDY ON BURIED WASTE IN THE OFF-SITE CONTAINMENT AREA

TESTING PROCEDURES

APPENDIX D

LOW TEMPERATURE THERMAL TREATMENT TREATABILITY STUDY ON BURIED WASTE IN THE OFF-SITE CONTAINMENT AREA

TESTING PROCEDURES

APPENDIX D - FIELD SAMPLING PLAN

THERMAL TREATABILITY STUDY
AMERICAN CHEMICAL SERVICE, INC.

SUBMITTED TO:

MONTGOMERY WATSON
4525 SO. WASATCH BLVD.
SUITE 200
SALT LAKE CITY, UTAH

December 6, 1996
Focus Project No. 119406

PREPARED BY:

FOCUS ENVIRONMENTAL, INC.
9050 EXECUTIVE PARK DRIVE
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KNOXVILLE, TENNESSEE 37923

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D.1.0 INTRODUCTION

D.1.1 OVERVIEW

The U.S. EPA has determined, as stated in the Record of Decision (ROD) issued in September 1992, that thermal desorption is the remedial technology of choice for the Off-Site Containment Area of the American Chemical Service, Inc. (ACS) Site. The ACS facility, located in Griffith Indiana, began operation in May, 1955 as a solvent recovery facility. In September, 1990, ACS ceased hazardous waste operations and therefore relinquished its Resource Conservation and Recovery Act (RCRA) interim status. This thermal treatability study will investigate the feasibility of implementing the thermal desorption component of the ROD. The workplan provides guidance for obtaining the data necessary to evaluate the remediation of the Site by thermal desorption technology.

D.1.2 OBJECTIVES

The overall objectives of this project plan are to determine if thermal desorption will meet the clean-up levels for the ACS site and collect data useful for evaluating the implementation of the chosen technology. Specific objectives are as follows:

- Determine the VOC reductions during feed preparation operations.
- Determine the concentrations of organic contaminants in the "prepared" treatability test samples.
- Prepare two composite soil samples (a "typical" and a "worst-case" condition) for thermal desorption testing based on analyses of the "prepared" treatability test samples.
- Determine the soil treatment temperature required to meet the cleanup objectives.
- Determine the total metals balance in the feed and treated soils.
- Determine the effect of thermal treatment on the leachability of metals in the soil.
- Characterize the off-gas from the thermally treated soil.
- Determine soil geotechnical properties to provide data useful in assessing material handling requirements.
- Determine if dioxin/furans are present in the starting soils, and if so, determine concentration reductions resulting from thermal treatment.
- Determine if dioxin/furans exist in the offgas for design purposes by analyzing the impinger water.
- Determine approximate partitioning of carbon and chloride to the soil and offgas.
- Determine approximate partitioning of carbon to the various components of the offgas (i.e., carbon monoxide, carbon dioxide, and light hydrocarbons).
- Conduct water and ash mass balances to determine test closure.

- Evaluate (if necessary) the effect of treatment conditions on small pieces of debris (wood, rubber, paper, plastic, etc.)

D.1.3 PERFORMANCE STANDARDS

The performance standards, as defined in the Record of Decision for the Site, are listed in Table D.1-1. One of the primary goals of this treatability study is to determine the correct thermal treatment conditions to meet these performance standards for Site soils from the Off-Site Containment Area.

D.1.4 PROJECT ORGANIZATION AND RESPONSIBILITY

Focus Environmental will manage and direct the treatability study for Montgomery Watson. Focus expects to subcontract the actual treatability laboratory work to IT Corporation's Technology Development Laboratory (TDL) located in Knoxville, Tennessee. The TDL would be responsible for performing all of the treatabilty tests as defined in this plan, with Focus providing project management, oversight, data evaluation and reporting. TDL responsibilities include sample receipt, sample preparation, tray testing, RTA testing, treated materials sampling and shipment, data collection and transfer to Focus, and residual disposal. The contaminant analytical work will be subcontracted to IEA Laboratories in Cary, North Carolina. The samples for chemical properties will be sent to Galbraith Laboratories in Knoxville, Tennessee. The samples for geotechnical analyses will be sent to IT Corporation, also located in Knoxville, Tennessee. After the treatability work has been concluded, Focus will prepare a Treatabilty Test Report, documenting the results of this study.

D.2.0 STUDY DESIGN

D.2.1 GENERAL APPROACH

The thermal treatability study will be performed at International Technology Corporation's (IT) Technology Development Laboratory (TDL) located in Knoxville, TN. Treatability testing for this project will be conducted using a tiered approach. The first test tier (laboratory-scale tray tests) includes a rough estimate of VOC fugitive emissions due to material handling operations as described in Section D.3.1.2. It also utilizes relatively simple and inexpensive tray tests to define soil treatment temperature operating conditions required to meet site cleanup levels. Tray tests generally require approximately 100 grams of material per test. Information from the tray testing will be used for establishing test conditions for the more sophisticated Tier 2 tests.

Tier 2 tests will consist of a limited number of bench-scale tests using a rotary thermal apparatus (RTA) which allows processing of a larger quantity of soil at conditions selected based on the results of the tray testing. The RTA tests will be used to (1) confirm treatment conditions required to meet soil cleanup levels; (2) gather information to perform a material balance on other parameters of concern (metals, carbon, sulfur, chlorine, etc.); and, (3) gather data on emissions from the soil during thermal treatment. RTA testing requires approximately 1,000 grams of material per test condition.

The results from the thermal treatability testing can be used to evaluate the applicability of thermal desorption at the ACS site. Numerous samples will be collected during the testing program as defined in Section D.4.0.

D.2.2 EQUIPMENT DESCRIPTION

D.2.2.1 Tray Tests

The experimental apparatus to be used for this test program consists of a Lindberg Model 51848 Muffle Furnace with an electronic temperature controller and a 1,600-watt heater. The oven has a double-shell construction with interior surfaces made of Moldatherm, a moldable aluminum-silicate insulation material. The oven is capable of operating at temperatures up to 2,012°F and has a relatively fast heat-up rate due to its low mass. The interior space is approximately 3.9-inches wide by 4.3-inches by 8.3-inches deep. A loose block of Moldatherm (approximately 1/2-inch thick) will be placed on the bottom of the oven to provide additional separation between the tray and the hot interior surface of the oven. This minimizes the potential for localized hot spots in the soil.

A custom fabricated tray will be used to contain the soil sample within the oven. The tray which weighs approximately 430 grams is 3.5-inches wide by 1.3-inches high by 7.6-inches long, and is constructed of incoloy (i.e., nickel alloy) to resist oxidation during high temperature testing. Figure D.2-1 provides the general configuration for the tray tests.

During the tests, the oven will be continuously purged with air, introduced through a 3/8-inch diameter incoloy tube connected to the back wall of the oven. The purge gas is directed against the back wall to promote even heating and gas distribution. The purge gas flow rate will be measured with a standard rotameter and maintained at approximately 100 cc/min during the test. This flow rate provides adequate purging of the oven, but does not affect the temperature control or heat-up rate of the oven. The purpose of this purge is to minimize the build-up of volatilized organic compounds in the oven. If these organics were not purged, a combustible mixture of gases could accumulate in the oven. The purge gas rotameter is calibrated quarterly using a wet test meter.

Two thermocouples will be used in the static tray test apparatus to measure temperature. One is a National Institute of Standards and Technology (NIST) traceable, Type K sheathed thermocouple placed approximately 3 centimeters above the test material at the center of the oven. This thermocouple will be used to measure the "Test Temperature" (i.e., oven gas temperature). The second thermocouple, also a NIST traceable, Type K sheathed thermocouple, is used to measure the temperature within the test matrix (i.e., soil). All thermocouples are calibrated every quarter using a standard reference source.

Temperatures will be recorded using a multipoint recorder and logged from the recorder into a computer data collection program. The 30 recorder channels are sampled every 4.5 seconds. All of these points are sent to the computer data logging program. The data from the computer will be transferred to a spreadsheet to generate graphical illustrations of the experimental test temperatures with respect to time.

D.2.2.2 Rotary Thermal Apparatus (RTA)

Figure D.2-2 provides a schematic of the RTA test equipment. The RTA will be operated in a batch mode. The rotating cylinder portion of the system is 5 inches in diameter by 11.5 inches long and is fabricated of Incoloy to resist oxidation and/or chemical reactions during high temperature tests. The RTA cylinder will rotate at 5 revolutions per minute and will be externally heated with a 7,000 watt Lindberg furnace. The chain driven tube is suspended on both ends by brass rollers and sealed by graphite packing rings at the inlet and outlet distribution boxes. The RTA cylinder will be purged with air during the test. One test will be conducted on the "worst case" matrix using nitrogen as the purge gas. This will provide a comparison between oxygenated and non-oxygenated environments for the potential to form dioxins and furans.

The main goals of the RTA tests are to verify the ability for the selected soil samples to meet the cleanup goals and characterize the off-gas from the thermally treated soil. The process gas flow rates will be field established to maintain a slight negative pressure in the RTA cylinder. This will ensure that all materials desorbed from the thermally treated soil will be directed toward the off-gas treatment system. The temperature of the soil bed and the temperature of the gas in the RTA cylinder will be measured with thermocouples. A sampling system will extract a small gas stream from the RTA cylinder through a water impinger and into an oxygen analyzer.

Figure D.2-3 shows a general flow diagram of the RTA system including the off-gas handling and monitoring system. The off-gas (purge gas and vaporized contaminants) from the RTA cylinder will exit through a three foot long quartz tube into a heated filter to remove particulates in the off-gas carried over from the RTA cylinder. The filter will be a 0.5 micron glass fiber filter. The filtered off-gas will pass through four glass impingers (in series) placed in an ice bath. The first three impingers will be partially filled with 400 grams of deionized water. The fourth impinger will be empty, acting as a "knockout pot" for the system.

Grab samples will be taken from the impinger offgas at 5 different soil treatment temperatures. These grab samples will be volumetrically syringed into a Tedlar bag and then analyzed by gas chromatography for the following compounds; hydrogen, oxygen, nitrogen, carbon monoxide, carbon dioxide, methane, ethane, acetylene, and propane/propanol. Concentrations of these compounds will be determined as a function of soil temperature.

The impinger offgas will be split into three separate streams to determine 1) THC/CO, 2) VOC, and 3) SVOC/PCB concentrations in the offgas. One stream will be analyzed continuously for THC and CO. A second stream (for VOC analysis) will pass through two impingers, one containing 400 grams of pure methanol and the second containing 400 grams of deionized water. The off-gas will then pass through two Tenax resin tubes (one primary tube and one backup). The third stream (for SVOC and PCB analyses) will pass through two XAD resin traps (one primary trap and one backup). The flow rates and/or volumes will be measured for stream during each test. It is assumed that if dioxin/furan's are determined to be present in the impinger water then some may be present in the XAD resin as well. The only purpose of analyzing for dioxin/furan's in the impinger water is to determine general fate of dioxin/furan's if present, therefore, dioxin/furan analysis of the XAD resin will not be conducted.

The driving force for gas flow will be provided by a dry gas meter/pump. The purge gas, seal gas, and system off-gas flow rates will be monitored using gas rotameters. Table D.2-1 provides a summary of the instrumentation and monitoring points for the RTA system.

D.3.0 STUDY EXECUTION

D.3.1 FEED SOIL PREPARATION

Selection of representative soil feed samples for testing is a critical component of the overall program. Representative samples are required for generating test results to evaluate the effectiveness of the thermal process and can aid in the evaluation of scale-up issues. There are five primary waste characteristics that are important for evaluating the effectiveness of a thermal process, including:

- Clay and silt content and types
- Contaminant types and concentrations
- Organic content
- Moisture content
- Debris content and type.

Soil samples will be collected during the screening study per the Pretreatment/Material Handling Study Plan (Appendix C to the Treatability Test QAPP), and shipped to the thermal treatability test laboratory. The samples received from the field will be sampled and analyzed to determine "as received" VOC contaminant concentrations. The as received samples will be dried and screened to generate "prepared samples" for analyses. Based on the results of the "prepared samples", two composite feed soil samples will be generated by blending the prepared samples to obtain desired concentrations. An optional third sample, consisting of soil and debris, may be tested if soils with significant quantities (approximately 1 % or greater) of small (less than 2 inch) organic debris are discovered during the Pretreatment/Material Handling Study. As long as organic debris can be screened from the soil, there is no concern for the impact to a thermal treatment system. The main purpose for collecting a debris sample for further testing would be to determine potential loadings to the air pollution control system associated with the small organic debris (less than 2 inches) that could enter the thermal treatment system. Field personnel will visually determine the need for additional sampling for debris testing purposes as defined in Appendix C.

The two composite feed soil samples will be developed to represent worst-case and typical conditions. The worst-case conditions will be defined as the 99% confidence interval around the average for the key contaminants of concern (VOCs, SVOCs, and PCBs) determined from past remedial investigation analyses of the Off-Site Containment Area. Typical conditions will be defined as the average of key contaminants of concern. If typical conditions for site soils cannot be obtained from the "prepared samples", only one sample matrix will be generated as close to worst case conditions as possible.

D. 3.1.1 Sample Receipt

The samples will be shipped to the TDL in 5-gallon buckets, as specified in the Pretreatment/Material Handling Plan. These buckets will be stored and sampled in a walk-in cooler at a temperature of 4°C to minimize the potential for loss of volatile organics. The field samplers will fill the buckets to the "rim", minimizing the sample headspace, and seal the buckets with the air-tight gasketed lids.

Each of the five samples received from the field (one from each test pit) will be sampled and analyzed for VOCs prior to pretreatment operations. The objective of this analysis is to determine an initial concentration of VOCs so that additional estimates of fugitive emissions due to VOC losses during material handling operations can be developed. These results will augment fugitive emission data collected during the PMHS. Three 50-gram, grab aliquots (one from the top third, one from the middle third, and one from the bottom third of the bucket) will be taken from each field sample. These aliquots will be blended gently for each individual field sample and then placed into sample VOA vials for analysis as defined in Section D.4.0.

A duplicate, a matrix spike, and a matrix spike duplicate sample will be taken for one of the field samples. All sampling activities will occur inside the walk-in cooler. The samples will be shipped at 39.2°F (4°C) to the analytical laboratory.

D.3.1.2 Prescreening and Conditioning

After the "as received" VOC samples have been taken, each contaminated field soil sample will then be individually blended with a spade, trowel, or other mechanical means to obtain a relatively homogeneous mixture. The blended soil will be spread out to dry in a uniform layer on a tray in a ventilated hood. When the soil is no longer cohesive, the dried soil will be broken up and passed through a 1/4" screen. Any debris (rocks, etc.) that does not pass through the screen will be removed, weighed and documented (type of debris and approximate weight percent of debris).

The prepared soils will be sampled for VOC analysis to determine VOC losses during sample preparation. A total of five grab samples (approximately 50 grams per aliquot) will be collected from each soil sample; one from each corner and one from the middle of the tray. These grabs will be blended and packaged in the appropriate containers for VOC, SVOC, and PCB analyses as defined in Section D.4.0. The samples will be shipped at 39.2°F (4°C) to the analytical laboratory.

D.3.1.3 Compositing and Blending

Two soil composite samples that represent worst-case and typical conditions will be generated from the prepared samples. These two conditions will be determined by utilizing data from past remedial investigations of the Off-Site Containment Area. The worst-case condition will be defined as the 99% confidence interval around the mean for the key contaminants of concern (VOC, SVOC, and PCB). This means that for any sample taken in the Off-Site Containment Area, there is a 99 percent probability that the contaminant concentrations will be less than the 99 percent confidence interval concentration. The typical condition will be defined as the average of key contaminants of concern.

Two composite feed soil samples will be generated by blending portions of the five dried and screened field samples. The mass of the portions for blending will be determined by reviewing the prepared soil analytical results and estimating the blending portions to generate the desired contaminant concentrations. The two composite feed soil samples will weigh approximately 10 kilograms each. Each will be placed into a 5-gallon bucket, sealed, and then placed on a drum roller for 1-hour to ensure sample homogeneity.

D.3.1.4 Sample Characterization

Samples from the two composite feed soil matrices will be analyzed on a normal turnaround time as defined in Section D.4.0. The results of these analyses will provide the baseline soil characterization for comparison with the treated soil analyses from the tray and RTA tests to determine the effectiveness of the thermal desorption technology.

D.3.2 TRAY TESTING

D.3.2.1 Test Conditions

The two composite feed soil matrices will be fully tested using the same protocols and analyzed for the same parameters. Aliquots from each soil matrix will be tested at four separate soil temperatures (500, 700, 900, and 1,100°F). The soil treatment temperatures were selected to bracket operating temperatures that are typically used in commercially available thermal desorption systems.

The 500°F temperature represents a "failure condition" at which thermal desorption is not expected to be successful in meeting the soil treatment objectives. The 700°F through 1,100°F temperatures represent the range of normal thermal desorber soil discharge temperatures. The soil aliquots will require approximately 18 minutes heat-up time (ramp time). The hold time at the soil treatment temperature will

be held constant at 0 minutes during all tray tests. These residence times are also representative of process operating conditions in full-scale thermal desorption equipment.

The optional third soil matrix containing a mixture of debris and soil may be processed at one set of tray test conditions (700°F and 0 minutes hold time at temperature). However, only qualitative observations will be made for the residue remaining after each test with specific attention placed on the condition of the debris. If the organic debris appears to have charred or is offgassing, a RTA test will be conducted using a debris laden aliquot of soil to determine the impact of the debris on the offgas from the thermally treated soil.

D.3.2.2 Test Protocol

The tray test protocol for this study is as follows:

- The oven will be baked at its maximum temperature for 1-hour prior to the tray tests. The oven will then be turned off and allowed to cool to ambient temperature.
- A representative aliquot (approximately 100 grams) of the feed soil will be transferred to the tray using a clean, stainless steel spatula.
- The soil will be spread evenly in the tray to achieve a uniform layer in the bottom of the tray, (approximately 1/4-inch depth for a 100-gram aliquot of soil).
- The purge gas flow will be turned on and the tray with the soil placed in the oven at ambient temperature.
- The oven door will be closed and the temperature controller will be set to the target test temperature and the timer started.
- The temperature and time will be monitored and recorded periodically throughout the test period and any pertinent observations will be recorded.
- When the prescribed soil treatment temperature is reached, the oven heater will be shut off, the oven door opened to remove the sample and allow the oven to cool. The cooled treated soil will be submitted for analysis. The trays will be washed between test runs.

D.3.2.3 Sampling and Analysis

The treated soil samples will be sent to the analytical laboratory for contaminant analysis as defined in Section D.4.0. Results of the tray tests will be used to select the optimum soil treatment temperatures for each soil matrix and to establish operating conditions for conducting the RTA testing.

D.3.3 ROTARY THERMAL APPARATUS (RTA) TESTING

D.3.3.1 Test Conditions

One RTA test will be conducted per soil matrix using an air purge. A second RTA test will be conducted on the "worst case" soil matrix using a nitrogen purge. One RTA test run may also be conducted for the soil matrix containing the debris (air purge). The same analyses and offgas monitoring will be conducted on each RTA test. The treatment conditions for these tests will be established based on the tray test results.

D.3.3.2 RTA Test Protocol

The protocol for the RTA tests is as follows:

- The weight of the test soil in the RTA cylinder, water in each impinger, and methanol in the VOC train impinger will be recorded prior to test initiation. The impinger that filters the gas sample extracted from the RTA tube for oxygen analysis will be filled with deionized water at the beginning of the test and the weight of water recorded.
- The RTA test equipment will be configured as shown in Figure D.2-3.
- The gas flows (purge gas, seal blankets, and exhaust) will be adjusted to provide a slight negative pressure in the purge gas inlet distribution boxes. The furnace settings will be adjusted to ramp as fast as possible to the target soil temperature (approximately 18 - 22 minutes). The furnace will be raised to enclose the RTA cylinder and the test will be initiated.
- Bag samples will be taken for analysis at 5 different temperatures (to be determined at the time of the test).
- When the planned soil temperature has been reached, the furnace will be lowered away from the RTA cylinder and a water spray will be used to indirectly cool the cylinder and its contents as quickly as possible.
- The RTA cylinder will be disassembled, and the treated soil will be removed, weighed and the weight recorded. The treated soil will be sampled and analyzed as defined in Section 4.0.
- The heated filter will be disassembled and the inlet and outlet glass connections will be washed with methanol to recover any condensed materials. The rinse will be collected and allowed to dry in a hood. The filter and filter catch will be combined with the dried residue from the rinse and weighed to provide an estimate of the carryover.
- The water impinger train, including the impinger on the oxygen analyzer, will be disassembled. The weight of the water remaining in each impinger will be measured and recorded. Any discoloration or turbidity in each of the impingers will be noted. All glass connections will be rinsed with deionized water. The rinse will be collected and combined with the water from the impingers, including the oxygen analyzer impinger to form one water sample. The weight of this

combined sample will be measured and recorded. A sample of the impinger water will be collected for analyses. If multiple layers of organics are visible in the water sample, aliquots will be taken for chemical/physical analyses, and the remainder of the sample will be extracted using hexane. The hexane layer will be separated and weighed. Samples of the hexane extract will then be submitted for contaminant analyses as defined in Section 4.0.

- The methanol impinger will be disassembled and the weight of the methanol will be measured and recorded. Any discoloration or turbidity will be noted. All glass connections will be rinsed with pure methanol. The rinse will be collected and combined to form one sample. The weight of this sample will be measured and recorded. The sample will be submitted for analyses as defined in Section D.4.0.
- The Tenax resin and the XAD-2 resin will be removed, the ends will be sealed with Teflon tape, and the tubes will be packaged in a Ziploc bag and sent to the laboratory for analyses.

This protocol will be repeated until all RTA tests have been conducted for all test soil matrices.

D.3.3.3 Sampling and Analysis

All samples will be packaged and shipped, as outlined in Section D.4.0, to the appropriate laboratories. The results from these analyses will be compared to the results from the starting test soil to evaluate the effectiveness of the technology at the chosen treatment conditions.

D.3.4 Residuals Management

All unused samples will be archived at the treatability laboratory (IT TDL) until notified otherwise by Focus. Upon notification by Focus, all samples and sample residuals will either be returned to the Site or they will be disposed of by the IT TDL at a permitted RCRA incineration facility in accordance with federal, state, and local regulations.

D.4.0 SAMPLING AND ANALYSIS

D.4.1 SAMPLE NOMENCLATURE

Table D.4-1 provides the sample name and a description for every sample that will be taken for the thermal treatability study. This list includes all QA/QC samples such as duplicates, matrix spikes, and matrix spike duplicates.

D.4.2 SAMPLE ANALYSIS

All sample analyses for each individual sample are listed in Tables D.4-2 through D.4-9. The tables specify to which laboratory the samples will be submitted, by what analytical methods each sample will be tested, the sample matrix, the data quality level of the method, and any QA/QC associated with the sample. Table D.4-10 provides sample quantities needed for the specific methods and preservation/shipping instructions.

D.4.3 SAMPLE CUSTODY

All sample movements between the treatability laboratory (IT TDL) and operating groups will be documented on a standard IT Request for Analysis/Chain of Custody Record. A copy of all Request for Analysis/Chain of Custody Records will be maintained in the project file. Sample preservation will follow the specified analytical method guidelines. All sample shipments will be packaged appropriately, according to Department of Transportation (DOT) regulations.

D.5.0 DATA MANAGEMENT

D.5.1 DOCUMENTATION/RECORDING

During the study, the treatability laboratory will record all data in laboratory notebooks. The TDL utilizes the following data management procedures.

- All laboratory notebooks are uniquely numbered and permanently bound with sequentially numbered pages. Project-specific notebooks will be signed out by the facility quality control coordinator to the individuals working on the project. All daily laboratory activities associated with the project will be recorded in the project-specific notebooks.
- At the completion of the project, the project-specific laboratory notebooks and logbooks will be returned to the laboratory quality control coordinator for retention.

The treatability laboratory (TDL) will provide Focus with a copy of all lab notebook pages for the treatability tests. The lab notebook will detail all project activities, test conditions, and sample descriptions. Specifically for the tray tests, the following data will be monitored and recorded:

- Oven temperature
- Soil temperature
- Heating time of sample
- Initial soil weight
- Final soil weight
- Any visual observation of the physical characteristics of the heated test soil

The following data will be monitored and recorded during the RTA tests:

- Oven temperature
- Soil temperature
- Offgas temperature
- Offgas continuous analyses (THC, CO)
- Heating time of sample
- Initial soil weight
- Final soil weight
- Any visual observations

TDL will also provide Focus with a short letter report detailing any experimental observations and a summary of the offgas treatment system (total flow rates, offgas concentrations, mass balance data, etc.). A disk (in Lotus) containing the time/temperature profile for each tray test and RTA test will also be provided. TDL will take pictures of each sample, before and after thermal treatment as well as pictures

of the tray test equipment, RTA and the offgas collection system. Copies of any other project paperwork (chain of custodies, shipping manifests, calibration records, etc.) will also be given to Focus at the close of the treatability study.

D.5.2 DATA REDUCTION AND EVALUATION

Analytical data reduction, validation and reporting will be handled according to the requirements defined in the body of the treatability testing QAPP. This section provides general information regarding reduction and reporting of test data and analytical results. Focus personnel will be responsible for all treatabilty data reduction. Data reporting will include:

- VOC removal from soil during sample preparation
- Prepared sample results
- Composite feed sample results
- Tray test temperature vs. time plots
- Tray test analytical results, and contaminant removal efficiencies. Results will be compared with cleanup goals
- RTA test temperature vs. time plots
- RTA test analytical results - soils
- RTA test analytical results - offgas
- Mass balance for carbon, chlorides, metals and organic contaminants of concern
- Change in metal leachability in soils due to thermal treatment by evaluation of TCLP data
- Summary of Dioxin/Furan concentrations (totals and 2,3,7,8-TCDD equivalents)
- Plots of CO and THC vs. soil treatment temperature.

Prior to releasing the draft report, the treatability test data will receive a technical review. This review will consist of verifying data transcriptions, manual calculations, and reasonableness of the data.

D.5.3 EQUIPMENT AND INSTRUMENT CALIBRATIONS

The designated treatability laboratory (TDL) maintains an active Quality Assurance Program with policies outlined in Standard Operating Procedures (SOPs). The Quality Assurance Officer (QAO) will monitor all personnel, methods, equipment practices, and records with controls to ensure compliance for this project.

Specific equipment calibration requirements for the tray tests and RTA tests include:

Equipment	Required Accuracy
Laboratory Balance	± 0.1% or ± 2 least significant digits, whichever is greater

Thermocouple	±7°C at 500°C
Flow Meter	± 5.0%
Chronograph	±1 minute per day

The TDL will supply copies of all applicable equipment calibration records to Focus. This will include any calibrations necessary for the off-gas sampling system such as span gases, total flow meters, etc. Instrument calibration records will also be provided as required in Table D.5-1.

D.6.0 STUDY REPORT

The final Thermal Treatability Report will contain the following sections:

1.0 Executive Summary

2.0 Introduction
- 2.1 Background
- 2.2 Purpose and Objectives
- 2.3 Treatment Standards
- 2.4 Workplan Deviations/Modifications

3.0 Soil Sampling and Characterization
- 3.1 Sample Preparation
- 3.2 Sample Characterization

4.0 Thermal Treatability Study
- 4.1 Approach
- 4.2 Tray Testing
- 4.3 RTA Testing
- 4.4 Geotechnical Testing

5.0 Conclusions

Table 2-1. RTA Tests - Instrumentation and Monitoring Points

Monitoring Point (a)	Monitoring Point Description	Monitoring Frequency
A1	RTA Cylinder Oxygen Concentration	Continuous
A2	RTA Offgas CO Concentration	Continuous
A3	RTA Offgas THC Concentration	Continuous
F1	Purge Gas Rate, Outlet Box	Continuous
F2	Purge Gas Rate, Outlet Box	Every Minute
F3	Purge Cas Rate, Inlet Box	Every Minute
F4	Inlet to VOST Train	Every Minute
F5	Inlet to SVOC Train	Every Minute
F6	System Exhaust Flow Rate	Every Minute
F7	Inlet to CO/THC Slip Stream	Every Minute
P1	Front Seal Pressure	Every Minute
P2	Back Seal Pressure	Every Minute
S1	RTA Offgas Sampling Port	5 grabs/test
T1	RTA Cylinder Vapor Temperature	Continuous
T2	Soil Temperature	Continuous
T3	Filter Inlet Temperature	Continuous
T4	Water Impinger Inlet Temperature	Continuous
T5	Water Impinger Outlet Temperature	Continuous
V1	VOST Train Sample Volume	Continuous
V2	SVOC Train Sample Volume	Continuous

(a) - See Figure 2-3

Table 4-1. Thermal Treatment Treatability Testing Samples

Sample Number	Sample Description
"As Received" Samples (VOC Analysis Only)	
AR1	Field Sampling Area #1
AR2	Field Sampling Area #2
AR3	Field Sampling Area #3
AR4	Field Sampling Area #4
AR5	Field Sampling Area #5
AR5-D	Field Sampling Area #5, duplicate
AR5-M	Field Sampling Area #5, MS/MSD
Prepared Samples (VOC, SVOC, and PCB Analyses)	
PS1	Field Sampling Area #1
PS2	Field Sampling Area #2
PS3	Field Sampling Area #3
PS4	Field Sampling Area #4
PS5	Field Sampling Area #5
Composite Feed Samples	
SC1	"Worst Case" Condition Soil Matrix
SC1-D	"Worst Case" Condition Soil Matrix, duplicate
SC1-M	"Worst Case" Condition Soil Matrix, MS/MSD
SC2	Typical Condition Soil Matrix
Tray Tests	
TT1-500	"Worst Case" Condition, 500°F Treated Soil
TT1-700	"Worst Case" Condition, 700°F Treated Soil
TT1-900	"Worst Case" Condition, 900°F Treated Soil
TT1-1100	"Worst Case" Condition, 1100°F Treated Soil
TT2-500	Typical Condition, 500°F Treated Soil
TT2-700	Typical Condition, 700°F Treated Soil
TT2-900	Typical Condition, 900°F Treated Soil
TT2-1100	Typical Condition, 1100°F Treated Soil
RTA Tests : Soil	
RTAN	"Worst Case" Soil, N2 Purge, Treated Soil
RTA1	"Worst Case" Soil, Air Purge, Treated Soil
RTA1-D	"Worst Case" Soil, Air Purge, Treated Soil, duplicate
RTA1-M	"Worst Case" Soil, Air Purge, Treated Soil, MS/MSD
RTA2	Typical Soil, Air Purge, Treated Soil
RTA3 (a)	Optional : Treated Debris/Soil, Air Purge
RTA Tests : Offgas	
RTAN-Water	"Worst Case" Soil, N2 Purge, Impinger Water
RTAN-MeOH	"Worst Case" Soil, N2 Purge, Impinger Methanol
RTAN-XAD	"Worst Case" Soil, N2 Purge, XAD Resin
RTAN-TNX	"Worst Case" Soil, N2 Purge, Tenax Resin
RTA1-Water	"Worst Case" Soil, Air Purge, Impinger Water
RTA1-MeOH	"Worst Case" Soil, Air Purge, Impinger Methanol
RTA1-XAD	"Worst Case" Soil, Air Purge, XAD Resin
RTA1-TNX	"Worst Case" Soil, Air Purge, Tenax Resin
RTA2-Water	Typical Soil, Air Purge, Impinger Water
RTA2-MeOH	Typical Soil, Air Purge, Impinger Methanol
RTA2-XAD	Typical Soil, Air Purge, XAD Resin
RTA2-TNX	Typical Soil, Air Purge,Tenax Resin
RTA3-Water (a)	Optional : Treated Debris/Soil, Air Purge, Impinger Water
RTA3-MeOH (a)	Optional : Treated Debris/Soil, Air Purge, Impinger Methan
RTA3-XAD (a)	Optional : Treated Debris/Soil, Air Purge, XAD Resin
RTA3-TNX (a)	Optional : Treated Debris/Soil, Air Purge, TNX Resin

(a) - The optional Debris Sample will be tested if determined necessary during the Pretreatment/Material Handling Study.

Table D.4-2 Analytical Requirements for "As Received" and "Prepared" Samples

IEA Laboratory (Contaminants Analyses) - Level C Data					
	Matrix	soil	soil	soil	soil
	Sample Number :	**AR1**	**AR2**	**AR3**	**AR4**
VOCs	SW-846 Method 8260A	x	x	x	x
SVOCs	SW-846 Method 8270B				
PCBs	SW-846 Method 8081				
	Matrix	soil	soil	soil	soil
	Sample Number :	**AR5**	**AR5-D**	**AR5-M**	**PS1**
VOCs	SW-846 Method 8260A	x	x	x	x
SVOCs	SW-846 Method 8270B				x
PCBs	SW-846 Method 8081				x
	Matrix	soil	soil	soil	soil
	Sample Number :	**PS2**	**PS3**	**PS4**	**PS5**
VOCs	SW-846 Method 8260A	x	x	x	x
SVOCs	SW-846 Method 8270B	x	x	x	x
PCBs	SW-846 Method 8081	x	x	x	x

Table D.4-3 Analytical Requirements for Sample Characterization

	Sample Number :	SC1	SC1-D	SC1-M	SC2
	Matrix	soil	soil	soil	soil
IEA Laboratory (Contaminants Analyses) - Level C Data					
VOCs	SW-846 Method 8260A	x	x	x	x
SVOCs	SW-846 Method 8270B	x	x	x	x
PCBs	SW-846 Method 8081	x	x		x
Dioxins/Furans	SW-846 Method 8290	x			x
Total Metals (a)	SW-846 Methods 6010/7000	x	x		x
TCLP Metals	SW-846 Methods 1311/6010/7000	x			x
Galbraith Laboratory (Chemical Properties) - Level A Data					
Ultimate/Proximate	ASTM D-5373/D-4239/E-442/D-5142	x	x		x
Leachable Chlorides	GLI G-40/ME-4	x			x
Ash Fusability	ASTM D1857	x			x
Soil pH	SW-846 Method 9045	x			x
Heating Value	ASTM D-1989	x	x		x
IT Corporation (Geotechnical Analyses) - Level A Data					
Particle Size Distribution	ASTM D-422	x			x
Atterberg Limits	ASTM D-4318	x			x
Proctor Density	ASTM D-698	x			x
Soil Classification	ASTM D-2487	x			x

a) Total Metals includes at a minimum arsenic, beryllium, cadmium, chromium, antimony, barium, lead, mercury, silver, thallium, sodium, potassium and phosphorus.

Table D.4-4 Analytical Requirements for Tray Testing

IEA Laboratory (Contaminants Analyses) - Level C Data					
	Matrix	soil	soil	soil	soil
	Sample Number :	**TT1-500**	**TT1-700**	**TT1-900**	**TT1-1100**
VOCs	SW-846 Method 8260A	x	x	x	x
SVOCs	SW-846 Method 8270B	x	x	x	x
PCBs	SW-846 Method 8081	x	x	x	x
Dioxins/Furans	SW-846 Method 8290	x		x	
	Matrix	soil	soil	soil	soil
	Sample Number :	**TT2-500**	**TT2-700**	**TT2-900**	**TT2-1100**
VOCs	SW-846 Method 8260A	x	x	x	x
SVOCs	SW-846 Method 8270B	x	x	x	x
PCBs	SW-846 Method 8081	x	x	x	x
Dioxins/Furans	SW-846 Method 8290	x		x	

Table D.4-5 Analytical Requirements for RTA Testing : Soil

	Sample Number :	RTAN	RTA1	RTA1-D	RTA1-M	RTA2	RTA3 (a)
	Matrix	soil	soil	soil	soil	soil	soil
IEA Laboratory (Contaminants Analyses) - Level C Data							
VOCs	SW-846 Method 8260A	x	x	x	x	x	x
SVOCs	SW-846 Method 8270B	x	x	x	x	x	x
PCBs	SW-846 Method 8081	x	x	x		x	x
Dioxins/Furans	SW-846 Method 8290	x	x			x	x
Total Metals (b)	SW-846 Methods 6010/7000	x	x			x	x
TCLP Metals	SW-846 Methods 1311/6010/7000	x	x			x	x
Galbraith Laboratory (Chemical Properties) - Level A Data							
Ultimate/Proximate	ASTM D-5373/D-4239/E-442/D-5142	x	x	x		x	x
Leachable Chlorides	GLI G-40/ME-4	x	x			x	x
Soil pH	SW-846 Method 9045	x	x			x	x

a) The optional Debris Sample will be tested if determined necessary during the Pretreatment/Material Handling Study.
b) Total Metals includes at a minimum arsenic, beryllium, cadmium, chromium, antimony, barium, lead, mercury, silver, thallium,

Table D.4-6 Analytical Requirements for RTA Testing ("Worst Case" Soil Matrix, N2 Purge) : Offgas

	Sample Number :	RTAN-Water	RTAN-MeOH	RTAN-XAD	RTAN-TNX
	Matrix	Water	Methanol	XAD Resin	Tenax Resin
IEA Laboratory (Contaminants Analyses) - Level B Data					
VOCs	SW-846 Method 8260A	x	x		x
SVOCs	SW-846 Method 8270B	x		x	
PCBs	SW-846 Method 8081	x		x	
Dioxins/Furans	SW-846 Method 8290	x			
Galbraith Laboratory (Chemical Properties) - Level A Data					
Acidity	EPA 310.1	x			
Water pH	EPA 150.1/SW-846 9040/9045	x			
TOC	EPA 415.1/SW-846 9060	x			
Total Sulfur	EPA 375.2/375.4/300.0	x			
Total Chloride	EPA 325.2/325.3	x			

Table D.4-7 Analytical Requirements for RTA Testing ("Worst Case" Soil Matrix, Air Purge) : Offgas

	Sample Number :	RTA1-Water	RTA1-MeOH	RTA1-XAD	RTA1-TNX
	Matrix	Water	Methanol	XAD Resin	Tenax Resin
IEA Laboratory (Contaminants Analyses) - Level B Data					
VOCs	SW-846 Method 8260A	x	x		x
SVOCs	SW-846 Method 8270B	x		x	
PCBs	SW-846 Method 8081	x		x	
Dioxins/Furans	SW-846 Method 8290	x			
Galbraith Laboratory (Chemical Properties) - Level A Data					
Acidity	EPA 310.1	x			
Water pH	EPA 150.1/SW-846 9040/9045	x			
TOC	EPA 415.1/SW-846 9060	x			
Total Sulfur	EPA 375.2/375.4/300.0	x			
Total Chloride	EPA 325.2/325.3	x			

Table D.4-8 Analytical Requirements for RTA Testing ("Typical Case" Soil Matrix, Air Purge) : Offgas

	Sample Number :	RTA2-Water	RTA2-MeOH	RTA2-XAD	RTA2-TNX
	Matrix	Water	Methanol	XAD Resin	Tenax Resin
IEA Laboratory (Contaminants Analyses) - Level B Data					
VOCs	SW-846 Method 8260A	x	x		x
SVOCs	SW-846 Method 8270B	x		x	
PCBs	SW-846 Method 8081	x		x	
Dioxins/Furans	SW-846 Method 8290	x			
Galbraith Laboratory (Chemical Properties) - Level A Data					
Acidity	EPA 310.1	x			
Water pH	EPA 150.1/SW-846 9040/9045	x			
TOC	EPA 415.1/SW-846 9060	x			
Total Sulfur	EPA 375.2/375.4/300.0	x			
Total Chloride	EPA 325.2/325.3	x			

Table D.4-9 Analytical Requirements for RTA Testing (Optional Debris Matrix, Air Purge) : Offgas (a)

	Sample Number :	RTA3-Water	RTA3-MeOH	RTA3-XAD	RTA3-TNX
	Matrix	Water	Methanol	XAD Resin	Tenax Resin
IEA Laboratory (Contaminants Analyses) - Level B Data					
VOCs	SW-846 Method 8260A	x	x		x
SVOCs	SW-846 Method 8270B	x		x	
PCBs	SW-846 Method 8081	x		x	
Dioxins/Furans	SW-846 Method 8290	x			
Galbraith Laboratory (Chemical Properties) - Level A Data					
Acidity	EPA 310.1	x			
Water pH	EPA 150.1/SW-846 9040/9045	x			
TOC	EPA 415.1/SW-846 9060	x			
Total Sulfur	EPA 375.2/375.4/300.0	x			
Total Chloride	EPA 325.2/325.3	x			

(a) - The optional Debris Sample will be tested if determined necessary during the Pretreatment/Material Handling Study.

Table D.4-10. Sample Containers, and Preservation Methods

Parameter	Analytical Method	Matrix	Number and Type of Container	Preservation Requirement
Analytical Laboratory (Contaminants Analyses)				
VOCs	SW-846 Method 8260A	soil	(1) 4-oz. glass jar with Teflon-lined cap	Cooled to 4°C
		water	(3) 40-ml glass VOA vials with Teflon-lined silicone septa	Cooled to 4°C
		resin	n/a	Cooled to 4°C
SVOCs PCB's	SW-846 Method 8270B SW-846 Method 8081	soil	(1) 500 ml amber, glass jar with Teflon-lined cap	Cooled to 4°C
		water	(2) 1-liter amber glass jar with Teflon-lined cap	add 3 ml 10% sodium thiosulfate per gallon, Cool to 4°C
		resin	n/a	Cooled to 4°C
Dioxins/Furans	SW-846 Method 8290	soil	(1) 250 ml amber, glass jar with Teflon-lined cap	Cooled to 4°C
		water	(2) 1-liter amber glass jar with Teflon-lined cap	Cooled to 4°C
Total Metals TCLP Metals	SW-846 Methods 6010/7000 SW-846 Methods 1311/6010/7000	soil	(1) 8-oz. glass jar with Teflon-lined cap	Keep from Freezing
Galbraith Laboratory (Chemical Properties)				
Ultimate/Proximate	ASTM D-5373/D-4239/E-442/D-5142	soil	(1) 8-oz. glass jar with Teflon-lined cap	Keep from Freezing
Leachable Chlorides	GLI G-40/ME-4			
Ash Fusability	ASTM D1857			
Soil pH	SW-846 Method 9045			
Heating Value	ASTM D-1989			
Acidity	SW-846 Method 310.1	water	(1) 4-oz. glass jar with Teflon-lined cap	Keep from Freezing
Chlorides	SW-846 Method 325.2			
Soils Laboratory (Geotechnical Analyses)				
Particle Size Distribution	ASTM D-422	soil	(1) 5-gallon bucket per soil matrix	Keep from Freezing
Atterberg Limits	ASTM D-4318			
Proctor Density	ASTM D-698			
Soil Classification	ASTM D-2487			

Table 5-1. Monitoring Instrument Calibration Requirements

Instrument Number	Instrument Description	Approximate Span	Last Calibration (a)
TRAY TESTING			
---	Gas Temperature Thermocouple	1,300 °F	6 months
---	Soil Temperature Thermocouple	1,300 °F	6 months
RTA TESTING			
T1	Gas Temperature Thermocouple	1,300 °F	6 months
T2	Soil Temperature Thermocouple	1,300 °F	6 months
F2	Inlet Purge Flowmeter	2 l/min	12 months
F3	Outlet Purge Flowmeter	2 l/min	12 months
F4	TENAX Train Flowmeter	2 l/min	12 months
F5	XAD Train Flowmeter	2 l/min	12 months
F6	Primary Exhaust Flowmeter	5 l/min	12 months
A1	RTA Cylinder Oxygen Analyzer	25 vol%	Day of Test
A2	CO Analyzer	30,000 ppmv	Day of Test
A3	THC Analyzer (as propane)	30,000 ppmv	Day of Test

Notes:

a) Records must be provided documenting last calibration within the time frame listed.

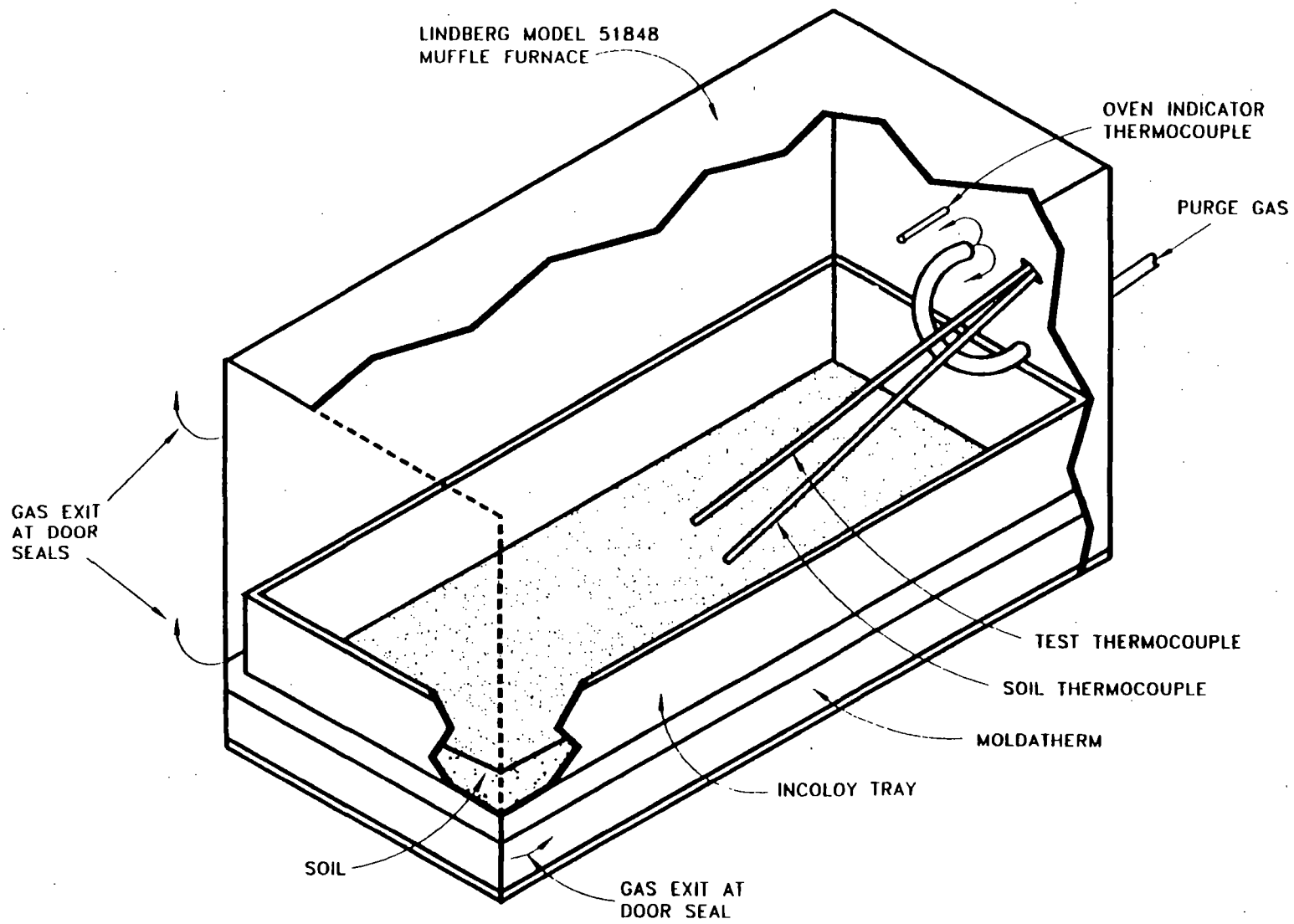

Figure 2-1. Tray Test - General Configuration

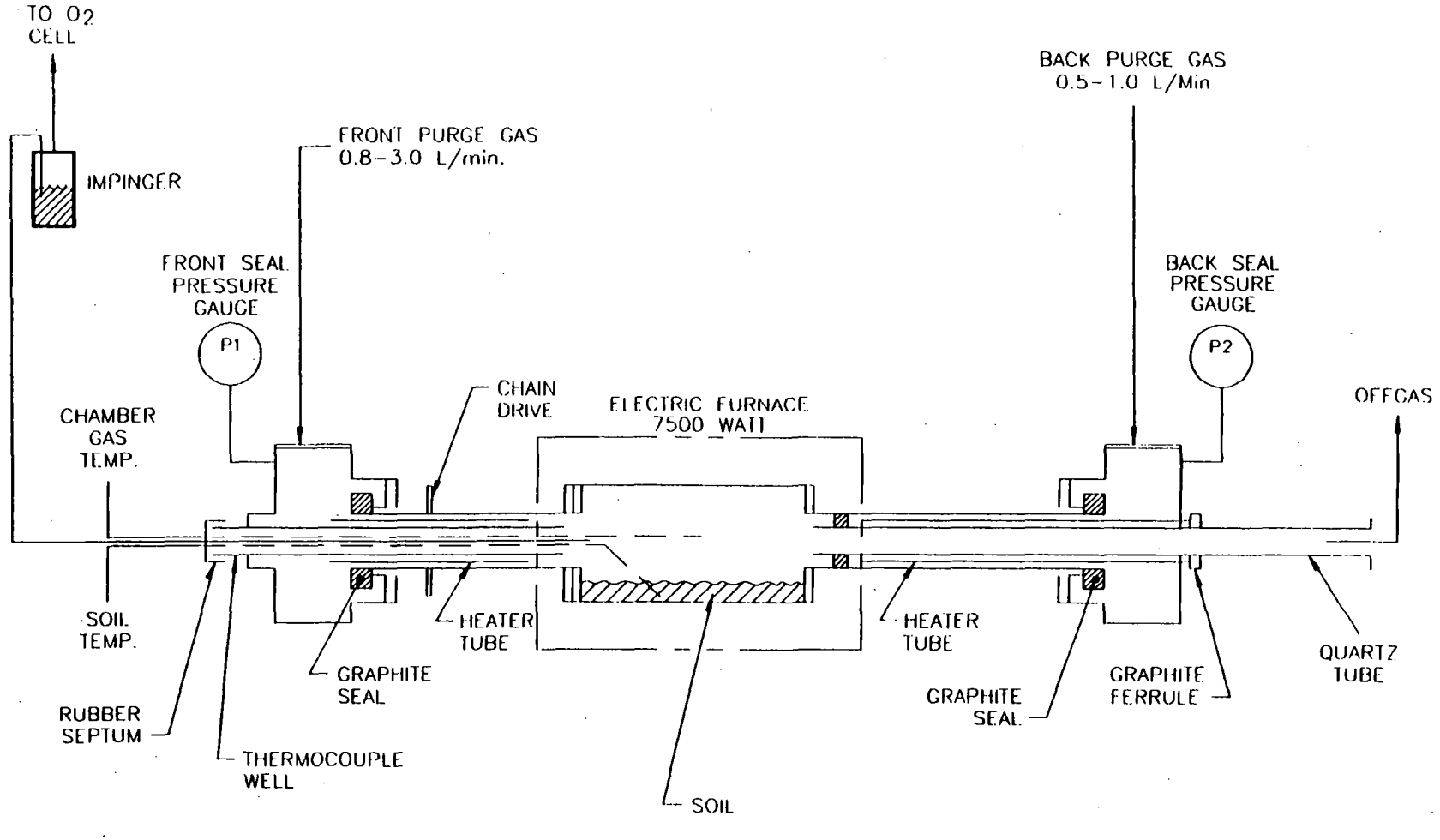

Figure 2–2. Schematic of RTA Test Equipment

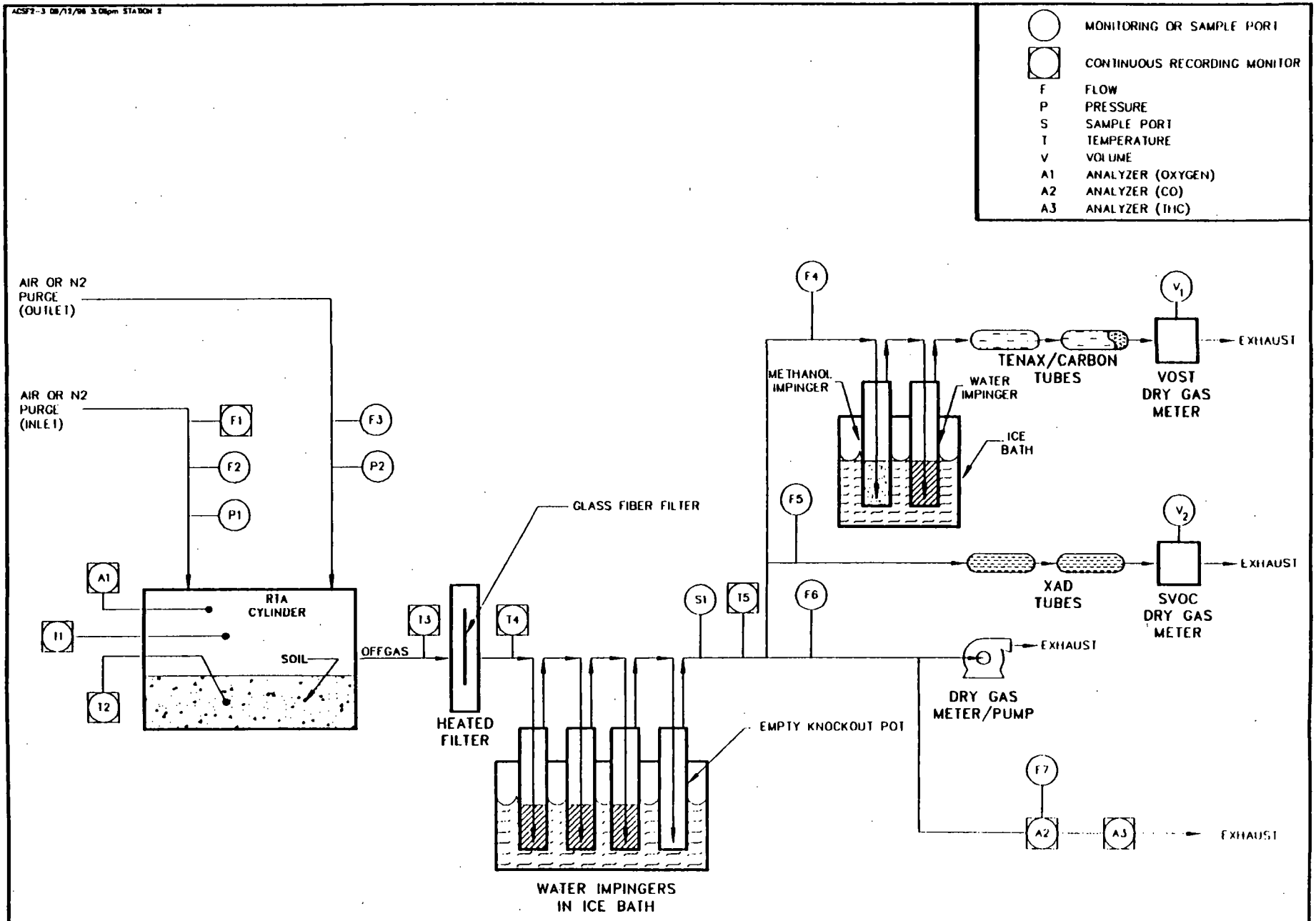

Figure 2-3. General Flow and Monitoring Diagram for RTA Testing

APPENDIX E

STANDARD OPERATING PROCEDURES FOR FIELD INSTRUMENT CALIBRATION

APPENDIX E

STANDARD OPERATING PROCEDURES FOR FIELD INSTRUMENT CALIBRATION

STANDARD OPERATING PROCEDURES
FOR
FIELD INSTRUMENT CALIBRATION

STANDARD OPERATING PROCEDURES
FOR
FIELD INSTRUMENT CALIBRATION

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1.0 INTRODUCTION

This guideline provides basic information on operating procedures for equipment that is typically used in the field. Field monitoring instruments are used when data quality objectives specify Level I and Level II analytical support. Level I analysis provides data for on site, real-time measurements, evaluation of existing conditions, refinement of sampling locations, and health and safety evaluations. The objective of Level I analysis is to generate data that are generally used in refining sampling plans and in estimating the extent of contamination at the site. This type of support also provides real-time data for health and safety purposes. The purpose of this SOP is to define the calibration and operating procedures for equipment used for field monitoring.

2.0 DEFINITIONS

None.

3.0 RESPONSIBILITIES

The following is a general description of responsibilities related to calibration and operating procedures for equipment used for field monitoring.

Project Manager: The Project Manger is responsible for identifying the appropriate equipment necessary to adequately define the parameters.

On-Site Safety Officer: The On-Site Safety Officer will work with the Project Manager in identifying the appropriate safety equipment. The On-Site Safety Officer will also perform audits to observe field personnel using the equipment. If the equipment is not being used properly, the On-Site Safety Officer will provide the necessary training and resources.

Field Team Leader (FTL): The FTL will be responsible for including a refresher course as part of the kick-off meeting on the proper use, calibration, and maintenance of all equipment to be used on the project. The FTL will ensure that all field team members continue to properly use the equipment through the duration of the project.

Field Team Members: Prior to mobilization, a field team member will be required to check out equipment or supplies requested for the project and sign an equipment

checklist form stating that all equipment is functioning, can be calibrated, is in good condition, all the necessary parts are included in the case, and all supplies requested are present. The warehouse manager and/or assistant warehouse manager will also be responsible for signing the equipment checklist. Upon demobilization, the checklist will be used to check the equipment back into the warehouse. At that time, all equipment will be calibrated, if applicable, and checked for proper use by a field team member and the warehouse manager and/or the assistant warehouse manager. Both the field team member and the warehouse manager and/or warehouse assistant manager will be required to sign the checklist.

The field team members will be responsible for carefully reviewing the SOP and instruction manuals for the equipment to be used on the project. All field team members will be required to sign a form indicating that they know how to properly operate each piece of equipment that will be used on their project.

Warehouse Supervisor: An appropriate training and certification procedure must be developed and incorporated into the responsible organization's training procedures. All users are trained and certified to operate equipment before using the instrument in the field. Refresher courses are given every 6 months and as part of each project's kickoff meeting.

Warehouse Manager/Assistant Warehouse Manager: When the instrument is scheduled or requires maintenance, these functions are conducted by qualified individuals. Maintenance responsibilities are restricted to two individuals who are responsible for logging the equipment in and out. Documentation of instrument user, dates of use, instrument number, maintenance and calibration functions, and project identification are maintained.

Before the instrument is taken into the field, it will be inspected and calibrated by a qualified individual to ensure that it is operating properly.

This SOP provides a summary of the calibration and operating procedures outlined in the manufacturer's instruction manual which accompanies each piece of equipment. This SOP should be reviewed and used in conjunction with the manufacturer's instruction manual by field team members when using the equipment.

4.0 GUIDELINES

4.1 APPLICABILITY

These procedures apply to all work conducted for Montgomery Watson clients, by Montgomery Watson, or under the direction of Montgomery Watson. The information in this SOP may be incorporated into project-specific plans. Deviations or modifications to procedures not addressed in the project plans must be handled as a corrective action.

To protect the equipment, it must be kept in the proper cases, packaged properly, and/or secured during transport. If equipment is damaged during transport because it was not properly secured in its case, resulting charges will be directly applied to the job.

Equipment or supplies at job sites should never be left unsecured where they can be lost or stolen. Warehouse equipment should not be left at job sites between jobs without the warehouse manager's approval. Warehouse equipment is also not to be kept in offices or garages. If equipment is lost or stolen while it is checked out to a project, that project will be responsible for replacing the equipment.

Any equipment failures are to be reported immediately to the warehouse manager and returned as soon as possible for repairs. This notification ensures that proper charges/credits are documented and that a replacement is delivered to the site.

All equipment will be decontaminated at the job site prior to returning the equipment.

4.2 TEMPERATURE

Temperature readings are used in the calculation of various forms of alkalinity, in studies of saturation and stability with respect to calcium carbonate, in the calculation of salinity, and in general laboratory operations. An outline of procedures for the proper calibration and measurement of temperature in groundwater samples while in the field follows.

4.2.1 Calibration Procedures

Any temperature measurement devices should be calibrated with a National Bureau of Standards-certified thermometer before field use. Readings are made with the

thermometer or device immersed in water long enough to permit complete equilibration. Results should be reported to the nearest 0.1 or 1.0 degree Celsius (°C), depending on the need.

4.2.2 Operating Procedures

Temperature measurements may be made with any good mercury-filled Celsius thermometer. At a minimum, the thermometer should have a scale marked for every 0.1°C, with markings etched on the capillary glass. The thermometer should have a minimal thermal capacity to permit rapid equilibration. Periodically, the thermometer should be checked against a precision thermometer certified by the National Bureau of Standards used with its certificate and correction chart. For field operations, a thermometer with a metal case should be used to prevent breakage.

Temperature is measured as follows: 1) measure temperature downhole or in a small flow through cell, 2) record measurements periodically throughout the time of purging, and 3) record the measurement in the field logbook and on the appropriate data form.

4.3 FIELD MEASUREMENT OF pH

Determining pH is critical for predicting and interpreting the reactions and migration of dissolved chemical constituents in ground or surface water. Ground or surface water pH should be determined when the sample is collected in the field.

The pH probe measures the hydrogen electric potential of the external solution by comparing it to an isolated solution with a known hydrogen electric potential. A thin glass membrane functions as a cation exchange surface. When the electric potential of the interior of the glass membrane is compared to the electric potential of a standard solution kept isolated from the environment, a quantitative determination of the change in the internal solution's electric potential, induced by the external solution, can be made.

There are numerous adequate pH meters available. Meters used should have temperature and slope adjustments and a repeatability of ± 0.01 standard pH units. Meters used for pH field measurement should also be of rugged construction. A foam-lined carrying case is convenient both for transport and for use as a work table. Also, a spare a electrode and batteries should be available in the field.

4.3.1 Hydac pH/Conductance/Temperature Meter

4.3.1.1 Calibration. Always calibrate the instrument according to the manufacturer's instructions taking into account the guidelines provided in this section. The meter must be calibrated before the start of each work day and checked periodically throughout the work day. Consult the Hydac instruction manual for further details.

1) The two buffers used for calibration should bracket the anticipated pH of the groundwater samples. For an anticipated pH of 6, calibrate with pH 4 and pH 7 buffers; for an anticipated pH of 8, calibrate with pH 7 and pH 10 buffers. Buffers and samples are kept at the same temperature. This action will eliminate the need to correct values for temperature effects. Theoretically, buffer solutions are stable indefinitely; however, they are susceptible to contamination. Therefore, old, partially full bottles are often contaminated and should be replaced.

2) Temperature and conductance are factory calibrated. However, conductance accuracy can be checked with a standard solution of known conductance. If recalibration is necessary, the following procedures are necessary. Remove the block plug revealing the adjustment potentiometer screw. Rinse sample cup with standard solution, discard, and refill. Adjust the potentiometer until the digital display indicates the known value of the conductivity standard solution. To increase the digital display reading, turn the potentiometer screw counterclockwise and clockwise to decrease.

3) The pH electrode and meter are standardized as follows. Place the pH in a container of 7.0 buffer solution. Adjust the ZERO potentiometer on the face of the meter so that the digital display indicates 7.00. Then place the pH electrode in a container with the pH 4 or 10 buffer, depending on estimated pH of the sample. Adjust the SLOPE potentiometer on the face of the meter so that the digital display indicates the value of the buffer chosen. Check the pH calibration by repeating step 2 several times.

4.3.1.2 Operation. The following procedures are for operation of the Hydac Meter.

1) Connect the pH cable connector onto the tester. Push and twist clockwise. Rinse the inside of the sample cup with sample to be measured.

2) Fill sample cup at least 2/3 full.

3) Slide function switch to TEMP and push the READ button. Read temperature on digital display panel and note in field logbook and appropriate data form. Adjust both temperature controls accordingly. If temperature reading is not stable, rinse the sample cup several times to bring the sample cup to the same temperature as the sample.

4) Slide function switch to COND and select the appropriate range. Push the READ button. Read the conductivity on the digital display panel and multiply the digital display reading by the factor indicated by the range

switch to determine the conductance. Note the conductivity reading in the field logbook and appropriate data form. If a single "1" appears on the lefthand side of the digital display panel, the conductivity of the sample is higher than the selected range. Slide the range switch to the next selection. If a decimal display appears (such as 0.11) move the range selector switch to the left until a 3- or 4-digit number appears.

5) Always obtain conductivity reading before placing the pH probe in the sample container to avoid cross contamination. Slide the function switch to pH. Remove the storage cap from the pH electrode. Rinse the electrode with distilled sample. Shake the electrode with a snap motion to remove residual drops of solution to avoid cross contamination. Place the pH electrode in the sample cup. Press the READ button. Note the pH value on the digital display panel in the field logbook and the appropriate data form.

4.3.1.3 Maintenance. The following procedures are for maintenance of the Hydac meter.

1) Do not subject the pH electrode to freezing temperatures. Electrodes should be immersed in a pH 4 or pH 7 buffer solution when not in use. Always rinse the electrode with distilled water prior to use.

2) The meter is NOT waterproof. Take care to keep the meter dry.

3) Keep the cotton in the electrode cap moist with distilled water. Always keep the cap on the electrode while being stored. If bubbles are seen in the bulb area of the electrode, hold the electrode by its cap and shake downwards.

4) Replace the 9-V battery when the LO BAT can be read on the digital display panel. Battery is located behind the snap-off cover on the bottom of the meter.

5) After each sample, wipe the sample cup with a paper towel or tissue and rinse again. The carbon electrodes in the cup may be cleaned with a mild abrasive on the end of the flat surface.

6) Keep in mind that all pH electrodes age with time. Aging is characterized by shortened span and slower speed of response. The pH meter slope control can be adjusted to compensate for electrode span errors and will not affect the speed of response. When the slope control can no longer be adjusted to compensate for the span errors, the electrode should be cleaned and/or reconditioned. If reconditioning does not restore electrode performance, the electrode should be replaced.

7) Coating of the pH bulb can lead to erroneous readings including shortened span. Soft coatings can be removed by vigorous stirring or by use of a squirt bottle. Organic chemical or hard coatings should be chemically removed. In no case should the bulb be mechanically cleaned; abrasion can lead to permanent damage.

4.3.2 Orion Model SA 250 pH Meter

4.3.2.1 Calibration. Always calibrate the instrument according to the manufacturer's instructions taking into account the guidelines provided in this section. The meter must be calibrated before the start of each work day and checked periodically throughout the work day. Consult the Orion Model SA 250 instruction manual for further details.

1) The two buffers used for calibration should bracket the anticipated pH of the groundwater samples. For an anticipated pH of 6, calibrate with pH 4 and pH 7 buffers; for an anticipated pH of 8, calibrate with pH 7 and pH 10 buffers. Keep buffers and samples at the same temperature. This action will eliminate the need to correct values for temperature effects. Theoretically, buffer solutions are stable indefinitely. However, they are susceptible to contamination. Therefore, old, partially full bottles are often contaminated and should be replaced.

2) Connect the pH electrode to the meter. Slide the mode switch to either pH 0.01 or 0.1. Choose either 4.01 and 7.00 or 7.00 and 10.01 buffers to calibrate the meter, whichever will bracket the expected sample range.

3) Place the electrode in the one of the buffers selected. Press CAL. The display will alternate between .1. and the pH value of the buffer, indicating this is the first buffer and a value has not been entered. Wait for a stable pH display and press ENTER. The correct display will freeze for three seconds then advance to .2. indicating the meter is ready for the second buffer.

4) Rinse the electrode and place into the second buffer. Wait for a stable pH display and press ENTER. After the second buffer value has been entered the letters pH will be displayed. The meter is now calibrated and automatically advances to sample mode.

4.3.2.2 Operation

1) Rinse the electrode and place into sample. Note the pH value displayed on the digital panel in the field logbook and appropriate field data form.

2) To manually compensate for temperature, use a thermometer accurate to ±1°C to determine the temperature of the solution to be measured. Slide the mode switch to TEMP. Scroll using ^, v or the x10 keys, until the correct temperature value is displayed. Press ENTER. Return the mode switch to either pH .1 or pH .01.

4.3.2.3 Maintenance

1) To verify the instruments calibrations, press the SLOPE key and note the reading on the display. A properly functioning electrode will have a 92 to 102 percent slope. The slope value is retained in the meter's memory until another two buffer calibration is performed or another value is entered.

2) Consult instruction manual for definition of error messages and associated remedy.

4.4 FIELD MEASUREMENT OF SPECIFIC CONDUCTANCE

Specific conductance is used as a indicator of water quality. It is a simple indicator of the change within a system and provides useful information to laboratory. Specific conductance is the reciprocal of electrical resistivity. The value of electrical resistivity is dependent upon the amount of ions in the solution. Pure water has 100 percent resistivity and 0 specific conductance.

There are numerous adequate specific conductance meters available. Any meter used to collect field specific conductance measurements should be equipped with a temperature compensator, and read directly in micromhos per centimeter (µmhos/cm) corrected to 25°C. A foam lined carrying case is essential. A relative accuracy of ±3 percent is adequate for the type of measurement being performed.

4.4.1 YSI Model 33 Conductivity Meter

The YSI Model 33 meter is designed to read conductivity measurements in ranges 0 to 500 (x1), 0 to 5,000 (x10), and 0 to 50,000 micromhos/cm (x100). The "micromho" designations on the meter are shorthand form for micromhos/cm. The YSI Model 33 meter does not compensate for temperature when measuring for conductivity.

4.4.1.1 Calibration. Always calibrate the instrument according to the manufacturer's instructions taking into account the guidelines provided in this section. The meter must be calibrated before the start of each work day and checked periodically throughout the work day. Consult the instruction manual for further details.

Reagent-grade potassium chloride (KCL) is universally used for the standardization of specific conductance equipment. Standard solutions are made up at 0.001 molar (M), 0.01M, 0.1M, or 1M KCL. A concentration of 0.01M is typically used because its specific conductance is closest to most natural samples.

The electrodes are calibrated by reading the specific conductance of standard KCL solutions. The following guidelines should be used in conjunction with the manufacturer's instructions:

The electrodes are calibrated by reading the specific conductance of standard KCL solutions. The following guidelines should be used in conjunction with the manufacturer's instructions:

1) Calibration is done at the factory. However, the instrument calibration should be checked periodically against a standard solution of KCL. Measure the temperature of the standard solution and the water used for the dilution if applicable. Ideally, they should be the same temperature. Note the temperatures in the field logbook and field form specified in the work plan.

2) Use the table furnished with the commercial standard to determine the expected specific conductance of the standard being used to calibrate the instrument at the measured temperature.

3) Large changes in the cell constant are generally an indication that the electrodes are dirty. Cleaning and replatinizing instructions are included below and can also be found in the instrument manual.

4.4.1.2 Operation

1) Switch MODE control to OFF. Use meter screw on the meter face to adjust meter needle to 0 conductance.

2) Plug probe into jack.

3) Switch MODE to RED LINE. Use the RED LINE control to adjust the needle to the red line on the conductivity scale.

4) Switch MODE to TEMPERATURE. Place probe in sample. Allow time for the probe temperature to come equilibrium with that of the sample. Read the temperature on the bottom scale of the meter in degrees Celsius. Note the temperature in the field logbook and the appropriate data form.

5) Switch to x100 on the conductivity scale. If the reading is below 50 on the 0-500 range, switch to x10 on the conductivity scale. If the reading is still below 50, switch to the x1 scale. Read the meter scale and multiply the reading appropriately. Measurements are not temperature compensated. Note the reading in the field logbook and the appropriate data form.

4.4.1.3 Maintenance

1) Replace batteries every 6 months. Two "D" size alkaline batteries will provide 200 hours of operation. Batteries should also be replaced if the redline adjustment cannot be accomplished. To replace the batteries, remove the screws from the rear cover. The battery covers are color coded. The positive end must go on red.

2) When the cell test indicates low readings, the probable cause is dirty electrodes. Hard water deposits, oils, and organic matter are the most likely contaminants. Either soak the electrodes in bathroom cleaner or a solution of 10 parts distilled water, 10 parts isopropyl alcohol, and 1 part

HCl. Always rinse the probe thoroughly in tap water, and distilled or deionized water after cleaning and before storage. If the cell test still indicates low readings, replatinizing is required.

3) Store the conductivity cell in deionized water. This will decrease the frequency that the cells need to be replatinized.

4) Large changes in the cell constant are generally an indication that the electrodes are dirty. To replatinize the cell, place the cell in a container filled with enough YSI 3140 Platinizing Solution to cover the electrodes. Plug the probe into the meter, switch to x100 scale, and allow approximately 10 minutes to platinize the electrode. Remove the probe and rinse with tap water.

4.4.2 YSI Model 3000 T-L-C Meter

The YSI T-L-C Meter measures temperature, water level or depth, conductivity, and temperature compensated conductivity for water quality parameters. By convention, the conductivity of a solution is referenced to 25°C. In both temperature compensated conductivity ranges, the reading is automatically corrected to 25°C.

4.4.2.1 Calibration. Always calibrate the instrument according to the manufacturer's instructions taking into account the guidelines provided in this section. The meter must be calibrated before the start of each work day and checked periodically throughout the work day. Consult the instruction manual for further details.

Reagent-grade potassium chloride (KCl) is universally used for the standardization of specific conductance equipment. Standard solutions are made up at 0.001 molar (M), 0.01M, 0.1M, or 1M KCl. A concentration of 0.01M is typically used because its specific conductance is closest to most natural samples.

The electrodes are calibrated by reading the specific conductance of standard KCL solutions. The following guidelines should be used in conjunction with the manufacturer's instructions:

1) The YSI T-L-C Meter is calibrated at the factory. There are no user adjustments inside the instrument. Monitor the instruments calibration using a standard solution. Rinse the probe with the solution to remove any contamination. Immerse the probe in the standard solution and set the function switch to one of the temperature compensated ranges, depending on the range of the standard solution. Compare the reading with the standard solution value to determine the instruments accuracy. Note the instruments reading and associated temperature. If the readings indicate

that the instrument is out of calibration, notify the Montgomery Watson warehouse manager and return the instrument to the factory for recalibration. The percent error calculated can be used to as a correction factor to improve the accuracy of measurement of the sample being tested. Use the following formula to calculate the corrected conductivity value if necessary:

4.4.2.2 Operation

1) To measure temperature, set the function switch to °C. Completely submerge the probe in sample to be measured and allow sufficient time for temperature of the probe and the sample to equilibrate, approximately 40 to 60 seconds. Read the displayed value after the reading has stabilized and note in field logbook and data form.

2) To measure conductivity, set the function switch to 2 m_/cm. Completely submerge the probe and allow sufficient time for it to reach equilibrium with the sample, approximately 10 to 20 seconds. If the over range signal is displayed (1.__), then the conductivity of the sample is greater than 1.99 m_/cm. Reset the function switch to 20 m_/cm. If the over range signal is still displayed, the conductivity is greater than 19.99 m_/cm and cannot be measured without dilution. Observe the displayed value after the reading is stable. Note measurement in the field logbook and appropriate data form.

3) To measure temperature compensated conductivity, set the function switch to 2 m_/cm TC to 25°C. Follow step 2 but reset the function switch to 20 m_/cm TC to 25°C if the over range signal is still displayed. Observe the displayed value after the reading is stable. Note measurement in the field logbook and appropriate data form.

4) To measure water level or depth, switch the function key to any of the conductivity modes. Lower the probe into the well or borehole and observe the display panel. When the value displayed rises the electrode is measuring the conductivity of the water. Raise the probe slowly out of the water until the displayed value goes back to approximately zero. Lower the probe very slowly until the reading rises suddenly. Observe the cable mark at the well reference point and note measurement in the field logbook and appropriate data form.

4.4.2.3 Maintenance

1) **Battery Replacement.** When BAT appears in the upper left corner of the display panel, approximately 8 hours of use remains. To replace batteries, six heavy-duty "C" batteries are necessary. Uncoil about one foot of the probe cable. Unscrew counterclockwise direction the screw located on the face of the instrument. Remove the electronics and probe reel assemblies from the housing, and disconnect the probe connector (located on the back of the electronics assembly) by turning its sleeve counterclockwise. Remove the probe reel from the electronics assembly. Remove the nine screws located on the back of the electronics assembly and separate the front and back halves. Install the batteries into the holders located on the

circuit board. Remove, dry, and replace the desiccant container. Reassemble.

2) **Desiccant Replacement.** When the case is opened for any reason, the interior will have the same relative humidity as the surrounding air. If resealed and later operated at a lower temperature, internal condensation might occur. The desiccant prevents this condition from happening. Periodically remove the desiccant container for drying or replacement if necessary.

3) **Seal Replacement.** To insure watertight integrity, the o-rings should be replaced annually. Disassemble and assemble the instrument as described in Step 1.

4) **Cleaning.** Clean and decontaminate the instrument at the end of each project. Use a solution of liquid detergent and water. Rinse the instrument with clean water. Do not clean the instrument while it is disassembled. Use a bathroom cleaner to clean the probe. Use isopropyl alcohol if a stronger cleaning solution is required. Rinse the probe several times with distilled or deionized water.

5) **Replatinizing the Probe.** If cleaning the probe does not restore the probe's performance, or if flaking or other defects in the platinum black coating are apparent, replatinizing is necessary. A YSI Model 3045 Platinizing Instrument and solution are necessary for this procedure. If the probe requires replatinizing while in the field, notify the warehouse manager of the situation and return the instrument to the warehouse. A backup or alternate meter should be used at this point.

6) **Storage.** Store the conductivity probe in deionized water to reduce the frequency of replatinizing. If the probes have been stored dry, it is necessary to soak them in deionized water for 24 hours prior to use.

4.5 LaMOTTE MODEL 2008 TURBIDITY METER

The LaMotte Model 2008 portable turbidity meter measures the amount of light scattered at right angles from a beam of light passing through the test sample. Turbidity readings are the measure of the interaction of light with suspended solid particles in the sample. Test results are read directly in Nephelometric Turbidity Units (NTUs) on the LCD digital readout. Nephelometry refers to the measurement of light scattering in the direction perpendicular to its propagation. The sample to be analyzed is placed in an optically selected sample tube. The sample tube is inserted in a light tight compartment. When the instrument is turned on, light passing through the turbid sample will be scattered and only the light that is scattered at right angles to the light source is detected by the dual photocells and registered by the meter. The display panel will register the appropriate NTU value on the calibrated scale. Two ranges are available; 0-20 and 0-200 NTUs. The low range (0-20 NTU) is used for monitoring drinking water quality, while the high range (0-200 NTU) may be used for a wide range of monitoring applications.

Turbidity measurements will be made and recorded in the field logbook for all groundwater samples collected.

4.5.1 Calibration

The turbidimeter is pre-calibrated in LaMotte laboratories, and a simple standardization is the only step required prior to testing. Two standards are supplied with each Model 2008 turbidimeter for standardization.

Always calibrate the instrument according to the manufacturer's instructions taking into account the guidelines provided in this section. The meter must be calibrated before the start of each work day and checked periodically throughout the work day. Consult the instruction manual for further details.

4.5.2 Operation

1) Collect a sample of the liquid to be measured and set aside to allow the sample time to equilibrate to ambient temperature and allow any entrained gasses to escape. Keep dust or other airborne contaminates from contact with the sample.

2) Rinse the sample tube with the liquid sample and shake the excess liquid out. Fill the sample tube to its neck, taking care to pour the sample gently down the side to avoid creating any bubbles.

3) Cap the sample tube. While holding the sample tube by the cap only, wipe the outside surface with a clean, lint-free, absorbent wipe until the tube is dry and smudge-free. Set the sample tube aside on a clean surface that will not contaminate or scratch the bottom of the tube.

4) Select the appropriate range, and insert a sample tube containing the standard with a value close to that of your sample. Make sure the tube is seated at the bottom of the chamber. Cap the chamber. The green LED on the front panel must be illuminated, indicating that the lamp is on. Adjust the STANDARDIZE control so that the display reads the known value of the standard.

5) Remove the standard and insert the sample tube. Make sure the tube is seated on the bottom and cap the chamber. The reading should stabilize within 15 seconds. Document the measurement in the field logbook and the appropriate data form. Remove the sample tube from the chamber and cap. The lamp is only illuminated when the instrument is in the measurement mode and a sample tube is seated in the bottom of the chamber. Removing the sample tube between measurements will enhance the battery and lamp life.

6) Periodically verify the stability of the readings by inserting the standard and adjusting if necessary.

4.5.3 Maintenance

1) Keep the instrument clean and dry, especially the sample chamber. Keep the sample chamber capped except while inserting or removing the sample tube. While holding the instrument upside down, clean the sample chamber with compressed gas used for cleaning lenses.

2) If the lamp burns out, return the instrument to the LaMotte Service Laboratory where the lamp can be replaced and the instrument can be recalibrated. To test the stability of the lamp, insert a clean, dry, empty sample tube into the chamber. The actual value under these conditions is unpredictable but should be stable. As long as the display is stable the lamp is useable.

3) Do not charge the instrument's battery until the battery low indicator is visible on the display panel. Recharging from the fully discharged condition (Bat low indicator visible) to the fully charged condition (red LED extinguished) should take 4 to 5 hours. If it takes notably less time to charge, it is a sign of diminished battery capacity. A small indicator will appear in a corner of the front panel when the batteries are nearly exhausted. At the end of the useful charge, the readings will become unstable; they will appear to drift. Recharging the battery is necessary.

4) While in the measurement mode, with the sample chamber empty and capped the instrument should read, if functioning properly and the standardization correctly, between -0.10 and -0.40 NTUs. This reading is a convenient check on the functionability and accuracy of the Model 2008 turbidity meter.

5) The accuracy and repeatability of the measurements is a function of the condition of the standards, technique, and the quality of the glassware. Standards will remain stable indefinitely as long as they are not exposed to excessively hot or cold environments; keep standards between 10°C and 40°C. Once the seal is broken on the standard, the standard is only good for nine months thereafter. When transferring standard to a sample tube, make sure the sample tube is absolutely clean. Rinse the inside of the sample tube with standard prior to transferring the standard. Always transfer the standards in a dust-free environment and promptly cap the containers.

6) Glassware must be clean and defect-free. Scratches and/or abrasions will permanently affect the accuracy of the readings. To limit the variability in measurements due to the imperfect geometry of the glassware, use the same glassware and always orient the sample tube in the chamber the same way.

7) The readings made on any one type of turbidimeter may not agree with a reading for the same sample from a different type of turbidimeter. there will be affixed and determinable relationship between the two readings, but the numerical values may not correspond.

4.6 HANNA MODEL HI 8043 DISSOLVED OXYGEN METER

The Hanna Model HI 8043 Dissolved Oxygen Meter comes with a polargraphic type probe, incorporating a thermistor for automatic temperature compensation. The tip of the probe consists of a cell enclosed by a selective membrane in a protective PVC holder containing the electrolyte and electrodes. The temperature thermistor is lodged inside the electrode body just above the sensor. The oxygen that diffuses through the membrane is reduced at the cathode when an external voltage is applied between the electrodes. The current produced is directly proportional to the amount of oxygen passing through the membrane and the electrolyte.

4.6.1 Calibration

Always calibrate the instrument according to the manufacturer's instructions taking into account the guidelines provided in this section. The meter must be calibrated before the start of each work day and checked periodically throughout the work day. Consult the instruction manual for further details.

1) The instrument is shipped factory calibrated. However, the calibration should be verified periodically and when the anode is completely reduced, the electrolyte or membrane is changed, the electrodes are cleaned, and after excessive use in aggressive solutions.

2) The probe must be polarized prior to use to maintain stable measurements with the same recurring degree of accuracy. When measurements are taken with a non-polarized probe, the oxygen level revealed is both that of the surrounding solution which penetrates into the probe as well as that which is already present in the electrolyte solution. In order to polarize the probe, the function switch should be in the STB mode and the probe covered with the protective cap filled with electrolyte solution for 6 hours.

3) The thermometer should be calibrated at least every 3 months. Immerse the probe and switch the instrument to °C. Agitate the water for at least 10 minutes until thermal equilibrium between the probe and water is achieved. Check the temperature of the water using a thermometer with an accuracy of at least 0.1°C and adjust the °C trimmer until the display reads the thermometer temperature.

4) **Zero Calibration.** Switch the function switch to the 0_2 position. Uncap the probe and immerse in zero calibration solution. Wait 5 minutes and do not stir as this will introduce more oxygen into the solution. The readings must be stable before proceeding. If the probe has not sufficiently polarized, the read-out will continue to fluctuate. In this case, switch back to STB position and wait for a few hours for complete polarization before proceeding. Once the readings are stable, use a small screwdriver to turn

the Zero Calibration screw until the display reads zero. If the zero point is passed, "1" will appear on the display. If you cannot get a zero reading the probe is probably defective and the membrane, electrode, and electrolyte solution should be checked. Rinse the probe with tap water after the zero calibration has been completed.

5) **Slope Calibration.** For an accurate calibration, the probe should be immersed with water in a air tight container. If an open container is used for calibration, the margin of error is approximately 0.1 ppm. Switch the instrument to °C, wait for readings to stabilize and note temperature of water in the field logbook and data form. Refer to Attachment 8 for the corresponding dissolved oxygen reading at displayed temperature. Switch the instrument to 02 and adjust the slope trimmer until the correct reading is displayed. If the calibration is performed above sea level, a correction should be made for the difference in altitude according to Attachment 9.

6) The calibration will remain constant for a period of about 20 hours from the time the electrode is activated. After this time, the surface of the anode is completely reduced and cleaning is necessary to reactivate the whole surface. To clean the anode, unscrew the PVC membrane holder from the probe. Drain the electrolyte solution. Wipe the surface of the dark-gray coil, the silver anode, with a cotton wad and a slightly abrasive cleaning paste. Thoroughly rinse with distilled water. To clean gold cathode, wipe it lightly using a piece of filter paper making sure not to scratch the surface.

4.6.2 Operation

1) Remove the instrument from the packing material and examine carefully to make sure that no damage has occurred during shipment. If noticeable damage is evident, immediately notify shipper and contact the Montgomery Watson warehouse manager.

2) Remove the protective cap before taking measurements. Switch the instrument to "0_2". Immerse the probe in the sample and agitate the probe. Allow several minutes for thermal equilibrium to occur between the probe and the sample. Note the reading in the field logbook and the data form.

3) If the sample contains significant salinity, the read-out values must be corrected, taking into account the lower degree of oxygen solubility present in such conditions. Use manufacturer's table to determine the corresponding quantity to be subtracted for every mg/l of salt present in the sample.

4.6.3 Maintenance

1) **Battery Replacement.** When the battery is low, the "V" symbol will appear on the display. To replace the battery, remove the protective cover on the back side of the instrument. If the battery is replaced immediately, the instrument can be used for measurements after approximately half an hour. If the instrument has remained without a battery for more than 1 minute, allow at least 6 hours for the probe to polarize.

2) After each use, wash the electrode with distilled water or remove any trace organic compounds, bacteria, etc. Wipe with slightly dampened cotton; do not use abrasive materials for cleaning.

3) If the membrane is damaged, replace it with a new membrane. Fill the membrane up with Hl Solution. Delicately tap the side of the holder to remove any bubbles trapped inside. Screw the holder onto the electrode body very slowly, keeping the holder in an upright position.

4) Always store the probe with the cap in place, filled with electrolyte.

4.7 SOLINST WATER LEVEL METER

4.7.1 Operation

1) Upon receipt of meter check set the switch to "on" position. To check the circuit submerse the electrode (probe) in tap water. This completes the circuit and activates the buzzer. Depress the test button to test the battery and circuitry.

2) The zero measurement on the Model 101 meter is at the tip of the inner electrode, visible near the center of the probe. The zero measurement on the Model 102 meter is at the base of the outer body electrode.

3) To adjust the sensitivity, remove the face plate on the reel. Located on the top of the circuit board is a small adjustable potentiometer screw. If the alarm continues after removal from water, the sensitivity should be reduced by turning the screw clockwise. If no alarm is heard and the battery is functioning, the sensitivity should be increased by rotating the screw counterclockwise.

4.7.2 Maintenance

1) After the depth of water has been recorded the cable should be carefully rewound onto the reel, the probe wiped dry and replaced into the probe holder.

2) Decontaminate the probe, cable, and reel between each use.

3) Always store and ship the meter in the padded carrying case.

4) To replace the battery, remove the face plate on the reel by unscrewing the three faceplate screws and carefully lifting off to the side to avoid to avoid damaging the wiring. Replace the 9-V battery making sure the polarity is correct. Replace the faceplate.

4.8 SOLINST MODEL 121 INTERFACE METER

The following is summarized from the manufacturer's manual. For additional information concerning the Solinst Model 121 Interface Meter, consult the instruction manual.

4.8.1 Operation

1) Turn main switch to the "on" position. Also twist probe to the "on" position. A flashing light on the face plate indicates that the probe in the "on" position but the main switch is not "on". A continuous buzz indicates that the main switch is "on", but the probe is "off."

2) Lower probe slowly until lights and audible tone are on. Raise and lower the probe gently to determine the exact upper level of the nonconductive floating product. Note level from marked tape. If no floating product exists, one single light will come on.

3) Continue to lower the probe until only one light is on. Shake the probe slightly at this point to clear any residual product from the conductivity sensor. Raise the probe slowly until both lights and the audible tone are on to determine the product/water interface. Read level directly from the tape.

4) Repeat steps 2 and 3 to confirm readings.

5) To determine if there are any sinking product is present in the well, continue lowering the probe to the bottom of the well. If both lights and buzzer come on, determine the top of the layer by reading directly from the tape.

Notes: One light indicates a conductive liquid (i.e., water).
Both lights plus the audible tone indicates a non-conductive liquid (i.e., product).
A flashing light indicates that the probe switch is "on".
A continuous buzzer indicates that the main switch is "on" but the probe switch is "off."

4.8.2 Maintenance

1) After each use, the tape should be wiped, and carefully rewound onto the reel. The probe should be cleaned with a cleaner, rinsed with a hexane and distilled water mixture, wiped and returned to the holder. Make sure both switches are turned off.

2) To replace the battery in the reel, remove the three screw in the faceplate and carefully lift to one side, to prevent damage to the wiring. Replace the heavy duty 9-V alkaline or lithium type battery, noting the proper polarity. Replace the faceplate and three screws being careful to keep all wires within the hub.

3) To replace the probe battery, remove the three screws (phillips type) at the top of the probe. Gently pull the probe body apart to expose the battery holder. Remove and replace the battery with a 9-V lithium or heavy duty alkaline battery. Ensure correct polarity when replacing the battery. Check the three wire connections between the battery holder and the probe body to ensure a tight connection. Push the probe body back together and replace the phillips screws.

4.9 SENSIDYNE PORTABLE FLAME IONIZATION DETECTOR (FID)

This detector uses a flame ionization detector (FID) for the detection and measurement of organic gases and vapors based on the principle of flame ionization. The carrier gas, hydrogen, and the sample are continuously supplied to the FID chamber by a constant flow pump. As organic vapors burn, a high voltage electrode inside the chamber produces an electrostatic field that collects the carbon-containing ions.

A baseline response (small current) is detected from the carrier gas alone. As organic vapors combine with the carrier gas, the current increases proportionally due to the intensified production of electrons and carbon containing ions. The resultant current is a measurement of the sample concentration.

The FID is factory aligned using methane and is calibrated using methane on a regular basis. The FIDs response is a response to methane. The standard FID measurement range is 0-1000 ppm and 0-10000 ppm methane. Measurement is displayed on a semi-logarithmic, analog meter.

The following is summarized from the manufacturers manual. For additional information concerning the sensidyne RID, consult the instruction manual.

4.9.1 Calibration

Calibration of the FID is recommended to be performed every three months using the Sensidyne FID Calibration Kit. The kit consists of a compressed gas cylinder containing 200 ppm methane in air; a flow regulator to deliver 2 LPM; a tubing assembly to fit both standard and straight probes; and calibration labels.

1) Start up the unit (see operating procedures).

2) Assemble the calibration kit. Turn the flow regulator valve fully clockwise to ensure that it is closed.

3) Screw the flow regulator onto the compressed gas cylinder of methane in air. Attach the tubing assembly to the flow regulator.

4) Open the flow regulator valve and insert the probe into the tubing.

5) Observe the meter and wait for a stable indication. If the meter indication is not 200 ± 50 ppm, recalibration is required. If re-calibration is not required, proceed to Step 6.

a) Remove the label covering the calibration adjust.
b) Using a small slotted screwdriver, turn the calibration adjust until the meter indicates exactly 200 ppm.
c) Replace the label covering the calibration adjust with a new label showing the current date.

6) Remove the probe from the tubing assembly and close the flow regulator valve.

7) Disassemble the calibration kit. Disconnect the tubing assembly from the flow regulator.

8) Unscrew the flow regulator from the compressed gas cylinder of methane in air.

9) Safely store the calibration kit until next use.

4.9.2 Operation

Startup:

1) Press the tubing connected to the probe handle into the filter assembly. Press the filter assembly into the probe inlet.

2) Insert the tapered, open end of the probe into the tapered, open end of the handle and press the ends together.

3) Lightly grease the o-ring on the fuel cylinder with lithium or silicone grease. Place the o-ring into the fuel cylinder fitting.

4) Carefully slide the gas cylinder onto the carriage below the housing.

5) Align the threads of the fuel cylinder valve with the threads of the fuel cylinder fitting. Carefully hand tighten the connection.

6) Turn the volume adjust fully clockwise. Turn the range switch to either range 1 or range 2. The intermittent beep will sound, indicating a flame out condition.

7) Verify that the battery level indicator is in the green sector. If the indicator is in the red sector, do not proceed. The battery requires charging.

8) Check the flowmeter. The float in the flowmeter will rise and the sound of the pump will be audible.

9) If necessary, turn the flow adjust until the float is centered on the middle line of the flowmeter. The float should remain centered on this line while the FID is operational.

10) Slowly open the fuel cylinder valve by turning the valve one quarter to one half turn counter clockwise.

11) Verify that there are no leaks around the connection of the fuel cylinder valve and the fuel cylinder fitting using a soap solution.

12) Read the pressure gauge and verify that the hydrogen pressure is equal to or greater than 190 psi to sustain proper operation of the FID for 1 hour. Fuel pressure should never be allowed to fall below 103 psi. If the indicated pressure is below 130 psi, the fuel cylinder must be refilled or replaced.

13) Allow 30 to 60 seconds to elapse and then depress the igniter. Ignition is indicated by deflection of the meter and a change from the intermittent beep that indicates flame out to a tone.

14) Turn the zero adjust until the analog meter indicates 0 ppm. The user will no longer hear a tone but a low pitched tick.

15) If it is not possible to obtain an indication of 0 ppm, there may be extraneous detectable substances in the environment. Remove the unit to clean air or supply clean air from another source to make the zero adjustment.

16) The unit is now ready for measurement.

Shut down:

1) Turn the fuel cylinder valve clockwise to stop fuel flow to the FID. Wait until the intermittent beep is heard and the indicator on the pressure gauge decreases slowly to zero. It will take 10 to 15 minutes for the pressure to discharge fully.

2) Turn the range switch to the "off" position.

3) Remove the fuel cylinder if the instrument is not used on a daily to weekly basis. Removal and safe storage of the fuel cylinder is recommended when there will be a long interval before the next use, or when the instrument must be forwarded to or returned from a test location.

4) Remove the fuel cylinder as follows:

a) Loosen the connection between the fuel cylinder valve and the fuel cylinder fitting by hand.
b) Slide the cylinder out of the carriage below the housing.
c) Safely store the fuel cylinder until the next use.
d) The o-ring may fall out of the fitting. Remove the o-ring from the fuel cylinder fitting and inspect it for wear. If no wear is evident, retain the o-ring. Replace the o-ring if it is badly worn.

4.9.3 Maintenance

1) To recharge the batteries, connect the Sensidyne battery charger plug to the signal output (Line)/battery charger plug on the FID.

2) Plug the battery charger power cord into a wall outlet. The "power on" and "fast charge" indicators on the battery charger will illuminate.

3) When the FID battery is completely recharged, the fast charge indicator will extinguish, and the battery charger will automatically switch to slow charge.

4) Unplug the battery charger from the power supply or the wall outlet. Disconnect the battery charger from the unit.

Refer to operation and service manual for trouble shooting and replacement part procedures.

4.10 MSA MODEL 360 CARBON MONOXIDE, COMBUSTIBLE GAS & OXYGEN INDICATOR

Ambient air monitoring at hazardous waste sites is a common safety practice. Activity at a site may cause disturbances that release hazardous vapors into the ambient air. These releases can be detected by commercially available portable air monitoring devices that register real-time data. This data can be used to establish the existence of hazards such as oxygen deficient or explosive atmospheres. Personnel protective levels may be based on these readings.

The combustible gas sensor of the Model 360 is designed to measure combustible gas or vapor content in air. It will not indicate the combustible gas content in an inert gas background, furnace stack, or in other atmospheres with less than 10 percent oxygen. Further, these instruments should not be used where the oxygen concentrations exceeds that of fresh air (oxygen enriched atmospheres) because the extra oxygen makes any combustible mix easier to ignite and, thus, more dangerous.

Combustible gases will burn or explode only when the fuel/air mixtures are within certain proportions. The minimum concentration of a particular combustible gas in air which will burn and continue to burn when ignited is defined as the lower explosive limit (LEL). The maximum concentration that can be ignited is defined as the upper explosive limit (UEL).

A small pump pulls the atmospheric sample through a filter and pushes it through the flow indicator and the manifold blocks in which the toxic gas, combustible gas and oxygen sensors are mounted. The flow is then exhausted to the side of the case. The approximate flow rate is 1.5 liters/minute.

It is very important that every work area be appraised by someone trained in hazard control to make sure that the correct instrument is chosen and to determine whether other instruments are necessary to access the hazard. Evacuate all personnel from the area if the instrument the alarm indicates the possibility of a hazardous condition.

4.10.1 Calibration

1) To establish a zero background reading, the explosimeter should be prepared for operation in an area known to be free of combustible gases and vapors. A flush of fresh air should be passed through the instrument to zero the meter needle.

2) Prior to each day's usage, sensitivity must be tested on a known concentration of each of the gases for which the instrument is calibrated. The indication must be equal to or higher that the actual concentration.

3) Calibration should be checked and adjusted in fresh air at the elevation where the instrument is to be used.

4) Attach the flow control to the 0.75% pentane/15% oxygen calibration gas tank to the instrument. Within 30 seconds, the LEL readout should stabilize and indicate between 47 and 55%. If the indication is not in the correct range, remove the right end of the indicator and adjust the LEL SPAN control to obtain 50%.

5) Verify the oxygen reading; it should be between 13 and 17%.

6) Attach the instrument to the 300 ppm carbon monoxide calibration gas tank. After approximately two minutes, the TOX readout should stabilize and indicate between 275 and 325 ppm. If the indication is not in the correct range, remove the right end of the indicator and adjust the TOX SPAN control to obtain 300 ppm.

4.10.2 Operation

1) Remove the carrying handle by loosening both knurled screws and spreading the handle beyond the collars. Lift the handle from the screw shafts. Remove the left side of the case (the end with the threaded inlet fitting) by removing the four mounting screws. Allowing left side to remain attached to the top panel (via tubing and charge jack wires) slowly slide the top panel from the case until the plastic connectors for the battery and speaker are accessible. Disconnect these connectors.

2) Slide the top panel completely off the remainder of the case. Open the protective bag containing the oxygen sensor and install the O-ring onto the threaded end of the sensor. Firmly screw the sensor into the manifold by hand so as to compress the O-ring and thereby prevent sample flow leakage.

3) Push the connectors onto the two terminals at the top of the sensor (gray to positive and yellow to negative).

4) Remove toxic sensor manifold from chassis by removing the two screws from the back chassis. Remove the plastic cap and packaging material and discard. Make sure the sintered metal disc is in place in the manifold. Firmly screw the sensor onto the manifold by hand so as to compress the O-ring and thereby prevent sample flow leakage.

5) Reinstall the flowblock onto the chassis and connect the mating electrical connectors. Reassemble the case by sliding the top panel back into position and reconnect the speaker and battery pack.

6) Place the case side in position and install the lid hinge pins into each side. Install the mounting screws.

7) Turn the FUNCTION control to the HORN OFF position; the HORN OFF indicator will light and the descriptor %LEL will show in the readout. Set the readout to zero by adjusting the LEL ZERO control. This step must be done within 30 seconds of turning the instrument on to prevent the possibility of activating the off-scale, LEL latching alarm.

8) Press the SELECT button firmly to obtain %OXY on the readout; then set the readout to 20.8% by adjusting the OXY CALIBRATE control.

9) Press the SELECT button firmly to obtain PPM TOX on the readout; then set the readout to zero by adjusting TOX ZERO control.

10) Press the RESET button.

11) Turn the FUNCTION control to MANUAL for continuous readout of any one gas or to SCAN for automatic scanning of the three gas readings.

12) Check the flow system and sample line for leaks by placing a finger over the sample inlet fitting or the end of the sample line. Observe that the FLOW indicator float drops, indicating no flow.

4.10.3 Maintenance

1) After each days of use the battery should be charged for a minimum of 14 hours. The descriptor BATT will appear in the readout when the battery voltage is too low. In addition, when operating in the ON position, the audible alarm activates when the battery voltage drops to a low level, indicating that recharging is necessary. Always recharge instrument in a non-hazardous location to prevent the potential ignition of combustible atmospheres. Do not operate the instrument while the battery is being charged. For optimum battery service, once a month the battery should be charged for 16 hours, run for 8 hours and then fully charged for 24 to 36 hours.

   When the rechargeable, 4.0-volt lead-acid battery pack no longer responds to recharging or no longer holds a charge, replace the pack by loosening the knurled screws holding the handle and remove the handle. Looking at the front panel of the instrument, remove the right (audible alarm side) panel by unscrewing the four side panel screws. Gently pull the side panel loose and tilt the instruments to help slide out the battery case. Disconnect

the molded nylon plug from the battery case. Do not disconnect the alarm speaker. Install the new battery by reversing the above steps.

2) Care should be taken when sampling over solids and liquids so that it is not drawn into the instrument. The sample inlet filter should be examined each time the instrument is recharged. If the filter element appears to be coated with dust or dirt, it should be washed, dried and reinserted or a new element substituted. Make sure that the inlet seal O-ring in the inlet filter cap is properly seated. If the O-ring is damaged or missing, the O-ring must be replaced before using the instrument.

4.10.4 Troubleshooting Guidelines

Consult the manufacturer's instruction manual for details.

4.11 GasTechtor Portable Carbon Dioxide / Oxygen Indicator Model 3252OX

The Gastechtor portable carbon dioxide (CO_2) / oxygen (O_2) detection instrument is designed to determine CO_2 and O_2 content in the air or in a vapor sample. The range of CO2 detected is between 0 and 5%. Oxygen is detected at ranges between 0 and 25%.

Samples are drawn through a hose by means of a built-in pump and analyzed for CO2 in a simplified NDIR (Non-Dispersive Infrared) cell and oxygen using an electrochemical cell. Solid-state amplifiers are used to amplify indications of the elements to give adequate voltage to drive the meter and the alarm circuits.

4.11.1. CO_2 Calibration

To calibrate for CO_2, the range switch should be in the CO_2 OUT position.

1) Turn the instrument on and allow it to warm up and stabilize (approximately 5 minutes). Be sure that the batteries are charged sufficiently and adjust zero to give a reading of 0.05% if the setting is fresh air, or 0.0 if detector is exposed to a known CO_2-free sample.

2) Open instrument case and locate the CO_2 SPAN potentiometer on underside of circuit board.

3) Connect instrument inlet to a known calibrating gas sample. Adjust meter to match the known CO_2 concentration of the sample when the meter reaches it's maximum reading. Adjustments can be made by turning the SPAN potentiometer using a small screwdriver.

4.11.2 O_2 Calibration

To calibrate for O2, the range switch should be in the OXY IN position.

1) With instrument open, identify the oxygen zero potentiometer in the rear of the oxygen circuit board.

2) Connect instrument to a known oxygen free sample such as nitrogen, argon, or helium.

3) Adjust the meter to zero by turning the ZERO potentiometer using a small screwdriver

4) After zero adjustment is completed, return probe inlet to normal atmospheric air. Readjust OXY CAL control as necessary to bring meter reading to 21%.

4.11.3 Operation

1) Connect probe and hose to fitting on front of instrument.

2) Press POWER switch to turn instrument on, with orange indicator dot showing.

3) With range select switch in CO2 (OUT) position, press BATT CK button and note meter reading. If reading is below BATT CK mark on meter, turn off instrument and recharge batteries.

4) Allow to warm up in CO2 range until meter stabilizes (about a minute). With probe inlet in a normal air location, turn CO2 ZERO shaft to bring meter to approximately 0.05%.

5) Next put range switch in OXY (IN) position. Verify that probe is in a normal -air location; then turn OXY CAL control to bring meter to 21% (CAL) indication.

6) Verify normal operations by breathing out through your mouth and letting the probe sample your expired breath. Oxygen should move downscale and activate the alarm at 19.5%. In CO2 range, reading should come up to about 2.5%. Both alarm lights and a steady audible alarm tone should come on during this test.

7) To take a reading, select meter range with range switch either in CO2 or O2 position. Place tip of probe in contact with the sample to be tested and watch meter.

4.11.4 Maintenance

1) Check battery voltage periodically by pressing BATTERY CHECK switch. This check must be done while range switch is in CO2 OUT position. Recharge before voltage reaches minimum.

2) When connecting the charger, confirm that the plug is inserted in the correct way, with the THIS SIDE UP label upwards. The socket is polarized, with the pins offset below the centerline, but can sometimes be forced on the wrong way, particularly if it has become worn with use.

3) Verify that a charge is actually entering the battery by confirming that the amber light is on. Leave connected until the green light comes on indicating that the 16 hour charge is completed.

4.11.5 Troubleshooting Guidelines

Consult the manufacturer's instruction manual for details.

4.12 Trace-Techtor Portable Hydrocarbon Vapor Tester

The portable Trace-Techtor is designed to detect hydrocarbon vapors over a broad range. The range can be set for 0-100 ppm, 0-1000 ppm, or 0-10,000 ppm with or without a methane response.

The following is a summary of the manufacturer's manual. For additional information concerning the Trace-Techtor portable hydrocarbon vapor tester, consult the instruction manual.

4.12.1. Calibration

The Trace-Techtor should be calibrated periodically depending on the frequency of usage. To insure data accuracy, this meter should be checked daily prior to use in the field. Hexane is the factory recommended calibration gas since it provides a conservative response representative of total petroleum hydrocarbon vapors present.

1) Turn the instrument on and allow a five minute warm up period. Verify that the battery is charged.

2) Open the instrument case and locate the potentiometer on the front corner of the circuit board marked PPM SPN. This is the span adjustment.

3) Attach hose, probe, and hydrophobic filter to the instrument as it would be in normal operation.

4) Turn to PPM range and zero the meter using the external ZERO adjustment knob. If zero cannot be adjusted with the external adjustment, use the internal coarse zero adjustment (see instruction manual).

5) Attach upper end of flowmeter to the probe with the short piece of tubing included in the calibration kit. Note flowmeter reading.

6) Attach valve to cylinder and flowmeter to valve with the remaining tubing. Open valve just enough so that flow is the same as observed in step 5.

7) Watch meter and note highest reading. The desired reading is the PPM value marked on the calibration gas cylinder. The selector switch should be in the appropriate position to read the concentration marked on the cylinder. If the reading does not match the cylinder value, turn PPM SPN adjustment to give desired reading.

Calibration kits and replacement cylinders are available from Gastech Environmental Monitors. The recommended cylinder is part number 81-0007E, which is a cylinder of nominal 40% LEL hexane marked with its PPM value, nominally 4400 PPM.

Calibrate the unit with a concentration in excess of 1000 ppm to minimize any calibration error that may occur due to humidity effects caused by the dry air which comes out of a compressed gas cylinder.

8) If zero cannot be adjusted, or if reading cannot be set high enough, replace detector.

4.12.2 Operation

1) Attach hydrophobic filter, hose and probe to the inlet fitting on front of instrument.

2) Turn rotary switch to BATT CK position and allow a 5 minute warm-up. Meter reading should be above the BATT CK mark on the meter. If close to or below this mark, recharge battery before use.

3) Any alarms heard during warm-up should be investigated. See instruction manual for troubleshooting guidelines.

4) Test the flow system by placing finger over inlet and verify that the low flow alarm activates. The inlet should be checked with all sampling accessories connected (hose, probe, hydrophobic filter), and finger placed over the probe tip. Allow a few seconds for flow alarm to activate when blocking inlet.

5) After a five minute warm-up, or when the reading in the PPM range has stabilized, adjust the external zero knob to obtain a "0" reading. This must be done with the selector switch in the PPM range and with the probe sampling from a gas free location. If impossible to adjust the zero within the range of the external zero adjust potentiometer, adjust internal coarse zero adjustment.

6) Turn selector switch to desired range and hold hose inlet at point to be tested. Watch meter and note highest reading obtained. If meter reads over full scale, then move selector switch to the nest position to change range to a less sensitive one. If reading rises above the alarm set point, a pulsed audible alarm will start and will continue as long as reading remains above alarm point. After completing readings, purge instrument with fresh air before turning off.

4.12.3 Maintenance

1) Check battery voltage periodically by turning the selector switch to BATT. Recharge before voltage reads minimum.

2) To charge battery, turn the selector switch to BATT position and note meter reading.

3) Plug charger into power source and then into the socket on the rear of the Trace-Techtor. The meter reading should rise slightly as soon as the

charger is connected. If it does not, verify that the AC outlet is active. If outlet is active but meter does not rise when charger is attached, then the charger may be defective. If meter reading does rise, turn instrument off and leave on charge for 16 hours.

4.12.4 Troubleshooting Guidelines

Consult the manufacturer's instruction manual for details.

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6.0 ATTACHMENTS

Equipment Calibration Form

EQUIPMENT CALIBRATION FORM

Project: _____ Project Number: _____
Personnel: _____ Date: _____
Weather: Clear _____ Rain _____ Snow _____ Temperature _____ Humidity _____

INSTRUMENTATION:
OVA _____ CO_2/O_2 _____ TVH _____ Other _____

CALIBRATION:

Date	Time	Calibration Standard	Calibration Standard Concentration (ppm)	Meter Reading	Comments

CALIBRATION CHECK:

Date	Time	Calibration Standard	Calibration Standard Concentration (ppm)	Meter Reading	Comments

MAINTENANCE AND/OR REPAIR:

Date	Problem	Return to Mfg. for Repair	In-House Repair	Type of * Maintenance or Repair	Effective	Comments

* *Does not include charging batteries.*
Comments should include whether or not it was a warranty repair, date equipment was repaired or was received from the manufacturer, and any other information you feel would be useful.

Note: If you run out of space in the maintenance and repair section please write on the back.

Appendix E
Attachment 1

Management Review / Other
Technical Review / Project Manager
Graphic Standards / Lead Professional
QUALITY CONTROL

This document has been developed for a specific application and may not be used without the written approval of Montgomery Watson.

Developed By	Drawn By	EQUIPMENT CALIBRATION FORM	Drawing Number 4077.0191
Approved By	Date 8/12/96		
Reference		TREATABILITY STUDY AMERICAN CHEMICAL SERVICE, INC. NPL SITE GRIFFITH, INDIANA	MONTGOMERY WATSON
Revisions			

APPENDIX F

STANDARD OPERATING PROCEDURES FOR FIELD SAMPLING AND DOCUMENTATION

STANDARD OPERATING PROCEDURES

SAMPLING AND MONITORING PROCEDURES FOR ISVE SYSTEM OPERATION AND DATA COLLECTION

STANDARD OPERATING PROCEDURES

SAMPLING AND MONITORING PROCEDURES FOR ISVE SYSTEM OPERATION AND DATA COLLECTION

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1.0 INTRODUCTION

The following methods are specific to the pilot-scale ISVE system operation monitoring and data collection. The data from each of the parameters to be monitored will be used to assess the system operation and to provide design data for a full-scale ISVE system.

2.0 DEFINITIONS

ISVE: In-Situ Vapor Extraction

3.0 RESPONSIBILITIES

The following is a general description of responsibilities related to monitoring and sampling procedures for the ISVE pilot test.

Project Manager: The Project Manger is responsible for identifying the appropriate equipment necessary to adequately define the parameters.

On-Site Safety Officer: The On-Site Safety Officer will work with the Project Manager in identifying the appropriate safety equipment. The On-Site Safety Officer will also perform audits to observe field personnel during sampling and monitoring. If the safety equipment is not being used properly, the On-Site Safety Officer will provide the necessary training and resources for safety of sampling activities.

Field Team Leader (FTL): The FTL will be responsible for including a refresher course as part of the kick-off meeting on the proper sampling procedures including the use, calibration, and maintenance of all equipment to be used on the project. The FTL will ensure that all field team members continue to properly conduct sampling and monitoring and to properly use the equipment through the duration of the project. During periods of short absences, the project manager or the FTL will assign appropriate personnel to function as their representative.

Field Team Members: Prior to mobilization, a field team member will be required to check out equipment or supplies requested for the project and sign an equipment checklist form stating that all equipment is functioning, can be calibrated, is in good condition, all the

necessary parts are included in the case, and all supplies requested are present. The warehouse manager and/or assistant warehouse manager will also be responsible for signing the equipment checklist. Upon demobilization, the checklist will be used to check the equipment back into the warehouse. At that time, all equipment will be calibrated, if applicable, and checked for proper use by a field team member and the warehouse manager and/or the assistant warehouse manager. Both the field team member and the warehouse manager and/or warehouse assistant manager will be required to sign the checklist.

The field team members will be responsible for carefully reviewing the SOPs and instruction manuals for the procedures and equipment to be used on the project. All field team members will be required to sign a form indicating that they know how to properly operate each piece of equipment that will be used on their project.

This SOP provides a summary of the sampling and monitoring procedures that are to be used in the field for the ISVE system operation and data collection. This SOP should be reviewed prior to field activities by all of the field members.

4.0 GUIDELINES

4.1. Vapor Extraction Flow Rates

Each extraction well and extraction trench system will have an air flow meter positioned in line between the vapor extraction well head (or extraction trench manifold) and the condensate collection system. Additionally, the air injection wells for the air sparging system will have flow meters positioned between the air injection blower and the well head. At the scheduled monitoring round, the flow rates will be read from each flow meter on the ISVE or ISVE/air sparging system and recorded on the appropriate field form. The time and date of the meter readings and the ambient weather conditions (dew point, barometric pressure, humidity, and temperature) will be recorded on the appropriate field form and in the field logbook. Any indications that a flow meter is not operating correctly will warrant immediate installation of a replacement meter.

4.2. Vacuum/Pressure Measurements

Each vapor extraction well head, vapor extraction trench manifold (outlet), and inlet and outlet of the blowers, will be equipped with vacuum gauges. Additionally, a vacuum gauge equipped with quick connect/disconnect fittings will be used to monitor the vacuum/pressure at each vapor monitoring probe. During each scheduled monitoring round, the vacuum/pressure reading on the ISVE and ISVE/air sparging systems and from each vapor monitoring probe, will be recorded on the appropriate field form. The time and date of the vacuum/pressure readings and the ambient weather conditions (dew point, barometric pressure, humidity, and temperature) will be recorded on the appropriate field form and in the field logbook. Any indications that a vacuum gauge is not operating correctly will warrant immediate installation of a replacement gauge.

4.3. Condensate Sampling and Volume Measurements

The condensate collection system is equipped with a knockout drum with a drain valve located on the bottom of drum. Condensate generated from the extracted vapors will be collected in the knockout drum. The knockout drum will also be equipped with a sight glass (level indicator) so that the volume of condensate in the drum can be determined by looking at the level of liquid in the drum and reading off the corresponding volume.

4.3.1. Sampling. Condensate samples will be collected bi-weekly according to the sampling schedule as stated in the Field Sampling Plan. Prior to collecting the condensate samples, the appropriate sample containers will be prepared and labeled as outlined in the QAPP. The drain valve located on the bottom of the knockout drum will be opened and allowed to drain into a bucket until the condensate draining from the drain valve is representative of the bulk volume of the condensate. The flow of condensate from the sampling port will then be adjusted to a trickle and VOC and SVOC samples will be collected in the appropriate sample containers. The sample container will be filled such that zero head space remains in the container. Immediately after a sample container is filled, the container will be capped, sealed, and placed in a cooler maintained at 4 degrees C. The samples will be packed, handled, and shipped to the appropriate laboratory as outlined in the QAPP. The condensate collected in the bucket will be disposed in accordance with the waste management plan for extracted ground-water from the site.

4.3.2. Volume Measurements. The volume of the condensate collected in the knockout drum will be recorded during each monitoring round or prior to draining. The volume measurement will be taken directly from the measurement listed on the side of the drum that corresponds to the liquid level in the drum and recorded on the appropriate field form and in the field logbook. The date and time of the measurement and any other observations regarding the condensate will also be recorded on the appropriate field form and in the field logbook.

4.4. Vapor Sample Collection

Vapor samples will be collected in Tedlar bags or Summa canisters. Tedlar bags will be used to collect vapor samples for field CO_2, O_2, and total volatile hydrocarbon measurements. Vapor samples collected for off-site analysis will be collected in either Tedlar bags or Summa canisters.

4.4.1. Field CO_2, O_2, and Hydrocarbon Measurements. All field samples will initially be collected in Tedlar bags either directly from the sampling ports at the extraction well heads or the vapor extraction trench manifold or by pumping from the vapor monitoring probes using a sampling vacuum pump. The field measurements of CO_2, O_2, and total hydrocarbons will initially be collected in a Tedlar bag. Collecting vapor samples directly from the vapor sampling port at the extraction well head would not be a true representation of the vapor concentrations due to forced air flow into the analyzer from the ISVE system blower. Additionally, each vapor monitoring probe will be purged with a vacuum pump prior to sample collection. This will insure that the sample is representative of the formation surrounding the vapor probe screen. Each sampling port will have dedicated Teflon tubing extending from the sampling port with quick connect/disconnect fittings. All field equipment will be calibrated prior to sampling on the same day in accordance with the field equipment calibration SOPs in Appendix F.

The following procedures will be used to collect each vapor sample for field measurements from the extraction well heads or from the vapor extraction trench manifold:

- Connect the Tedlar bag to the regulator on a nitrogen gas cylinder and open the valve on the Tedlar bag.
- Slowly open the regulator valve and allow the Tedlar bag to become partially filled.

- Close the valve on the regulator.
- Deflate the Tedlar bag by pushing on each side of the bag to complete a nitrogen purge of the Tedlar bag.
- Connect the Tedlar bag to the extraction well head vapor sampling port.
- Slowly open the valve on the vapor sampling port until the Tedlar bag becomes partially inflated.
- Close the vapor sampling port valve.
- Close the valve on the Tedlar bag.
- Connect the tubing from the Tedlar bag to the field instrument.
- Open the valve on the Tedlar bag.
- Allow the meter to equilibrate. Record the measurement from the meter, time, date, and the ambient air conditions (barometric pressure, dew point, humidity, temperature) on the appropriate field sampling log and in the field logbook.

The following procedures will be used to collect each vapor sample for field measurements from the vapor monitoring probes:

- Purge the dedicated Tedlar bag with nitrogen using the steps outlined above.
- Connect the sampling pump to the vapor monitoring probe using Teflon tubing and quick connect/disconnect fittings. There should be a moisture trap connected in line between the pump and the vapor monitoring probe connection.
- Turn the pump on and begin purging the vapor monitoring probe tubing. Each probe should be purged until five vapor probe tubing volumes have been evacuated.
- Lower the flow rate on the pump.
- Connect the Tedlar bag to the discharge side of the pump and open the valve on the Tedlar bag.
- Slowly increase the flow rate on the pump until the Tedlar bag becomes partially inflated.
- Turn the pump off and close the valve on the Tedlar bag.
- Connect the tubing from the Tedlar bag to the field instrument.
- Open the valve on the Tedlar bag.
- Allow the meter to equilibrate. Record the measurement, time, date, weather conditions, etc. on the appropriate field sampling log and in the field log book.

4.4.2. Vapor Sample Collection for Laboratory Analysis. Vapor samples to be sent off-site for laboratory analyses will be collected in either Tedlar bags or Summa canisters. Each sample collected will be properly labeled, handled, and shipped according to the procedures outlined in Section 3.0 of the QAPP.

The procedures for collecting field measurements will also be used for collecting laboratory samples. Each sample will first be analyzed for total volatile hydrocarbons in the field using the Trace-Techtor portable hydrocarbon vapor tester connected to the Tedlar bag. The meter reading will be recorded on the appropriate field sampling form and in the field logbook. Another Tedlar bag will then be purged using nitrogen and partially filled for the laboratory

sample. If Summa canisters are used, the sample can be drawn directly from the sampling ports or vapor probe tubing after the field measurement is taken from the Tedlar bag.

All samples will be labeled, handled, and shipped to the appropriate analytical laboratory using the procedures outlined in Section 3.0 of the QAPP.

Note that during the air sparging test conducted without the ISVE system in operation in the ONCA, samples will still be collected from the vapor extraction trench manifold. For purposes of collecting samples representative of the trenches, the ISVE blower will be started up for the duration of sample collection only. The samples will be collected from the trench manifold sampling port as stated in the above procedures.

4.5. Water Level Measurements

Groundwater level measurements will be made at each of the piezometers and groundwater monitoring wells as scheduled in the FSP. Additionally, the water level from within the deeper vapor extraction well will be measured during the scheduled round of groundwater level measurements. Each measurement will be made using either a dual-phase probe (oil-water interface probe) or a single phase, reel type, electric water level indicator. Groundwater levels in monitoring wells or piezometers known to contain free-phase hydrocarbons will be measured using the dual-phase probe. The procedures for water level measurements and operation of the water level probes are detailed in the SOP for Operating and Calibration Procedures for Field Equipment in Appendix F.

STANDARD OPERATING PROCEDURES

CALCULATIONS FOR AIR PERMEABILITY

APPENDIX

RECOMMENDED ESTIMATION METHODS FOR AIR PERMEABILITY

The U.S. Environmental Protection Agency's Risk Reduction Engineering Laboratory recently reviewed several field, laboratory, and empirical methods for determining soil gas permeability (k) and for their appropriateness in determining the feasibility of soil vapor extraction (Sellers and Fan, 1991). The conclusion of this literature review was a strong endorsement for a modified field drawdown method (Johnson et al., 1990).

The field drawdown method is based on Darcy's Law and equations for steady-state radial flow to or from a vent well. A full mathematical development of this method and supporting calculations are provided by Johnson et al. (1990). A computer program known as HyperVentilate™ has been produced by Johnson for storing field data and computing k and R_t. This program will be used to speed the calculation and data presentation process. The two solution methods for k are presented below. The first solution is based on carefully measuring the dynamic response of the soil to a constant injection or extraction rate. The second solution for k is based on steady-state conditions and the measurement or estimation of R_t at steady state. The limitations and recommended application of each method are presented below. Whenever possible, field data will be collected to support both solution methods, because one or both of the solution methods may be appropriate, depending on site-specific conditions.

Dynamic Method

This test method requires that air be extracted or injected at a constant rate from a single venting well, while measuring the pressure changes at several soil gas monitoring points throughout the contaminated soil volume. The equation:

$$P' = \frac{Q}{4\pi\, m(k/\mu)} \cdot \frac{[-0.5772 - \ln(r^2 e\mu) + \ln(t)]}{4k\, Patm} \qquad (1)$$

is used to describe the dynamic changes in soil gas pressure/vacuum where:

P' = "gauge" pressure measured at distance r from the vent well at time t ($g/cm\text{-}s^2$)

m = stratum thickness, generally the vent well screened interval (cm)

r = radial distance from monitoring point to vent well (cm)

k = soil gas permeability (cm^2)

μ = viscosity of air (1.8×10^{-4} g/cm-s at 18°C)

e = soil's air-filled void volume (dimensionless)

t = time from the start of the test (s)

Q = volumetric flow rate from the vent well (cm^3/s)

Patm = ambient pressure (at sea level 1.013×10^6 $g/cm\text{-}s^2$)

Equation (1) predicts that the dynamic range of P′-vs.-ln(t) is a straight line with a slope of A where:

$$A = \frac{Q}{4\pi m\,(k/\mu)}$$

solving

$$k = \frac{Q\mu}{4A\pi m}$$

The HyperVentilate™ model is based on the dynamic method and a determination of the slope, A. This method of determining k requires accurate field measurements of Q at the vent well and P′s-vs.-time at each monitoring point. It is most appropriately applied at sites with less permeable soils where changes in P′ occur over a longer time period (10 minutes or more to monitoring point steady state). This method can be accurate for fine sandy soils where the screened interval extends to depths of over 10 ft and when monitoring points are screened at depths of 10 ft or greater. It is less accurate for sites where a high water table or shallow contamination limits the total depth of the vent well screen and monitoring points to less than 10 ft. In shallow and coarse-grained soils, vacuum or pressure levels reach steady state too rapidly to accurately plot P′-vs.-ln(t). Venting systems on shallow sandy sites are subject to higher vertical airflow which is not as accurately described by this one-dimensional radial flow equation.

Steady State-Method

This method for determining k can be used in situations where the dynamic method is inappropriate. This method is based on the steady-state solution to equation (1).

$$k = \frac{Q\mu \ln(Rw/R_I)}{H\pi\, Pw\,[1 - (Patm/Pw)^2]} \qquad (2)$$

Note: Equation (2) applies only to vent wells operating under a vacuum. If air is being injected into the vent well the equation is modified as shown below:

$$k = \frac{Q\mu \ln(Rw/R_I)}{H\pi\, Patm\,[1 - (Pw/Patm)^2]} \qquad (3)$$

where Q, m, μ, and Patm have been previously defined, and

Rw = the radius of the venting well (cm)

H = depth of screen (cm)

R_I = the maximum radius of venting influence at steady state (cm)

Pw = the absolute pressure at the venting well ($g/cm\text{-}s^2$)

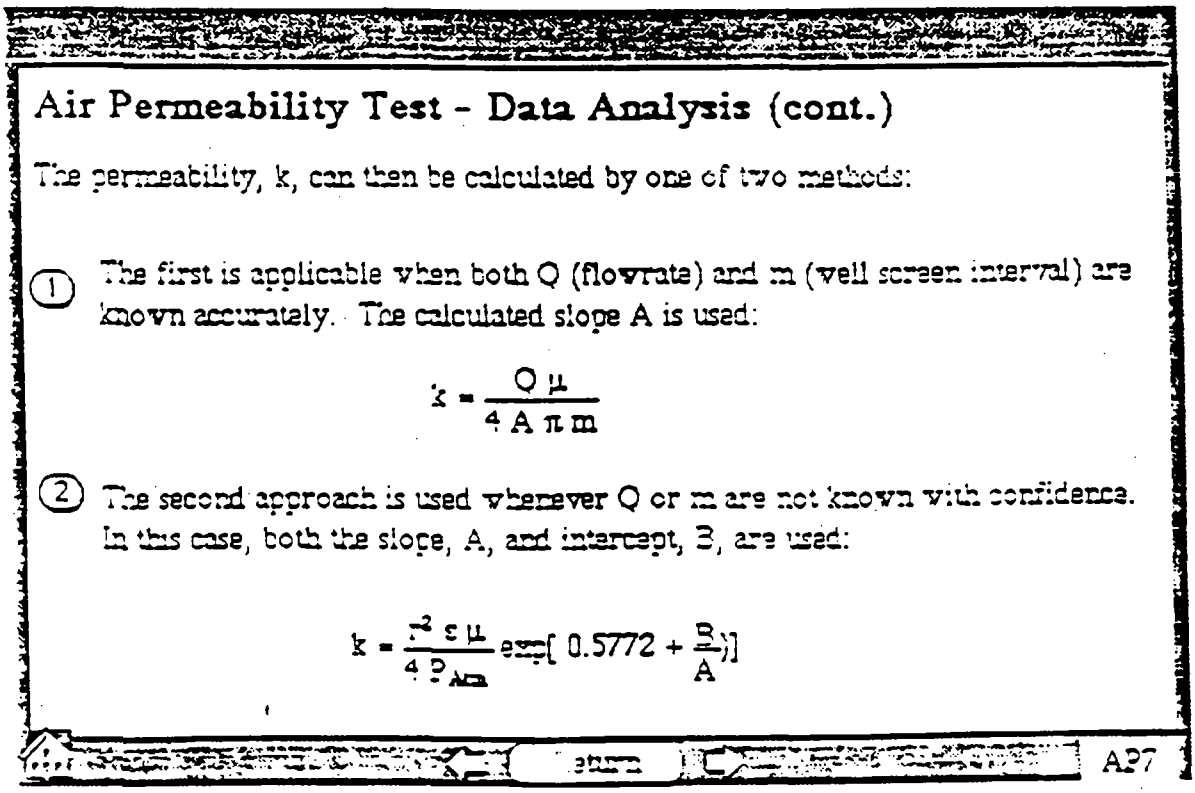
The value of R_I can be determined by actually measuring the outer limit of vacuum/pressure influence under steady-state conditions, or by plotting the vacuum/pressure at each monitoring point vs. the log of its radial distance from the vent well and extrapolating the straight line to zero vacuum or pressure. An example of this solution method is included in Calculation Data Set Two below.

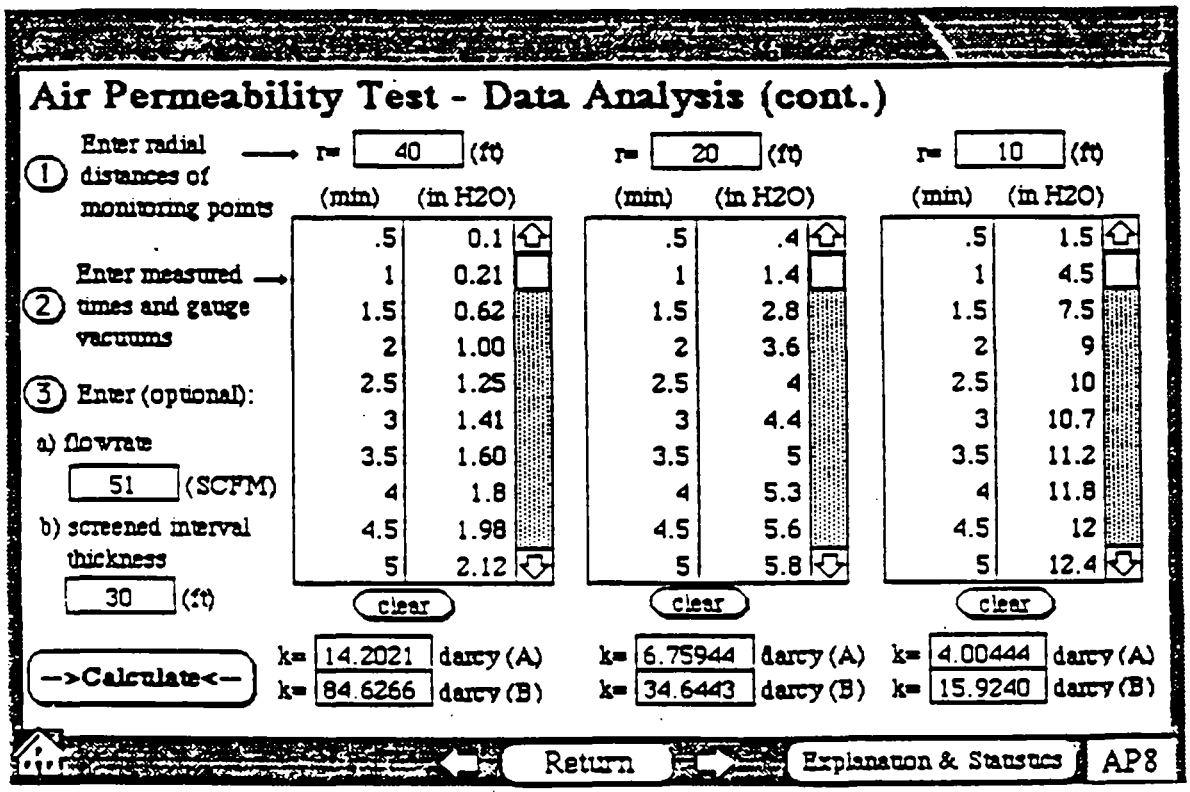
Sample Calculations

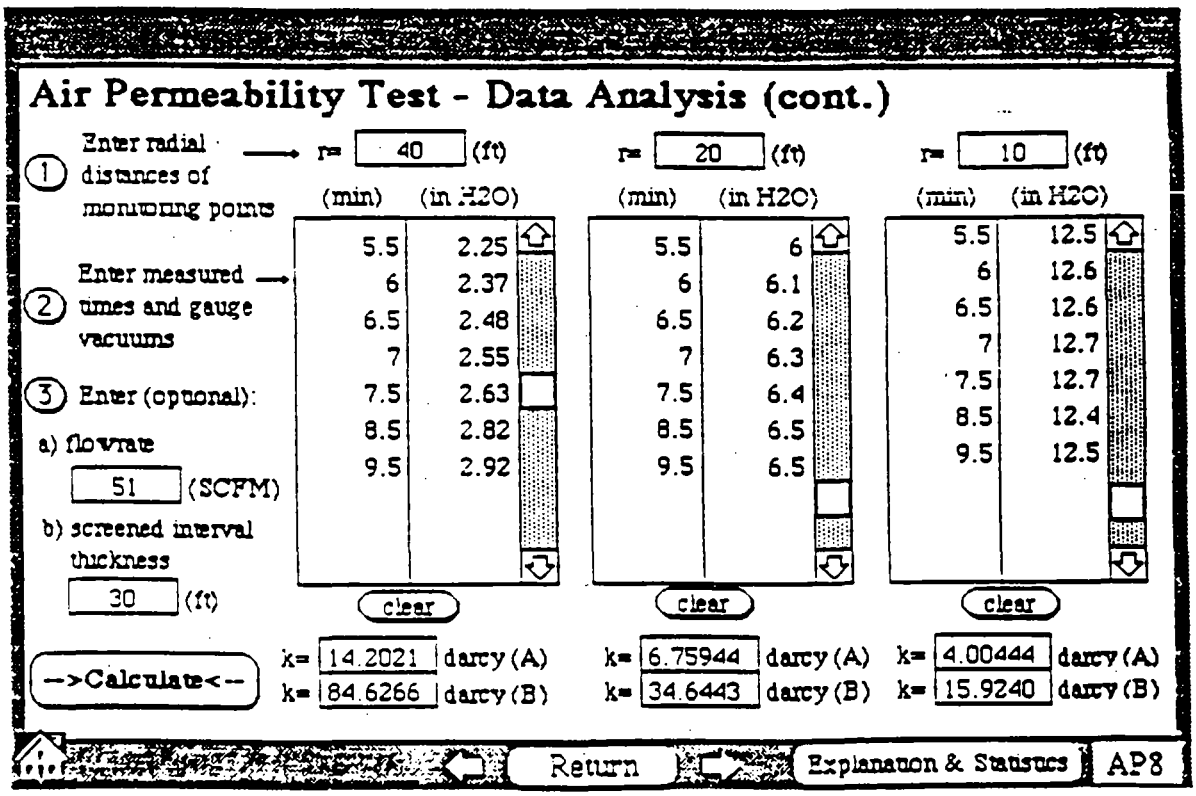
Data Set One

Table A-1 and Figure A-1 present the results of an air permeability test conducted at Beale AFB, CA. The soils on this site were silty with a contaminated interval (and vent well screen interval) extending from 10 to 40 feet below ground surface. Note that the plot of P'-vs.-ln(time) is a relatively straight line during the initial 10 minutes. ln (10) = 2.3, making these data good candidates for the dynamic solution method. Data from the initial 10 minutes of this test were entered into the Hyper-Ventilate™ computer model to calculate a range of k values. An example of the input and output data for this model is provided in windows AP7 and AP8.

HyperVentilate© 1991







HyperVentilate© 1991

TABLE A-1. Air ...meability Data Set

Steady-State Flow Rate 51 SCFM

Test Time Elapsed (min)	In Time (min)	Vacuum (inches of water) at Monitoring Points (MPs) MP 1	MP 2	MP 3	MP 4	MP 5	MP 6	MP 7	M	MP 9
0.0	–	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00
0.5	–	–	–	–	–	–	–	0.10	0.40	1.50
1.0	0.00	–	–	–	–	–	–	0.21	1.40	4.50
1.5	0.41	–	–	–	–	–	–	0.62	2.80	7.50
2.0	0.69	–	–	–	–	–	–	1.00	3.60	9.00
2.5	0.92	–	–	–	–	–	–	1.25	4.00	10.00
3.0	1.10	–	–	–	–	–	–	1.41	4.40	10.70
3.5	1.25	–	–	–	–	–	–	1.60	5.00	11.20
4.0	1.39	–	–	–	–	–	–	1.80	5.30	11.80
4.5	1.50	–	–	–	–	–	–	1.98	5.60	12.00
5.0	1.61	–	–	–	–	–	–	2.12	5.80	12.40
5.5	1.70	–	–	–	–	–	–	2.25	6.00	12.50
6.0	1.79	–	–	–	–	–	–	2.37	6.10	12.60
6.5	1.87	–	–	–	–	–	–	2.48	6.20	12.60
7.0	1.95	–	–	–	–	–	–	2.55	6.30	12.70
7.5	2.01	–	–	–	–	–	–	2.63	6.40	12.70
8.5	2.14	–	–	–	–	–	–	2.82	6.50	12.40
9.5	2.25	–	–	–	–	–	–	2.92	6.50	12.50
10.5	2.35	–	–	–	–	–	–	2.96	6.50	12.50
14.0	2.64	–	–	–	–	–	–	3.00	6.50	12.40
19.0	2.94	–	–	–	–	–	–	3.05	6.40	11.90
24.0	3.18	–	–	–	–	–	–	3.10	6.20	11.00
29.0	3.37	–	–	–	–	–	–	3.37	6.00	10.40
34.0	3.53	–	–	–	–	–	–	3.40	5.80	9.90
39.0	3.66	–	0.8	0.4	0.7	2.2	1.7	3.40	–	–
44.0	3.78	0.3	–	–	–	–	–	–	–	–
								40	20	10	<-- Distance from VE = 2
		27.5–29.5	18–20	13–15	14–16	38–40	30–32	38–40	38–40	38–40	<-- Screen interval depth

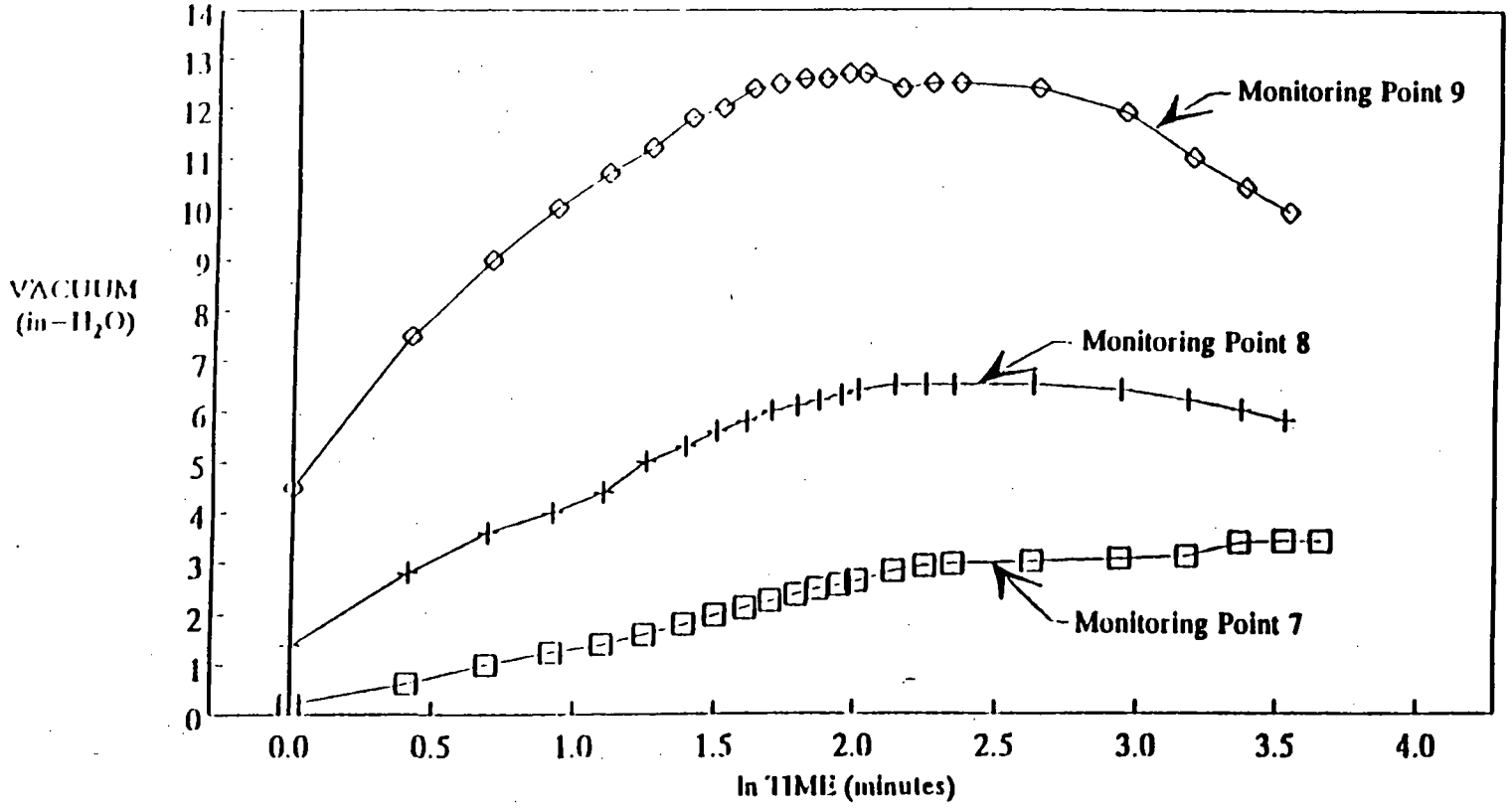


Figure A-1. Vacuum vs. ln Time, Test 2, Bioventing Pilot Test, Site 22-A20, Beale AFB, California

Computer window AP7 provides a summary of two mathematical solutions for air permeability (k) using the dynamic method. Window AP8 is the example data entry and solution sheet. The calculated range of k values for this test is shown at the bottom of window AP8. Permeability values of 4 to 14 darcy are based on Equation 1 in window AP7 and provide the most accurate estimate, because both the extraction rate (Q) and the screened interval (m) were known for this test. The more conservative range of 4 to 14 darcy will be used for full-scale design. These air permeability values are approximately one order of magnitude higher than would be expected for silty soils. The presence of 10 to 15% sand (by weight) in this soil has increased the average permeability at this site.

Data Set Two

Table A-2 and Figure A-2 are the results from a test conducted in a silty loam with a contaminated interval of only 5.2 ft and a screened interval from 2.7 to 5.2 ft below ground surface. Note that the almost immediate steady state reached at this site does not produce the P'-vs.-ln(time) plot required for the dynamic solution method. In this case the steady-state solution offers the only approximation of k and R_I.

$$k = \frac{Q\mu \ln(Rw/R_I)}{H\pi Pw\,[1 - (Patm/Pw)^2]}$$

For this test:

$Q = 1.4 \times 10^{-4}$ cm^3/s

H = 2 ft (61 cm)

$\mu = 1.8 \times 10^{-4}$ g/cm-s

$Pw = 80"H_2O$ vacuum $\times 3.61 \times \frac{10^{-2}\ psia}{"H_2O} = 2.88$ psia

Pw absolute = 14.7 psia – 2.88 psia = 11.82 psia

$$11.82\ psia \times 6.9 \times \frac{10^4 g/cm\text{-}s^2}{psia} = 8.16 \times 10^5 g/cm\text{-}s^2$$

$Patm = 1.01 \times 10^6 g/cm\text{-}s^2$

Rw = 1 in. = 2.54 cm

R_I = ~15 ft (457 cm) based on all monitoring points reported in Table A-2

TABLE A-2. Field Test Data for Soil Determination of Soil Permeability at a Gasoline-Contaminated Site

Time (min)	Air Flow (cfm)	Vacuum (inches of water) measured at various monitoring points										
		Unit	Well	F	E	G	D	H	C	I	B	A
0.0	0	0	2	0.10	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00
0.5	30	109	80	1.90	0.90	0.25	0.15	0.00	0.00	0.00	0.00	0.00
1.5	30	109	80	1.90	0.90	0.30	0.20	0.05	0.00	0.00	0.00	0.00
5.0	30	109	80	1.90	0.90	0.30	0.20	0.05	0.00	0.00	0.00	0.00
10.0	30	109	80	1.90	0.95	0.30	0.20	0.05	0.00	0.00	0.00	0.00
15.0	30	109	80	1.90	0.95	0.30	0.20	0.05	0.00	0.00	0.00	0.00
20.0	30	109	80	1.90	0.95	0.30	0.20	0.05	0.00	0.00	0.00	0.00
		Distance from well (ft)		3	6	9	12	15	18	21	24	27

Rw = 2.54 cm

H = 60.96 cm

Q = 14,158 cm^3/sec

$\mu = 1.8 \times 10^{-4}$ g/cm-s

Patm = 8.14×10^5 Dynes/cm^2

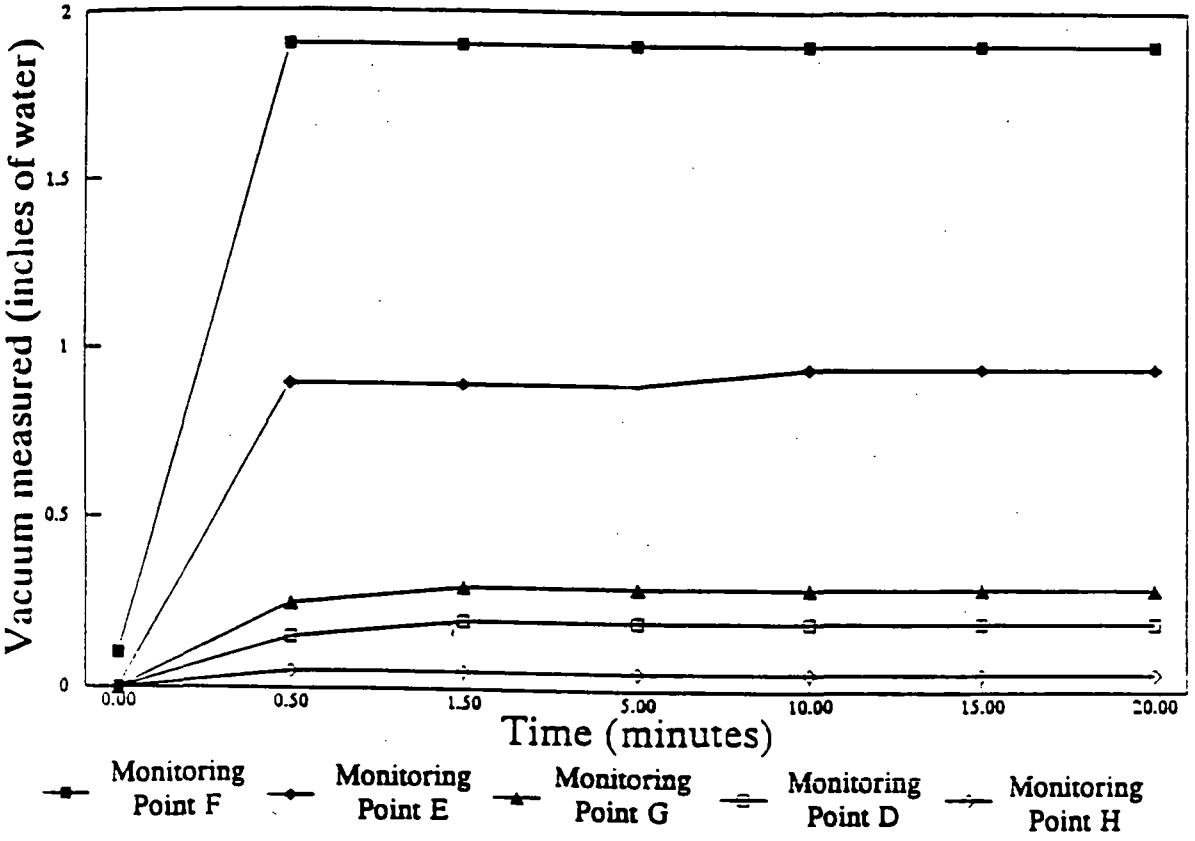


Figure A-2. Results of a Field Test to Determine Soil Permeability to Airflow, k, September 16, 1991

STANDARD OPERATING PROCEDURES

GROUNDWATER SAMPLING AND FIELD MEASUREMENTS

STANDARD OPERATING PROCEDURES

GROUNDWATER SAMPLING AND FIELD MEASUREMENTS

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1.0 INTRODUCTION

This guideline is a general reference for the proper equipment and techniques for groundwater sampling. The purpose of these procedures is to enable the user to collect representative and defensible groundwater samples and to facilitate planning of the field sampling effort. These techniques should be followed whenever applicable, although site-specific conditions or project-specific plans may require adjustments in methodology.

To be valid, a groundwater sample must be representative of the water quality of the aquifer being sampled. The physical, chemical, and bacteriological integrity of the sample must be maintained from the time of collection to the time of analysis in order to minimize changes in water quality parameters. Acceptable equipment for withdrawing samples from completed wells include bailers and various types of pumps. The primary considerations in obtaining a representative sample of the groundwater are to avoid collecting stagnant (standing) water in the well, to avoid physically or chemically altering the water due to improper sampling techniques, sample handling, or transport, and to document that proper sampling procedures have been followed.

This guideline describes suggested well evacuation methods, sample collection and handling, field measurement, decontamination, and documentation procedures. Examples of sampling and chain-of-custody (COC) forms are attached.

2.0 GENERAL WATER SAMPLING GUIDELINES

2.1 WELL EVACUATION AND SAMPLING EQUIPMENT

There are many methods available for well purging. A variety of factors must be considered when choosing evacuation and sample collection equipment including: the depth and diameter of the well, the recharge capacity of the well, and the analytical parameters that will be tested. Few sampling devices are suitable for the complete range of groundwater parameters. For example, an open bailer is acceptable for collecting major ion and trace metal samples, but it may lead to erroneous analytical results if used for the collection of samples that are analyzed for volatile organics, dissolved gases, or even pH. Generally, the best pumps to use are positive displacement pumps, such as bladder and helical rotor pumps that minimize the aeration of the groundwater as it is

sampled, and therefore yield the most representative groundwater samples. Although it is possible to use different equipment to evacuate the well and to sample the well, this is not recommended because of the increased decontamination requirements and possibilities for cross contamination. It is recommended that a flow rate as close to the actual groundwater flow rate be employed to avoid further development, well damage, or the disturbance of accumulated corrosion or reaction products in the well (Puls and Barcelona, 1989).

Positive displacement pumps, such as bladder pumps, are generally recommended for both well evacuation and sample collection. Other methods of sample collection such as bailing or the use of gas lift pumps should be avoided, because of the geochemical changes can occur if the water is aerated during collection. Also, the use of these sample devices may entrain suspended materials such as fine clays and colloids which are not representative of mobile chemical constituents in the formation of interest (Puls and Barcelona, 1989).

Specific instructions for the use of several of the sampling devices are discussed in the next sections. All purging and sampling equipment should be decontaminated before beginning work and between wells in accordance with Section 4.4.

Bailers. Bailers represent the simplest and least expensive method of collecting groundwater sample from a well. However, they may not be suitable for all analyses. For most applications, the bailer should be constructed of Teflon or stainless steel. Disposable bailers constructed of polyethylene may also be acceptable for some applications (e.g., sampling for petroleum hydrocarbons), and they represent a simple method of avoiding cross-contamination between samples without the time-consuming need for decontamination. The following issues should be considered when using bailers for sampling:

- Bailers should be decontaminated per Section 4.4 of these guidelines and then isolated from any type of contamination prior to use for purging or sampling. The bailer should be decontaminated prior to the first well and between each subsequent well.

- Stainless steel or Teflon-coated stainless steel wire is recommended for lowering and retrieving the bailer from the well. At no time should the bailer or the line touch the ground during the sampling process. This can be done by coiling the line in a bucket or on a sheet of polyethylene. Polypropylene line may be substituted for the stainless steel wire, but should be discarded after each use.

- When lowering the bailer into the well, care should be taken to minimize agitation in the well, such as when the bailer contacts the water-table surface. The bailer should be lowered beneath the top of the screened interval.

Peristaltic/Centrifugal Pumps. Peristaltic and centrifugal pumps are widely used for purging wells with water levels close to the surface (less than 30 feet). They are reasonably portable, light, and easily adaptable to allow monitoring of field parameters with a flow-through cell. These pumps require minimal downhole equipment and can easily be cleaned in the field, or the entire tubing assembly can be changed for each well. The following procedures should be considered when using these pumps:

- Prior to use, the exterior and interior of all intake tubing for use with the peristaltic/centrifugal pump should be thoroughly flushed with tap water and then double rinsed with distilled water. New tubing should be used at each well and then discarded. If a gas-powered generator is used, it should be downwind of the well.

- The intake of the suction tubing should be lowered to the midpoint of the well screen. Alternatives to this procedure may be necessary if drawdown from purging causes the water level to fall below the pump intake. The suction line should be lowered slowly into the well until it pumps water continuously but not lower than 1 foot above the bottom of the well.

- If parameters are to be monitored with a flow-through cell, connect the instrumentation header to the pump discharge and begin flushing the well. Continuously monitor the parameters (pH, Eh, temperature, and specific conductivity) and measure the volume of groundwater being pumped. Alternatively, parameters may be monitored in a beaker filled from the pump discharge.

- After purging, remove the intake tubing from the well while the pump is still operating to prevent backwash of water into the well. Stop the pump and disconnect the tubing from the pump for cleaning or disposal.

- If tubing is to be reused (not recommended), clean the interior of the tubing by flushing thoroughly with tap water. Double rinse the tubing with distilled water. Using Alconox and water, wash the exterior of the tubing, and then rinse with tap water and distilled water.

Submersible Pumps. Submersible pumps take in water and push the sample up a sample tube to the surface. The power sources for these pumps may be compressed gas or electricity. The operation principles vary, and the displacement of the sample can be by an inflatable bladder, sliding piston, gas bubble, or impeller. Bladder or helical rotor pumps are recommended for sampling when sensitive parameters are to be analyzed. Pumps are available for 2-inch-diameter wells and larger, and these pumps can lift water up to several hundred feet. The entire pump assembly and tubing should be decontaminated before beginning purging and between wells, as described in Section 4.4.

Limitations of this class of pumps include:

- They may have low delivery rates.
- Submersible pumps are often more expensive than bailers and other sampling methods.
- Compressed gas or electricity is needed.
- Sediment in water may clog valves or erode pump impellers.
- Decontamination of internal pump components is often difficult and time consuming.

Advantages of this class of pumps include:

- Delivery of low-turbidity samples.
- Adjustable to very low flow rates.
- Some types (e.g., bladder pumps) are relatively inexpensive and easy to install as dedicated systems.
- Some types (e.g., bladder pumps) can be easily disassembled for decontamination.

2.2 WELL EVACUATION METHODS

2.2.1. Purging Requirements

To obtain a representative groundwater sample it must be understood that the composition of the water within the well casing and in close proximity to the well is probably not representative of the overall groundwater quality in the target aquifer. This is due to the possible presence of drilling materials near the well and because important environmental conditions such as the oxidation-reduction (redox) potential may differ drastically near the well from the conditions in the surrounding water-bearing materials. For these reasons it is necessary to pump or bail the well until it is thoroughly flushed of standing water and contains fresh water from the aquifer. The recommended amount of purging before sampling is dependent on many factors including the characteristics of the well, the hydrogeological nature of the aquifer, the type of sampling equipment being used, and the parameters that are to be analyzed.

Rather than relying on the removal of a specific volume of water (such as five casing volumes) prior to sample collection, physical parameters such as pH, specific conductivity, temperature, and possibly redox potential should be used to evaluate when enough water has been removed from the well to obtain a representative groundwater sample. However, it is recommended that where possible, a minimum of three casing volumes should be purged prior to sampling. The sensitivity of the above parameters to changes as a result of exposure of groundwater to surface conditions (i.e., changes in the partial pressure of dissolved gases or the conditions of the purging system) make *in-situ* monitoring desirable. An alternative to this would be to conduct these measurements in a closed cell attached to the discharge side of the pump system. Puls and Barcelona (1989) suggest that an initial estimate for the time of pumping necessary to collect representative water from a formation is around two times the time required to get stable values for the above parameters. For example, the parameters may be considered stable when several consecutive measurements (collected at least one-half a casing volume apart) do not change by more than the following:

- Conductivity ±10 percent
- pH ±0.1 unit
- Temperature ±1°C

When evacuating low-yield wells (wells that are incapable of yielding at least five casing volumes), the well should be evacuated to dryness once (USEPA, 1986). As soon as the well recovers sufficiently, the samples should be collected in the order of the parameter volatilization sensitivity. The samples should be retested for field parameters after sampling as a check on the stability of the water samples over time. Whenever full recovery exceeds two hours, the sample should be collected as soon as sufficient volume is available for a sample for each parameter. However, allowing a well to recover overnight is not acceptable. At no time should the well be pumped dry if the recharge rate causes the formation water to vigorously cascade down the sides of the screen and cause an accelerated loss of volatiles. In this case, samples should be collected at a rate slow enough to maintain the water level at or above the top of the screen to prevent cascading.

Other factors that will influence the amount of purging required before sampling include the pumping rate and the placement of the pumping equipment within the column of water in the well. For example, recent studies have shown that if a pump is lowered immediately to the bottom of a well before pumping, it may take some time for the column of water above it to be exchanged if the transmissivity of the aquifer is high and the well screen is at the bottom of the casing. In these cases, the pump will be drawing

water primarily from the aquifer. Purging from higher in the well or just below the water surface provides a more complete removal of the casing water.

2.2.2. Calculation of Casing Volume

To ensure that an adequate volume of water has been removed from the well prior to sampling, it is first necessary to determine the volume of standing water in the well and the volume of water in the filter pack below the well seal. The volume can be easily calculated by the following method (calculations should be entered in the field logbook):

1. Obtain all available information on well construction (e.g., location, casing, screen, depth).
2. Determine well or casing diameter.
3. Measure and record static water level (depth below ground level or top of casing reference point) using one of the methods described in Section 2.3.1.
4. Determine depth of well by sounding using a clean, decontaminated weighted tape measure or an electronic water-level probe.
5. Calculate the volume of water in the casing using the following formula:

$$V = 7.481\ (\pi r^2 h)$$

Where: V = Casing Volume (gal)
r = Well radius (ft) = well diameter (ft)/2
h = Linear feet of water in well = total well depth (ft) - static water depth (ft)

Alternatively, the casing volume can be calculated by multiplying the linear feet of water in the well by the volume per linear foot taken from Attachment 1 or other similar tables. Always be sure that the units in your calculation are consistent. In the equation above, 7.481 is the conversion factor from cubic feet to gallons.

2.2.3. Calculation of Annulus Volume

Some groundwater sampling protocol require the evacuation of casing and annulus volumes prior to sampling. In these cases the volume of water contained in the annular space between the casing and the borehole wall is calculated by the following formula:

$$V^C = (C_b - C_c)\ x\ (h)\ x\ (0.30)$$

Where:

C_b = Borehole Capacity (Volume in Gal./ft)
C_c = Casing Capacity (Volume in Gal./ft)
h = Amount of standing water in the well
0.30 = Average porosity of typical sand pack

The annulus volume is added to the casing volume prior to multiplying by the number of volumes to be evacuated.

2.2.4. Purge Water Handling and Disposal

Because of the potential for spreading environmental contamination, planning for purge water disposal is a necessary part of well monitoring. Alternatives range from releasing it on the ground (not back down the well) to full containment, treatment, and disposal. If the well is believed to be contaminated, the best practice is to contain the purge water and store it in drums labeled "purge water" or in aboveground portable storage until the water samples have been analyzed. Once the contaminants are identified, appropriate treatment or disposal requirements can be determined.

2.3 SAMPLE COLLECTION METHODS

Prior to sampling, the sampling team will document any signs of tampering or well deterioration. After opening the well, a PID will be used to scan headspace air for organic vapors and an oxygen/combustible gas indicator will be used to scan for explosive atmospheres. These measurements will be recorded in the field logbook. A plastic sheet will be placed around the well head beneath all sampling equipment to prevent sampling equipment and contamination of surface soil during purging. All groundwater samples should be collected using a clean, dry decontaminated bailer made of either stainless steel or Teflon unless a HydroPunch® groundwater system is being used.

2.3.1. Sample Containers

A complete set of sample containers should be prepared by the laboratory prior to going into the field. The laboratory should provide the proper containers with the required preservatives. The laboratory's QA manual should provide a complete description of the procedures used to clean and prepare the containers. The containers should be labeled in the field with the date, well designation, project name, collectors' name, time of

collection, parameters to be analyzed, and preservative. The sample containers should be kept in a cooler (at 4°C) until they are needed (i.e., not left in the sun during purging). One cooler should be used to store the unfilled bottles and another to store the samples.

All sample bottles and equipment will be kept away from fuels and solvents. If required for generators, gasoline will be transported in a different vehicle from bailers, sample bottles, purging pumps, etc. If possible, the person designated to handle samples should not also handle gasoline.

The sample bottles will be filled in order of the volatility of the analytes so that the containers for volatile organics will be filled first, and samples that are not pH-sensitive or subject to loss through volatilization will be collected last. A preferred collection order (as listed in USEPA, 1986) is as follows:

- Volatile organics (VOCs)
- Total petroleum hydrocarbons (TPH)
- Total organic halogens (TOX)
- Total organic carbon (TOC)
- Extractable organics (e.g., BNAs, pesticides, herbicides)
- Total metals
- Dissolved metals
- Phenols
- Cyanide
- Sulfate and chloride
- Turbidity
- Nitrate and ammonia
- Radionuclides

When groundwater samples are to be analyzed for volatile organic, samples will be carefully transferred from the bailer or sample collection device using a valved bottom discharging device or in a manner than minimizes volatilization. The VOA vials will be filled by inserting the spout from the bailer to the bottom of the VOA vial with discharge of the bailer contents into the vial such that the tip of the spout is kept beneath the surface of the liquid in the vial as it is filled until there is a convex meniscus over the neck of the bottle. The Teflon side of the septum will be positioned against the meniscus, and the cap screwed on tightly; the sample will be inverted, and the bottle tapped lightly. The absence of an air bubble indicates a successful seal; if a bubble is evident the sample will be discarded. Refilling of VOA vials will not be allowed.

2.3.2. Field Filtration for Dissolved Metals

Filtration should be performed in the field with no air contact and immediate preservation and storage. In-line pressure filtration is prefered with as small a filter pore size as practically possible (e.g., 0.45, 0.10 micron). Disposable in-line filters are recommended for convenience and avoiding cross-contamination. The filters should be pre-rinsed with distilled water; work by Jay (1985) showed that virtually all filters require pre-washing to avoid sample contamination.

In the absence of filters, sample turbidity can generally be reduced by using bladder pumps. USEPA (1986) recommends that the sample turbidity should be less than 5 nephelometric turbidity units (NTUs).

2.4 FIELD MEASUREMENTS

A variety of field measurements are commonly made during the sampling of groundwater including: water level, pH, conductivity, and temperature. The accuracy, precision, and usefulness of these measurements is dependent on the proper use and care of the field instruments. Valid and useful data can only be collected if consistent practices (in accordance with recommended manufacturers instructions) are followed. The instruments should be handled carefully at the well site and during transportation to the field and between sampling sites.

2.4.1. Water Level

Water levels can be measured by several techniques, but the same steps should be followed in each case. The proper sequence is as follows:

1. Check operation of measurement equipment above ground. Prior to opening the well, don personal protective equipment as required.

2. Record all information specified below on a sampling form or in the field notebook if a form is not available.

3. Record well number, top of casing elevation, and surface elevation if available.

4. Measure and record static water level and total depth to the nearest 0.01 foot (0.3 cm) from the surveyed reference mark on the top edge of the inner well casing. If no reference mark is present, record in the log book

where the measurement was taken from (i.e., from the north side of the inner casing).

5. Record the time and day of the measurement.

6. Some water-level measuring devices have marked metal or plastic bands clamped at intervals along the measuring line used for reference points to obtain depth measurements. The spacing and accuracy of these bands should be checked before each round of measurements because they may loosen and slide up or down the line, resulting in inaccurate reference points.

Electric Water Level Indicators. These devices consist of a spool of small-diameter cable or tape and a weighted probe attached to the end. When the probe comes in contact with the water, an electrical circuit is closed and a meter, light, and/or buzzer attached to the spool will signal the contact. This is the recommended method for obtaining accurate water-level measurements.

There are a number of commercial electric sounders available, none of which is entirely reliable under all conditions likely to occur in a contaminated monitoring well. In conditions where there is oil on the water, groundwater with high specific conductance, water cascading into the well, or a turbulent water surface in the well, measuring with an electric sounder may be difficult.

For accurate readings, the probe should be lowered slowly into the well. The electric tape is marked at the measuring point where contact with the water surface was indicated. The distance from the mark to the nearest tape bank is measured using a ruler or steel tape and added to the band reading to obtain the depth to water. Band spacing should be checked periodically as described above.

2.4.2. pH

The pH meters should be calibrated against two ASTM traceable standard pH solutions, either 4 and 7 or 7 and 10, depending on whether previous pH measurements have been less than or greater than 7, respectively. Calibration measurements will be recorded in the field logbook. The meter readings will be adjusted, and the probe should then be rinsed thoroughly with distilled water. The probe should then be immersed in the water sample, and the pH and temperature recorded in the field log or on the sampling form. Calibration standards will be measured again as a check after sample measurement and recorded in the field logbook. The manufacturer's directions for calibration, maintenance,

and use should be read and closely followed. Any problems with the functioning of the meter should be noted in the field log and reported to the office equipment manager.

2.4.3. Conductivity

Specific conductivity meters should be standardized by immersing a decontaminated specific conductivity probe into an ASTM traceable standard solution of conductivity buffer. The conductivity of the standard solution should be within the same order of magnitude as that anticipated for the water sample. Calibration results will be recorded in the field logbook. The meter reading will be adjusted to the buffer solution value, and the probe will then by thoroughly rinsed with distilled water. The probe should then be immersed in the well water sample, and the conductivity value recorded. Calibration standards will be measured again as a check after sample measurement and recorded in the field logbook. The manufacturer's directions for calibration, maintenance, and use should be read and closely followed. Calibrant solutions should be dated and discarded on their expiration date. Any problems with the functioning of the meter should be noted in the field log and reported to the office equipment manager.

2.4.4. Temperature

Temperature measurements should be made with either a mercury or electronic thermometer capable of accurately reading to 0.1°C. The temperature reading should be recorded in the field log or on the sampling form.

2.5 DECONTAMINATION

The general decontamination procedure for all non-dedicated groundwater sampling equipment (bailers, pumps, water-level probes) consists of the following steps:

1. Scrub and wash with laboratory-grade detergent (such as Alconox) and tap water;

2. Rinse with reagent-grade isopropyl alcohol or methanol and allow to air dry; and

3. Triple rinse with deionized water.

If available, a steam cleaner can also be used for decontaminating sampling equipment. Steam cleaning is the desired method since it does not introduce any additional chemicals

into the system. If a steam cleaner is available it should be used instead of any other type of decontamination procedure. As with other procedures documented in this SOP, decontamination procedures may be determined by the client or regulatory agency involved in the project.

4.0 REFERENCES

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5.0 ATTACHMENTS

1 - Volume of Schedule 40 PVC Pipe

2 - Ground-water/Surface Water Sampling Form

VOLUME OF SCHEDULE 40 PVC PIPE

Nominal Casing Diameter (inches)	Outer Casing Diameter (inches)	Inner Casing Diameter (inches)	Volume (gal/linear ft)
1 1/4	1.660	1.380	0.08
2	2.375	2.067	0.17
3	3.500	3.068	0.38
4	4.500	4.026	0.66
6	6.625	6.065	1.5
8	8.625	7.981	2.6
12	12.750	11.938	5.8

Management Review ___ Other ___

Technical Review ___ Project Manager ___

Graphic Standards ___ Lead Professional ___

QUALITY CONTROL

GROUND-WATER/SURFACE WATER SAMPLING FORM

Sample Location_____ Surface Water/Ground Water_____ Sample Identification _____

Sampling Personnel_____ Date_____ Weather_____

MEASUREMENT SUMMARY:

Time_____ Depth to Water_____ Depth to Product_____ Product Thickness_____

Total Casing Depth_____ Borehole Diameter_____ Calculated Purge Volume_____ Gallons

Measuring Point_____ Final pH_____ Final SC_____ Final Temp(°C)_____

SAMPLING SUMMARY:

Sampling Method: Dedicated Bladder Pump _____ Portable Bladder Pump _____ Bailer_____

Pump Started_____ Pump Stopped_____ Total Gallons_____ FID Reading at Well Head_____

Time	pH	SC (umhos/cm)	Temp (°C)	Turb. (NTU)	Flow Rate (gpm)	Vol (gal.) Evacuated	Comments

INSTRUMENTATION: pH Meter: Orion ☐ Cole-Parmer ☐ Calibration Buffers: 4 ☐ 7 ☐ 10 ☐

Specific Conductivity Meter: Markson/Amber ☐ YSI ☐ Standard Solution _____umhos/cm

Turbidimeter: HF Scientific ☐ Other ☐ Reference Solution _____NTUs

SAMPLES COLLECTED AND TIME: VOCs _____ SVOCs _____ TPH _____

Pest./PCBs_____ Metals _____ Hexavalent Chromium _____

Cl^-, F^-, SO_4^- _____ NO_3/NO_2 _____ TDS _____ Other_____

Appendix F
Attachment 2

This document has been developed for a specific application and may not be used without the written approval of Montgomery Watson.

Developed By / Drawn By	**GROUNDWATER SAMPLING FORM**	Drawing Number 4077.0191
Approved By / Date 8/12/96	TREATABILITY STUDY AMERICAN CHEMICAL SERVICE, INC. NPL SITE GRIFFITH, INDIANA	MONTGOMERY WATSON
Reference		
Revisions		

STANDARD OPERATING PROCEDURES

GROUNDWATER MONITORING WELL DEVELOPMENT

STANDARD OPERATING PROCEDURES

GROUNDWATER MONITORING WELL DEVELOPMENT

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1.0 INTRODUCTION

The goal of monitoring well development is to remove fines and drilling fluid residue from the gravel pack and the natural formation in the vicinity of the screened interval. Proper well development ensures that a sample collected from the well will be representative of the water quality in the aquifer.

The well development process involves: (1) the application of sufficient energy in a monitoring well to create groundwater flow reversals (surging) in and out of the well and the gravel pack to release and draw fines into the well; and (2) pumping or bailing to draw drilling fluids out of the borehole and adjacent natural formation, along with fines that have been surged into the well.

2.0 WELL DEVELOPMENT

2.1 GENERAL

The following general guidelines are applicable to well development regardless of method.

2.1.1 Decontamination

It is essential that every effort be made to avoid outside contamination and the cross-contamination of monitoring wells. This can best be done by ensuring that all equipment introduced into a well is clean. The level of effort for decontamination is a site- and project-specific issue to be resolved individually for each project.

2.1.2 Documentation

A critical part of monitoring well development is the recording of significant details and events in either a field log book or on a well development form (attached to this SOP). Listed are some important details to document.

- Well identification number
- Installation date
- Date and time of development
- Quantity of drilling fluid lost during well installation

- All PID readings
- Measured well depth (pre-development and post-development)
- Water level
- Height of water column
- Pumping rate and water level drawdown (if applicable)
- Recharge rate (slow, moderate, rapid)
- Periodic field parameter readings
- Sample observations
- Type of equipment used
- Total amount of water removed
- Completion time

2.1.3 Calculating Purge Volume

Before the development process begins, you will need to calculate the minimum number of gallons to be removed.

Information needed to calculate purge volume:

1. Total depth of well (TD)
2. Measured static water level (WL)
3. Screen length (SL)
4. Well casing inner diameter (ID)
5. Borehole Diameter (BD)
6. Number of gallons of water used during well drilling/construction
7. If the standing water column (SC) is longer then the screen length, you will need to note the number of feet of filter pack installed above the top of the screen

Calculating one well volume:

- To calculate standing water column (SC): TD - WL = SC
- Use a well volume chart to find a multiplier in the "Gallons per foot" (Table 1) column that coincides with the wells ID
- SC x ID multiplier = gallons of water in one well volume

TABLE 1

VOLUME OF SCHEDULE 40 PVC PIPE

Nominal Casing Diameter (inches)	Outer Casing Diameter (inches)	Inner Casing Diameter (inches)	Volume (gal/linear ft)
1 1/4	1.660	1.380	0.08
2	2.375	2.067	0.17
3	3.500	3.068	0.38
4	4.500	4.026	0.66
6	6.625	6.065	1.5
8	8.625	7.981	2.6
12	12.750	11.938	5.8

Calculating one annulus volume (2 Options):

Option 1, if SC is shorter than the screen length

- Portion of saturated annulus = SC
- Use a volume chart to find a multiplier in the 'Gallons per foot' column that coincides with the wells BD
- BD multiplier - ID multiplier = annulus multiplier
- Feet of saturated annulus x annulus multiplier x 30%(assumed porosity) = gallons of water in one annulus volume

Option 2, if SC is longer than the screen length

- Portion of saturated annulus is = to the screen length + the number of feet of filter pack above the top of the screen
- Use a volume chart to find a multiplier in the 'Gallons per foot' column that coincides with the wells BD
- BD multiplier - ID multiplier = annulus multiplier
- Feet of saturated annulus x annulus multiplier x 30%(assumed porosity) = gallons of water in one annulus volume

Calculating the minimum gallons to be removed:

- well volume + annulus volume + number of gallons lost during well drilling/construction = one purge volume

2.2 DEVELOPMENT METHODS

2.2.1 Bailing, Surging, and Pumping

In relatively clean, permeable formations where water flows freely into the borehole, bailing, surging, and pumping is an effective development technique. First, the bottom of well is tagged to measure the amount of sand/silt before and after surging, then a bailer should be lowered down the well to clean out any fines that have settled on the bottom of the well. Then a surge block, approximately the same diameter as the well casing, is used to agitate the water, causing it to move in and out of the screen, thus drawing in fines from the gravel pack and surrounding formation, and breaking up any bridges that may

have occurred during the placement of the gravel pack. After surging for a few minutes (depending on the height of the water column and length of screen), the bailer should then be lowered again to clean out any fines that were drawn into the casing as a result of surging. This surge/bail technique should continue until minimal fines are being pulled out with the bailer. A submersible pump will then be lowered down the well. Pumping should begin at the top of the saturated portion of the screened interval to prevent sand locking of the pump. The pump should be lowered at intervals of 5 feet or less until the pump is approximately 1 foot above the bottom of the casing. Monitor the water level continuously during the first few minutes of pumping so as not to draw the water level below the pump intake and break the suction. Increase the discharge flow rate (if possible) until the well is pumping at its maximum yield without drawdown below the pump intake.

2.2.2 Overpumping and Backwashing

Wells may be developed by overpumping (pumping or bailing the well at a rate that exceeds the ability of the formation to deliver water) and then reversing the flow direction (backwashing) so that the water is passing from the well into the gravel pack and formation. This back-and-forth movement of water through the well screen and gravel pack serves to remove fines from the formation immediately adjacent to the well, while preventing bridging (wedging) of sand grains. Backwashing can be accomplished by several methods including pouring water into the well and then bailing, or forcing water into the well under pressure through a water-tight fitting. Care should be taken when backwashing not to apply too much pressure, which could damage or destroy the well screen. Where no backflow prevention valve is installed, a pump can be alternately started and stopped. This starting and stopping allows the column of water that is initially picked up by the pump to be alternately dropped and raised in a surging action. This surging loosens bridging of fine particles and draws them into the well where they can be pumped out.

2.2.3 Compressed Air

Compressed air can be used to develop a well by either of two methods: backwashing or surging. Backwashing is done by forcing water out through the screens, using increasing air pressure inside a sealed well, then releasing the pressurized air to allow the water to flow back into the well. Care should be taken when using this method so that the water level does not drop below the top of the screen, thus reducing well yield. Surging, or the

"open well" method, consists of alternately releasing large volumes of air suddenly into an open well below the water level to produce a strong surge by virtue of the resistance of water head, friction, and inertia. The well can then be pumped with the air lift method.

2.2.4 Developing Wells With Floating Product

It is important to disturb the formation as little as possible in wells that contain floating product. Surge blocks should not be used because they can smear the screen and the casing with product when the block is being withdrawn. Wells which contain floating product should be developed using a bail/pump method. A bailer should be lowered gently into the well, so as to not agitate the water column, removing any fines that have settled on the bottom. If the well produces sufficient water, lower a pump down the well and start pumping at a low flow rate. Monitor the product/water level constantly for the first few minutes, do not allow the product level to come within 2 feet of the pump intake. Continue pumping until at least the quantity of drilling fluid lost has been purged, the parameters have stabilized, and the discharge water is visibly clear.

2.2.5 Developing Wells In Tight Formations

For wells installed in clay or silt the method of development should be bailing only. Surging of such wells has been found to substantially increase the turbidity of the water and does not significantly improve hydraulic response.

3.0 REFERENCES

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4.0 ATTACHMENTS

Well Development Form

QUALITY CONTROL | Graphic Standards / Lead Professional | Technical Review / Project Manager | Management Review / Other

WELL DEVELOPMENT FORM

DATE: _____ WEATHER: _____

WELL DESIGNATION: _____

FIELD PERSONNEL _____

WELL SUMMARY:

Depth to NAPL (if applicable): _____

Initial depth to water: _____

Initial total well depth: _____

Final depth to water: _____

Final well depth: _____

Location Map

Construction: _____

_____

INSTRUMENTATION

pH meter (model): _____ Calibrated with buffers: _____4 _____7 _____10

SC meter (model): _____ Calibrated with standard solution: _____ µmhos/cm

Turbidity meter (model): _____ Calibrated with: _____

DEVELOPMENT SUMMARY:

Development method: Bailer_____ Pump (type) _____

Final pH: _____ Final SC: _____ Final Temp (°C): _____ Final Turbidity: _____

Time	pH	SC (µmhos/cm)	Temp (°C)	Turbidity (NTU)	Pumping Rate	Gals. Evacuated	Visual Appearance/Comments

Appendix F
Attachment 3

This document has been developed for a specific application and may not be used without the written approval of Montgomery Watson.

Developed By	Drawn By	**WELL DEVELOPMENT FORM**	Drawing Number 4077.0191
Approved By	Date 8/12/96		
Reference		TREATABILITY STUDY AMERICAN CHEMICAL SERVICE, INC. NPL SITE GRIFFITH, INDIANA	**MONTGOMERY WATSON**
Revisions			

STANDARD OPERATING PROCEDURES

FIELD DOCUMENTATION

STANDARD OPERATING PROCEDURES

FOR FIELD DOCUMENTATION

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1.0 INTRODUCTION

This guideline is a general reference for the required documentation to be completed by company personnel during field investigations. Documentation in the form of field logbooks, reports, and forms should be completed for every activity in the field. Records should be maintained on a daily basis as the work progresses. All field documentation should be accurate and legible because it is part of the client's product and may potentially serve as a legal document.

Sample field documentation forms are attached.

2.0 FIELD DOCUMENTATION GUIDELINES

Field documentation serves as the primary foundation for all field data collected that will be used to evaluate the project site. All field documentation should be accurate, legible and written in indelible ink. Absolutely no pencils or erases are to be used. Mistakes written in the field books, logs, or on forms that need to be deleted should be crossed out with one line, initialed, and dated. Skipped pages or blank sections at the end of a page should be crossed out with an "X" covering the entire page or blank section; "No Further Entries," initials, and date should be written by the person making the correction. The responsible field team member should write his/her signature, date, and time after the day's last entry. To further assist in the organization of the field books, logs, or forms, it is important to write the date on top of each page and the significant activity description (e.g., boring or well number). Each project job number should have its own field book. In addition, all original field documentation should be submitted to the project files.

The descriptions of field data/documentation given below serve as an outline; individual projects will vary in documentation needs.

2.1 FIELD LOGBOOKS

The field logbook is a bound, weatherproof book with numbered pages that serves primarily as a daily log of the activities carried out during the investigation. All entries should be made in indelible ink. A field logbook should be completed for each operation undertaken during the investigation, such as field team leader notes, drilling, groundwater sampling/development, and site visitors. The logbook should serve as a diary of the events of the day.

Field activities will vary from project to project; however, the concept and general information that should be recorded will remain similar. A detailed description of three basic logbooks in which field activities should be documented is given below. These field logbooks include the Field Team Leader (FTL) logbook, rig geologist/sampling team logbook, and groundwater sampling/development logbook. The following sections describe the minimum information that should be recorded in each of these logbooks.

FTL Logbook

The field team leader's responsibilities include the general supervision, support, assistance, and coordination of the various field investigation activities. As a result, a large portion of the FTL's day is spent rotating between operations in a supervisory role. Records of the FTL's activities as well as a summary of the field team's activities should be maintained in a logbook. The FTL's logbook will be used to fill out daily quality control reports (DQCRs), and as such should contain all information required in these reports (refer to Section 3.3). Items to be documented include:

- Record of tailgate meetings
- Personnel and subcontractors on job site and time spent on the site
- Field operations and personnel assigned to these activities
- Site visitors
- Log of FTL's activities: time spent supervising each operation and summary of daily operations as provided by field team members
- Problems encountered and related corrective actions
- Deviations from the sampling plan
- Records of communications: discussions of job-related activities with the client, subcontractor, field team members, and project manager
- Information on addresses and contacts
- Record of invoices signed and other billing information
- Field observations

Rig Geologist/Sampling Team Logbook

The rig geologist or sampling team leader is responsible for recording the following information:

- Health and Safety Activities
  - Calibration records for health and safety equipment (type of PID, calibration gas used and associated readings, noise dosimeters, etc.)
  - Personnel contamination prevention and decontamination procedures
  - Record of daily tailgate safety meetings
- Weather
- Calibration of field equipment
- Equipment decontamination procedures
- Personnel and subcontractors on job site and time spent on the site
- Site name and well or soil boring number
- Drilling activities
  - Sample location (sketch)
  - Drilling method and equipment used
  - Borehole diameter
  - Drill cuttings disposal (number of drums, roll off-bins, etc.)
  - Type and amount of drilling fluids used (mud, water, etc.)
  - Depth and time at which first groundwater was encountered, depth to water at completion of drilling, and the stabilized depth to water. The absence of water in the boring should also be noted.
  - Total drilling depth of well or soil boring
  - Type and amount of materials used for well installation
  - Well construction details [depth of grout (mixture, weight), bentonite seal, filter pack, etc. [include type and amount used, calculate estimated amount that should be used]
  - Type and amount of material used to backfill soil borings
  - Time and date of drilling, completion, and backfilling
  - Name of drilling company, driller, and helpers

- Sampling
  - Date and time of sample collection
  - Sample interval
  - Number of samples collected
  - Analyses to be performed on collected samples
- Disposal of contaminated wastes (PPE, paper towels, visqueen, etc.)
- Field observations
- Problems encountered and corrective action taken
- Deviations from the sampling plan
- Site visitors

Groundwater Sampling/Development Logbook

The groundwater sampling and development team members are responsible for recording the following information.

- Health and Safety Activities
  - Calibration records for health and safety equipment (i.e. type of PID, calibration gas used and readings, noise dosimeters etc.)
  - Personnel contamination prevention and decontamination procedures
  - Record of daily tailgate safety meetings
- Weather
- Calibration of field equipment
- Equipment decontamination procedures
- Personnel and subcontractors on job site and time spent on the site
- Equipment decontamination procedures
- Disposal of contaminated wastes (PPE, paper towels, visqueen, etc.)
- Site name, well number

- Water levels and product levels [time, date, and datum from which water levels are measured (i.e. top of casing)]. Purging of the well (include calculations, well volumes) with the following information:
  - Measured field parameters (temperature, pH, conductivity, odor, color, cloudiness, etc.)
  - Amount of water purged
  - Purge method: indicate bailer/pump, diameter and length of bailer, material that the bailer is composed of, type of pump, new nylon rope, etc.
- Purge water disposal/containment (Baker tank/ drums, number used, identification, etc.)
- PID readings from inside of well, purged water, and breathing zone
- Background PID readings
- Well sampling
  - Number of samples collected and type of containers used
  - Date and time of sample collection
  - Type of analyses
  - QA/QC samples collected; names given to blind samples
- Field observations
- Problems encountered and corrective actions taken
- Deviations from the sampling plan
- Site visitors

2.2 TAILGATE SAFETY MEETINGS

Tailgate safety meetings are held at the beginning of each day before the initiation of work. All personnel, subcontractors, and others who will be on the job site are required to attend. The meetings are usually conducted by the FTL, on-site safety officer, or other qualified team member. The topics discussed at the meeting should include the following:

- Protective clothing and equipment
- Chemical hazards
- Physical hazards
- Special equipment
- Emergency procedures
- Emergency phone numbers
- Directions to the hospital

All site personnel are required to sign the tailgate safety meeting form. The original form should be kept on site, and a copy should be retained in the office.

2.3 DAILY QUALITY CONTROL REPORTS

The preparation of Data Quality Control Reports (DQCRs) is the responsibility of the field team leader. DQCRs are completed on a daily basis and should summarize the events of the day and supplement the information that is already recorded in the field logbook. DQCRs should be completed regardless of the duration of the field effort. Depending on the client, copies of the report should be distributed to the Montgomery Watson Project Manager, Montgomery Watson Project Geologist, Client Project Manager (depending on the project), field office file, and home office file. Information recorded in this report should include the following.

- Date and Weather Information. date, daily temperatures, wind speed and direction, humidity.
- Montgomery Watson Personnel and Time Spent on Site
- Subcontractors and Time Spent on Site
- Special Equipment on Site. PID, Smeal Water Sampling Rig, Hollow-Stem Auger Rig, pH meter, conductivity meter, etc.
- Work and Sampling Performed. Personnel performing specific site activities, a summary of samples collected, and a thorough explanation of the work completed.
- Quality Control Activities. Activities such as decontamination procedures, QA/QC samples taken, calibration of field equipment, etc.
- Health and Safety Levels and Activities. Field parameter measurements, including calibration of equipment. Includes daily tailgate safety meetings, level of protection used, etc.
- Problems Encountered/Corrective Actions Taken. Any technical difficulties, for example problems encountered during drilling or equipment breakdowns. Any problems that could potentially affect the quality of the samples should be included.
- Special Notes. Any information that does not fit under the categories listed above, but is important to record.
- Next Day's Expectations
- Signature of Individual Completing the Report.

2.4 BORING LOGS

The preparation of drill logs is the responsibility of the field team members assigned to the drill rig. A detailed description of well logging is provided in the SOP for that subject. Several examples of drilling logs are given in the attachments. The format is dependent upon the job and the client; however, the following basic information should be recorded on the log regardless of the format.

- Project and site name
- Name of driller and drilling company
- Well/soil boring ID and location (sketch)
- Drilling and backfilling dates and times
- Reference elevation for all depth measurements
- Total depth of completed soil boring/well
- Depth of grouting, sealing, and grout mixes
- Signature of the logger.
- Description of unconsolidated materials
    - Geologic lithology description
    - Descriptive Unified Soil Classifications System (USCS) classification
    - USCS symbol
- Color (use appropriate soil color chart)
    - Penetration resistance (consistency or density)
    - Moisture content
    - Grain size information
    - Miscellaneous information (odor, fractures, visible contamination, etc.)
- Description of consolidated materials
    - Geologic rock description
    - Rock type
    - Relative hardness
    - Density
    - Texture
    - Color (use appropriate rock color charts)
    - Weathering
    - Bedding
    - Structures (fractures, joints, bedding, etc.)
    - Miscellaneous information (presence of odor, visible contamination, etc.)

- Stratigraphic/lithologic changes; depths at which changes occur
- Depth intervals at which sampling was attempted and amount of sample recovered
- Blow counts
- Depth intervals from which samples are retained
- Analyses to be performed on collected samples
- Depth at which first groundwater was encountered, depth to water at completion of drilling, and the stabilized depth to water. The absence of water in the boring should also be noted.
- Loss and depth of drilling fluids, rate of loss, and total volume of loss
- Use of drilling fluids
- Drilling and sampling problems
- PID readings

2.5 WELL CONSTRUCTION DIAGRAMS

The preparation of well construction diagrams are the responsibility of field team members assigned to the drilling operations. This topic is further discussed in the SOP for Well Installation. The format of the diagram is dependent on the job and the client; however, the following basic information should be recorded and/or illustrated on the diagram regardless of the format:

- Project and site name
- Well identification number
- Name of driller and drilling company
- Depth and type of well casing
- Description of well screen and blank
- Borehole diameter
- Any sealing off of water-bearing strata
- Static water level upon completion of the well and after development
- Drilling and installation dates
- Type and amount of annulus materials used; depth measurements of annulus materials

- Other construction details (filter pack type and interval, location of centralizers, etc.)
- Surface elevation and reference elevation of all depth measurements

2.6 GROUNDWATER SAMPLING/DEVELOPMENT LOGS

The groundwater sampling/development log should be used any time that a well is developed or sampled. The following information should be recorded on the log:

- Project name and site
- Well identification number
- The date and time of sampling/development
- The water level and reference elevation
- Volume of water to be purged
- Pertinent well construction information (total depth, well diameter, etc.)
- Measurement of field parameters such as pH, turbidity, conductivity, and temperature, as well as the times at which the readings were taken.
- Type of purging and sampling equipment used
- Type of samples collected
- Sampler's initials

2.7 DOCUMENTATION OF SAMPLING ACTIVITIES

Documentation to be made during sampling activities includes sample labels, sample seals, Chain-of-Custody Records, and sample register.

2.7.1 Sample Labels

A sample label should be affixed to all soil and water sample containers, and completed with information written in indelible ink. Required information on sample labels may vary from job to job; however, the following should be included at a minimum:

- Sample number
- Type of sample (grab or composite)
- Type of preservative, if applicable
- Date and time of collection

- Project location
- Analyte(s)
- Initials of sampling personnel

2.7.2 Custody Seals

Custody seals consist of security tape with the initials of the sampler and the date placed over the lid of each cooler containing samples. The tape should be placed such that the seal must be broken to gain access to the contents. Custody seals should not be placed directly onto the volatile organic compound (VOC) sample bottles. Custody seals should be placed on coolers prior to the sampling team's release to a second or third party (e.g., shipment to the laboratory).

2.7.3 Chain-of-Custody Records

Chain-of-custody (COC) procedures allow for the tracing of possession and handling of individual samples from the time of field collection through laboratory analysis. Documentation of custody is accomplished through a chain-of-custody record that lists each sample and the individuals responsible for sample collection, shipment, and receipt. A sample is considered in custody if it is:

- In a person's possession.
- In view after being in physical possession.
- Locked or sealed so that no one can tamper with it after it has been in an individual's physical custody.
- In a secured area, restricted to authorized personnel.

A COC record is used to document the samples collected and the analyses requested. Information recorded includes time and date of sample collection, sample number, and the type of sample, the sampler's signature, the required analysis, and the type of containers and preservatives used. A copy of the COC record should be retained by the sampler prior to release to a second or third party. Shipping receipts should be signed and filed as evidence of custody transfer between field sampler(s), courier, and laboratory.

The COC Record will be properly signed and the date of collection and shipment recorded, along with the sample site identifications and requested analyses for each sample.

2.7.4 Sample Register

The sample register is a field record book with prenumbered pages. A full description of each sample is recorded in the book. The information included in the sample register should include the following:

- Sample number (identification)
- Duplicate and split sample numbers (identification)
- Location of sample
- Client
- Project number
- Collection method
- Number and size of bottles for each analysis
- Destination of the sample
- Type of analysis
- Date and time of collection
- Name of sampler

Other observations may be included as the situation dictates to ensure a thorough record that could be used to reconstruct the events concerning that sample. All information should be recorded in indelible ink.

3.0 ATTACHMENTS

4 - Tailgate Safety Forms and Health and Safety Documentation

5 - Daily Quality Control Reports

QUALITY CONTROL | Graphic Standards Lead Professional | Technical Review Project Manager | Management Review Other

TAILGATE SAFETY MEETING FORM

Date: _____ Time: _____ Job Number: _____

Client:: American Chemical Service

Scope of Work: _____

SAFETY TOPICS PRESENTED

Protective Clothing/Equipment: _____

Chemical Hazards: _____

Physical Hazards: _____

Special Equipment: _____

Other: _____

Emergency Procedures: _____

Hospital: _____ Phone: _____ Ambulance Phone: _____

Hospital Address and Route: _____

ATTENDEES

NAME PRINTED	SIGNATURE

Meeting Conducted By: _____ _____

Name Printed Signature

Project Safety Officer: _____ Project Manager: _____

Appendix F
Attachment 4

Developed By	Drawn By	**TAILGATE SAFETY MEETING FORM**	Drawing Number 4077.0191
Approved By	Date 8/12/96		
Reference		TREATABILITY STUDY AMERICAN CHEMICAL SERVICE, INC. NPL SITE GRIFFITH, INDIANA	MONTGOMERY WATSON
Revisions			

This document has been developed for a specific application and may not be used without the written approval of Montgomery Watson.

Management Review ____ Other ____

Technical Review ____ Project Manager ____

Graphic Standards ____ Lead Professional ____

QUALITY CONTROL

DATE _____

DAY	S	M	T	W	TH	F	S

A-E DAILY QUALITY CONTROL REPORT

WEATHER	Bright Sun	Clear	Overcast	Lt. Rain	Rain
TEMP	To 32	32–50	50–70	75–85	85 up
WIND	Still	Moder.	High	Report No.	
HUMIDITY	Dry	Moder.	Humid		

ACS TECHNICAL MANAGER _____

PROJECT American Chemical Service

JOB NO. _____

CONTRACT NO. _____

A-E PERSONNEL ON SITE:

SUB-CONTRACTORS ON SITE:

EQUIPMENT ON SITE:

WORK PERFORMED (INCLUDING SAMPLING):

A-E DAILY QUALITY CONTROL REPORT

Page ____ of ____

Appendix F
Attachment 5

This document has been developed for a specific application and may not be used without the written approval of Montgomery Watson.

Developed By	Drawn By	A-E DAILY QUALITY CONTROL REPORT	Drawing Number 4077.0191
Approved By	Date 8/12/96		
Reference		TREATABILITY STUDY AMERICAN CHEMICAL SERVICE, INC. NPL SITE GRIFFITH, INDIANA	MONTGOMERY WATSON
Revisions			

(Continuation Sheet)

PROJECT American Chemical Service **REPORT NO.** _____

JOB NO. _____ **DATE** _____

QUALITY CONTROL ACTIVITIES (INCLUDING FIELD CALIBRATIONS)

HEALTH AND SAFETY LEVELS AND ACTIVITIES:

PROBLEMS ENCOUNTERED/CORRECTION ACTION TAKEN:

SPECIAL NOTES:

TOMMORROW'S EXPECTATIONS:

BY _____ **TITLE** _____

Page ____ of ____

QUALITY CONTROL | Graphic Standards Lead Professional | Technical Review Project Manager | Management Review Other

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Appendix F
Attachment 5

Developed By / Drawn By	**A-E DAILY QUALITY CONTROL REPORT**	Drawing Number 4077.0191
Approved By / Date 8/12/96	TREATABILITY STUDY AMERICAN CHEMICAL SERVICE, INC. NPL SITE GRIFFITH, INDIANA	**MONTGOMERY WATSON**
Reference		
Revisions		

APPENDIX G

STANDARD OPERATING PROCEDURES FOR WELL, PIEZOMETER, AND VAPOR PROBE INSTALLATION

STANDARD OPERATING PROCEDURES

DRILLING AND SOIL SAMPLING

STANDARD OPERATING PROCEDURES

DRILLING AND SOIL SAMPLING

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3.0	DRILLING AND SOIL SAMPLING		1
3.1	Drilling Equipment and Procedures		1
3.2	Sampling Procedures		
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1.0 INTRODUCTION

The purpose of this SOP is to describe the procedures and equipment to be used for drilling and soil sampling with hollow stem augers and split spoon samplers during this project.

2.0 RESPONSIBILITIES

Project Manager: Selects site specific soil sampling methods with input from the FTL and site geologist. Oversees and/or prepares drilling subcontracts.

Site Geologist: Selects site specific drilling/sampling options. Helps prepare technical provisions of drilling subcontracts.

Field Team Leader: Implements selected drilling program. Aids in the selection of drilling methods and preparation of subcontracts.

Rig Geologist: Supervises and/or performs actual sampling procedures.

3.0 DRILLING AND SOIL SAMPLING

3.1. Drilling Equipment and Procedures. Hollow stem auger drilling will be used to drill the boreholes for the extraction wells, vapor monitoring probes, and groundwater monitoring wells. Each borehole will be drilled from the ground surface to the depth specified in the FSP. The borehole will be drilled with 7 3/4-inch OD hollow-stem augers. The augers will consist of 5-foot lengths of 3 3/4-inch inside diameter (ID) pipe with 2-inch wide auger flights welded in spirals around the outside of the pipe. Sections of auger will be joined together as the hole is advanced. The boreholes will have an effective diameter of approximately 8 inches. A center plug will be used to prevent liquefied sands from entering the inside of the auger string as the borehole is advanced. No circulating fluid, drilling muds, or other additives will be used during hollow stem auger drilling.

3.2. Sampling Procedures

3.2.1. Soil Sampling. Each borehole will be continuously sampled using a 2-foot long, 2-inch diameter split-spoon sampler. A catcher will be placed at the end of the sampler

so that saturated, unconsolidated soils are not lost as the sampler is retrieved from the borehole. The sampler will be advanced by blows from a standard 140 pound hammer falling 30 inches. The number of blows required to drive the sampler will be recorded on the Soil Boring Log Form provided at the end of this SOP. At the depths that soil samples are scheduled for laboratory analyses, stainless steel spoons or trowels will be used to remove the soil from the sampler and place the soil into the appropriate sample container as described below. All soils retrieved from the sampler will be screened for VOCs and described according to the Unified Soil Classification System (USCS).

3.2.2. Sample Collection Procedures. After the sampler is retrieved from the borehole the soil in the sampler tip will be screened for the presence of VOCs using an organic vapor meter. The maximum meter reading will be recorded on the Soil Boring Log Form provided at the end of this SOP The soil in the sampler will be inspected for obvious signs of contamination (i.e., discolored soil or strong odors). If the soil in the sampler is from a scheduled laboratory sample interval then the soil will be placed in the appropriate sample containers. Soil for VOC analyses will be removed as quickly as possible from the sampler using a stainless steel spoon or trowel, and placed directly into a chilled air tight VOC container. The container will be filled so that there is no head space. The appropriate information will be entered on the sample label and the sample will then be placed in a cooler maintained at a temperature of 4°C. If a blind duplicate sample also is scheduled to be collected, soil adjoining the soil collected for the environmental samples will also be collected at this time and preserved using the procedures discussed above.

After the samples for VOC and SVOC have been collected, a portion of the remaining sample will be homogenized by placing the soil in a stainless steel bowl and mixing it with a stainless steel spoon. The homogenized soil sample will then be divided into the remaining sample containers. The scheduled analyses and sample containers to be used for the soil samples are summarized in the QAPP. All samples scheduled for laboratory analysis will be labeled, handled, and shipped according to the procedures outlined in the QAPP.

The soil remaining in the sampler will then be described according to the Unified Soil Classification System (USCS), as described in the SOP for Soil Logging in Appendix G. The soil will be classified based on grain size, degree of sorting, stiffness, plasticity, and density. The soil description will also include Munsell color (wet), soil particle angularity, moisture content, and visual signs of contamination. All lithologies data will be recorded on the Monitoring Well/Soil Boring Log Form (Attachment 1).

Documentation procedures are discussed in more detail in the SOP for Field Documentation in Appendix F.

3.2.3. Soil pH Analysis. After collecting the sample for laboratory analyses and completing the USCS soil description, a field pH analysis will be conducted on the soil most representative of the soil remaining in the sampler. The field pH analysis will be conducted using a Hach Kit (or equivalent) in accordance with the manufacturers instruction manual. Each field pH kit is equipped with a calibrated container with fill lines for soil and water. Soil is placed in the container to the soil fill line for a standard volume. Water is added to the container to the water fill line. The soil and water are then mixed and allowed to equilibrate for a time specified by the manufacturer. A pH probe is then lowered into the soil-water mixture and the pH of the mixture is recorded on the field form.

3.2.4. Physical Soil Sampling. In addition to the soil samples collected for chemical analysis, samples for physical analyses will also be collected. These samples will be collected from approximately the same locations that samples are collected for chemical analyses. Physical soil samples will be collected for moisture content and particle size (sieve and hydrometer) analyses. The project specific Chain of Custody will be filled out according to the procedures outlined in the QAPP and will accompany the sample cooler at all times.

3.2.5. Sample Containers and Preservation. A summary of the sample containers and the preservation required for each of the scheduled chemical analysis is presented in the QAPP, which identifies the analyses that will be performed on the soil from each sample container. The soil sample containers for VOC and BNAE analyses will be stored at 4°C in a cooler containing double bagged ice prior to sample collection. Sample labeling and handling procedures will be described in more detail in the QAPP.

3.2.6. Equipment Calibration Procedures. The organic vapor meter used to screen the soil samples for VOCs will be calibrated on a daily basis prior to use, as described in the SOP for Operating and Calibration Procedures for Field Equipment in Appendix F. The FID will be recalibrated any time that drift is suspected, and the calibration will be checked at the end of each day of use. All of the calibration information will be recorded on the Equipment Calibration Form provided in the QAPP.

4.0 ATTACHMENTS

1 - Monitoring Well/Soil Boring Log Form (2 pages)

2 - ASTM D 1586-84

QUALITY CONTROL | Graphic Standards / Lead Professional | Technical Review / Project Manager | Management Review / Other

BORING LOCATION

Project:
Date Drilled: _____ Date Completed: _____
Logged By: _____

Water Elevation (ft.): _____
Date Measured: _____

Total Depth (ft.) : _____
Diameter (in.): _____

Monitoring Well No.: _____
Northing: _____ Easting: _____

Ground Surface Elevation (ft.): _____
Measuring Point (MP) Elevation (ft.): _____
MP is Ground Surface Datum: NGVD (1929)

Drilling Contractor: _____
Drilling Method: _____

Well Screen: Diameter _____ Depth _____ Slot Size _____
Casing: Diameter _____ Length _____ Type _____
Sand _____ Bentonite Seal _____ Cement Grout Seal _____

DEPTH (FEET)	GRAIN SIZE: % GRAVEL	% SAND	% FINES	MAX. PID READING (ppm)	BLOWS (6 IN.)	SAMPLE TYPE*	SAMPLE RECOVERY	USCS CLASSIFICATION	LITHOLOGIC DESCRIPTION (USCS name; color; size and angularity of each component or plasticity; density; moisture content; additional facts)	ELEVATION (FEET)
0										
1										
2										
3										
4										

* C California Split Spoon Sampler (2.5" I.D.)
S Standard penetration test sampler
c Cuttings
▼ Elevation of ground water

PAGE 1 OF ____

Appendix G
Attachment 1

This document has been developed for a specific application and may not be used without the written approval of Montgomery Watson

Developed By	Drawn By.	**MONITORING WELL/ SOIL BORING LOG FORM**
Approved By	Date 8/12/96	TREATABILITY STUDY AMERICAN CHEMICAL SERVICE, INC. NPL SITE GRIFFITH, INDIANA
Reference		
Revisions		Drawing Number 4077.0191 **MONTGOMERY WATSON**

QUALITY CONTROL | Graphic Standards — Lead Professional | Technical Review — Project Manager | Management Review — Other

Project:
Monitoring Well No.:_____

DEPTH (FEET)	GRAIN SIZE % GRAVEL	% SAND	% FINES	MAX. PID READING (ppm)	BLOWS (6 IN.)	SAMPLE TYPE*	SAMPLE RECOVERY	USCS CLASSIFICAT.	LITHOLOGIC DESCRIPTION (USCS name; color; size and angularity of each component or plasticity; density; moisture content; additional facts)	ELEVATION (FEET)
5										
6										
7										
8										
9										
0										
1										
2										
3										
4										

PAGE ____ OF ____

Appendix G
Attachment 1

This document has been developed for a specific application and may not be used without the written approval of Montgomery Watson.

Developed By	Drawn By	**MONITORING WELL/ SOIL BORING LOG FORM**	Drawing Number 4077.0191
Approved By	Date 8/12/96		
Reference		TREATABILITY STUDY AMERICAN CHEMICAL SERVICE, INC. NPL SITE GRIFFITH, INDIANA	**MONTGOMERY WATSON**
Revisions			

ATTACHMENT 2

ASTM DESIGNATION: D1586-84

Standard Test Method for Penetration Test and Split-Barrel Sampling of Soils

Taken From: 1991 ANNUAL BOOK OF ASTM STANDARDS - Section 4, Construction, Volume 04.08.

Designation: D 1586 – 84 (Reapproved 1992)[1]

Standard Test Method for Penetration Test and Split-Barrel Sampling of Soils[1]

This standard is issued under the fixed designation D 1586; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

This standard has been approved for use by agencies of the Department of Defense. Consult the DOD Index of Specifications and Standards for the specific year of issue which has been adopted by the Department of Defense.

[1]NOTE—Editorial changes were made throughout October 1992.

1. Scope

1.1 This test method describes the procedure, generally known as the Standard Penetration Test (SPT), for driving a split-barrel sampler to obtain a representative soil sample and a measure of the resistance of the soil to penetration of the sampler.

1.2 *This standard does not purport to address all of the safety problems, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.* For a specific precautionary statement, see 5.4.1.

1.3 The values stated in inch-pound units are to be regarded as the standard.

2. Referenced Documents

2.1 *ASTM Standards:*

D 2487 Test Method for Classification of Soils for Engineering Purposes[2]

D 2488 Practice for Description and Identification of Soils (Visual-Manual Procedure)[2]

D 4220 Practices for Preserving and Transporting Soil Samples[2]

D 4633 Test Method for Stress Wave Energy Measurement for Dynamic Penetrometer Testing Systems[2]

3. Terminology

3.1 *Descriptions of Terms Specific to This Standard*

3.1.1 *anvil*—that portion of the drive-weight assembly which the hammer strikes and through which the hammer energy passes into the drill rods.

3.1.2 *cathead*—the rotating drum or windlass in the rope-cathead lift system around which the operator wraps a rope to lift and drop the hammer by successively tightening and loosening the rope turns around the drum.

3.1.3 *drill rods*—rods used to transmit downward force and torque to the drill bit while drilling a borehole.

3.1.4 *drive-weight assembly*—a device consisting of the hammer, hammer fall guide, the anvil, and any [illegible] drop system.

3.1.5 *hammer*—that portion of the drive-weight assembl consisting of the 140 ± 2 lb (63.5 ± 1 kg) impact weigh which is successively lifted and dropped to provide th energy that accomplishes the sampling and penetration.

3.1.6 *hammer drop system*—that portion of the drive weight assembly by which the operator accomplishes th lifting and dropping of the hammer to produce the blow.

3.1.7 *hammer fall guide*—that part of the drive-weigh assembly used to guide the fall of the hammer.

3.1.8 *N-value*—the blowcount representation of the pen tration resistance of the soil. The *N*-value, reported in blow per foot, equals the sum of the number of blows required drive the sampler over the depth interval of 6 to 18 in. (1 to 450 mm) (see 7.3).

3.1.9 Δ*N*—the number of blows obtained from each the 6-in. (150-mm) intervals of sampler penetration (see 7.

3.1.10 *number of rope turns*—the total contact ang between the rope and the cathead at the beginning of t operator's rope slackening to drop the hammer, divided 360° (see Fig. 1).

3.1.11 *sampling rods*—rods that connect the drive-weig assembly to the sampler. Drill rods are often used for th purpose.

3.1.12 *SPT*—abbreviation for Standard Penetration Te a term by which engineers commonly refer to this metho

4. Significance and Use

4.1 This test method provides a soil sample for identifi tion purposes and for laboratory tests appropriate for s obtained from a sampler that may produce large shear str disturbance in the sample.

4.2 This test method is used extensively in a great vari of geotechnical exploration projects. Many local correlati and widely published correlations which relate SPT blo count, or *N*-value, and the engineering behavior of ear works and foundations are available.

5. Apparatus

5.1 *Drilling Equipment*—Any drilling equipment t provides at the time of sampling a suitably clean open h before insertion of the sampler and ensures that the pene tion test is performed on undisturbed soil shall be accepta The following pieces of equipment have proven to

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[1] This method is under the jurisdiction of ASTM Committee D-18 on Soil and Rock, and is the direct responsibility of Subcommittee D18.02 on Sampling and Related Field Testing for Soil Investigations.

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[2] *Annual Book of ASTM Standards*, Vol 04.08.

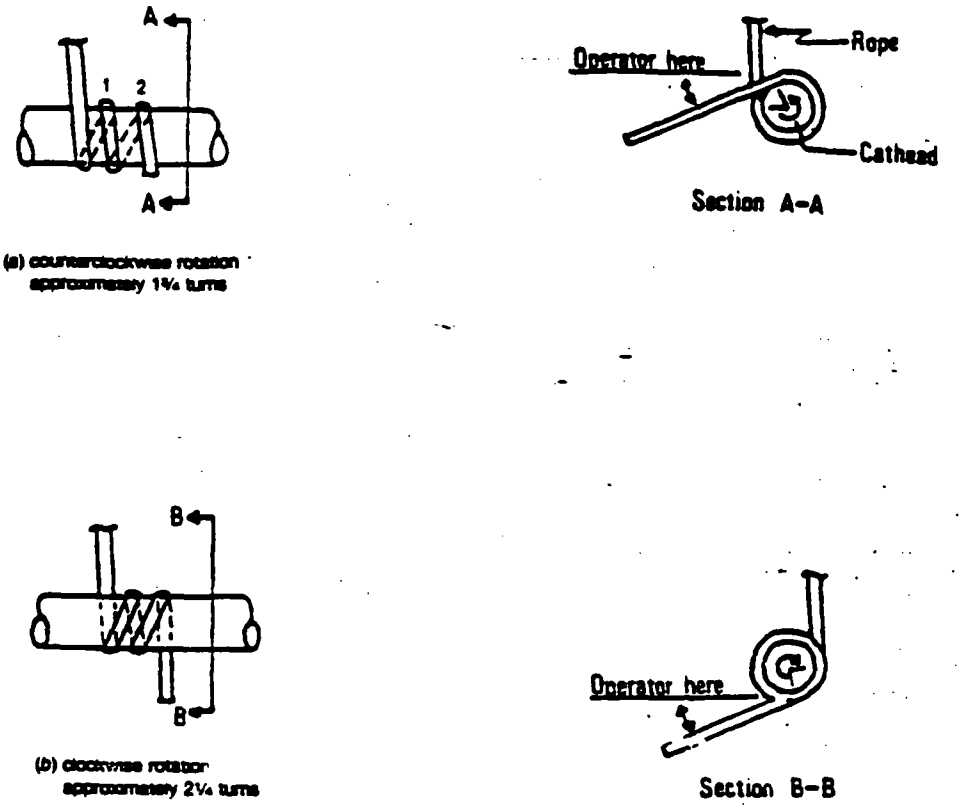


FIG. 1 Definitions of the Number of Rope Turns and the Angle for (*a*) Counterclockwise Rotation and (*b*) Clockwise Rotation of the Cathead

suitable for advancing a borehole in some subsurface conditions.

5.1.1 *Drag, Chopping, and Fishtail Bits*, less than 6.5 in. (162 mm) and greater than 2.2 in. (56 mm) in diameter may be used in conjuction with open-hole rotary drilling or casing-advancement drilling methods. To avoid disturbance of the underlying soil, bottom discharge bits are not permitted; only side discharge bits are permitted.

5.1.2 *Roller-Cone Bits*, less than 6.5 in. (162 mm) and greater than 2.2 in. (56 mm) in diameter may be used in conjunction with open-hole rotary drilling or casing-advancement drilling methods if the drilling fluid discharge is directed.

5.1.3 *Hollow-Stem Continuous Flight Augers*, with or without a center bit assembly, may be used to drill the boring. The inside diameter of the hollow-stem augers shall be less than 6.5 in. (162 mm) and greater than 2.2 in. (56 mm).

5.1.4 *Solid, Continuous Flight, Bucket and Hand Augers*, less than 6.5 in. (162 mm) and greater than 2.2 in. (56 mm) in diameter may be used if the soil on the side of the boring does not cave onto the sampler or sampling rods during sampling.

5.2 *Sampling Rods*—Flush-joint steel drill rods shall be used to connect the split-barrel sampler to the drive-weight assembly. The sampling rod shall have a stiffness (moment of inertia) equal to or greater than that of parallel wall "A" rod (a steel rod which has an outside diameter of 1⅝ in. (41.2 mm) and an inside diameter of 1⅛ in. (28.5 mm).

NOTE 1—Recent research and comparative testing indicates that rod used, with stiffness ranging from "A" size rod to "N" size rod, usually have a negligible effect on the *N*-values to depths of at least 100 ft (30 m).

5.3 *Split-Barrel Sampler*—The sampler shall be constructed with the dimensions indicated in Fig. 2. The drive shoe shall be of hardened steel and shall be replaced or repaired when it becomes dented or distorted. The use of liners to produce a constant inside diameter of 1⅜ in. (35 mm) is permitted, but shall be noted on the penetration record if used. The use of a sample retainer basket is permitted, and should also be noted on the penetration record if used.

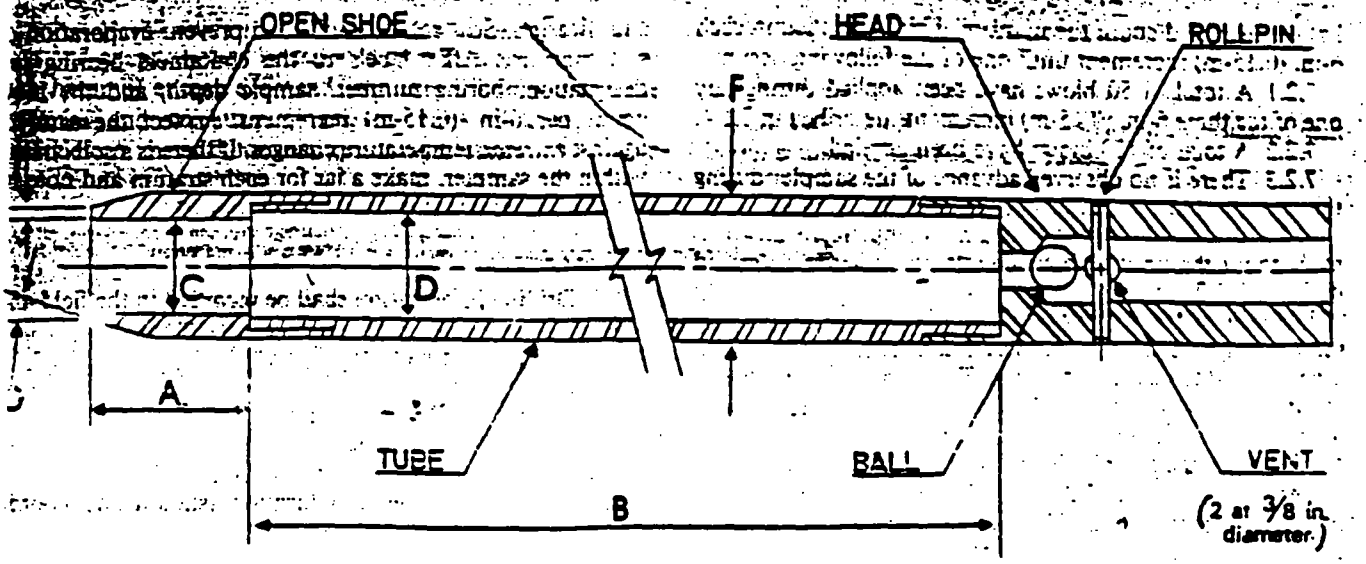
NOTE 2—Both theory and available test data suggest that *N*-values may increase between 10 to 30 % when liners are used.

5.4 *Drive-Weight Assembly*:

5.4.1 *Hammer and Anvil*—The hammer shall weigh 140 ± 2 lb (63.5 ± 1 kg) and shall be a solid rigid metallic mass. The hammer shall strike the anvil and make steel on steel contact when it is dropped. A hammer fall guide permitting free fall shall be used. Hammers used with the cathead and rope method shall have an unimpeded overlift capacity of at least 4 in. (100 mm). For safety reasons, the use of a hammer assembly with an internal anvil is encouraged.

NOTE 3—It is suggested that the hammer fall guide be permanently marked to enable the operator or inspector to judge the hammer height.

5.4.2 *Hammer Drop System*—Rope-cathead, trip, automatic, or automatic hammer drop systems may be used providing the lifting apparatus will not cause penet-



= 1.0 to 2.0 in. (25 to 50 mm)
= 18.0 to 30.0 in. (0.457 to 0.762 m)
= 1.375 ± 0.005 in. (34.93 ± 0.13 mm)
= 1.50 ± 0.05 − 0.00 in. (38.1 ± 1.3 − 0.0 mm)
= 0.10 ± 0.02 in. (2.54 ± 0.25 mm)
= 2.00 ± 0.05 − 0.00 in. (50.8 ± 1.3 − 0.0 mm)
= 16.0° to 23.0°

The 1⅛ in. (38 mm) inside diameter split barrel may be used with a 16-gage wall thickness split liner. The penetrating end of the drive shoe may be slightly rounded. Metal or plastic retainers may be used to retain soil samples.

FIG. 2 Split-Barrel Sampler

he sampler while re-engaging and lifting the hammer.

5.5 *Accessory Equipment*—Accessories such as labels, ample containers, data sheets, and groundwater level measuring devices shall be provided in accordance with the requirements of the project and other ASTM standards.

. Drilling Procedure

6.1 The boring shall be advanced incrementally to permit intermittent or continuous sampling. Test intervals and locations are normally stipulated by the project engineer or geologist. Typically, the intervals selected are 5 ft (1.5 mm) or less in homogeneous strata with test and sampling locations at every change of strata.

6.2 Any drilling procedure that provides a suitably clean and stable hole before insertion of the sampler and assures that the penetration test is performed on essentially undisturbed soil shall be acceptable. Each of the following procedures have proven to be acceptable for some subsurface conditions. The subsurface conditions anticipated should be considered when selecting the drilling method to be used.

6.2.1 Open-hole rotary drilling method.

6.2.2 Continuous flight hollow-stem auger method.

6.2.3 Wash boring method.

6.2.4 Continuous flight solid auger method.

6.3 Several drilling methods produce unacceptable borings. The process of jetting through an open tube sampler and then sampling when the desired depth is reached shall not be permitted. The continuous flight solid auger method shall not be used for advancing the boring below a water table or below the upper confining bed of a confined non-cohesive stratum that is under artesian pressure. Casing may not be advanced below the sampling elevation prior to sampling. Advancing a boring with bottom discharge bits is not permissible. It is not permissible to advance the boring for subsequent insertion of the sampler solely by means of previous sampling with the SPT sampler.

6.4 The drilling fluid level within the boring or hollow-stem augers shall be maintained at or above the in situ groundwater level at all times during drilling, removal of drill rods, and sampling.

7. Sampling and Testing Procedure

7.1 After the boring has been advanced to the desired sampling elevation and excessive cuttings have been removed, prepare for the test with the following sequence of operations.

7.1.1 Attach the split-barrel sampler to the sampling rods and lower into the borehole. Do not allow the sampler to drop onto the soil to be sampled.

7.1.2 Position the hammer above and attach the anvil to the top of the sampling rods. This may be done before the sampling rods and sampler are lowered into the borehole.

7.1.3 Rest the dead weight of the sampler, rods, anvil, and drive weight on the bottom of the boring and apply a seating blow. If excessive cuttings are encountered at the bottom of the boring, remove the sampler and sampling rods from the boring and remove the cuttings.

7.1.4 Mark the drill rods in three successive 6-in. (0.15-m) increments so that the advance of the sampler under the impact of the hammer can be easily observed for each 6-in. (0.15-m) increment.

7.2 Drive the sampler with blows from the 140-lb (63.5-

kg) hammer and count the number of blows applied in each 6-in. (0.15-m) increment until one of the following occurs:

7.2.1 A total of 50 blows have been applied during any one of the three 6-in. (0.15-m) increments described in 7.1.4.

7.2.2 A total of 100 blows have been applied.

7.2.3 There is no observed advance of the sampler during the application of 10 successive blows of the hammer.

7.2.4 The sampler is advanced the complete 18 in. (0.45 m) without the limiting blow counts occurring as described in 7.2.1, 7.2.2, or 7.2.3.

7.3 Record the number of blows required to effect each 6 in. (0.15 m) of penetration or fraction thereof. The first 6 in. is considered to be a seating drive. The sum of the number of blows required for the second and third 6 in. of penetration is termed the "standard penetration resistance," or the "*N*-value." If the sampler is driven less than 18 in. (0.45 m), as permitted in 7.2.1, 7.2.2, or 7.2.3, the number of blows per each complete 6-in. (0.15-m) increment and per each partial increment shall be recorded on the boring log. For partial increments, the depth of penetration shall be reported to the nearest 1 in. (25 mm), in addition to the number of blows. If the sampler advances below the bottom of the boring under the static weight of the drill rods or the weight of the drill rods plus the static weight of the hammer, this information should be noted on the boring log.

7.4 The raising and dropping of the 140-lb (63.5-kg) hammer shall be accomplished using either of the following two methods:

7.4.1 By using a trip, automatic, or semi-automatic hammer drop system which lifts the 140-lb (63.5-kg) hammer and allows it to drop 30 ± 1.0 in. (0.76 m ± 25 mm) unimpeded.

7.4.2 By using a cathead to pull a rope attached to the hammer. When the cathead and rope method is used the system and operation shall conform to the following:

7.4.2.1 The cathead shall be essentially free of rust, oil, or grease and have a diameter in the range of 6 to 10 in. (150 to 250 mm).

7.4.2.2 The cathead should be operated at a minimum speed of rotation of 100 RPM, or the approximate speed of rotation shall be reported on the boring log.

7.4.2.3 No more than 2¼ rope turns on the cathead may be used during the performance of the penetration test, as shown in Fig. 1.

NOTE 4—The operator should generally use either 1¾ or 2¼ rope turns, depending upon whether or not the rope comes off the top (1¾ turns) or the bottom (2¼ turns) of the cathead. It is generally known and accepted that 2¼ or more rope turns considerably impedes the fall of the hammer and should not be used to perform the test. The cathead rope should be maintained in a relatively dry, clean, and unfrayed condition.

7.4.2.4 For each hammer blow, a 30-in. (0.76-m) lift and drop shall be employed by the operator. The operation of pulling and throwing the rope shall be performed rhythmically without holding the rope at the top of the stroke.

7.5 Bring the sampler to the surface and open. Record the percent recovery or the length of sample recovered. Describe the soil samples recovered as to composition, color, stratification, and condition, then place one or more representative portions of the sample into sealable moisture-proof containers (jars) without ramming or distorting any apparent stratification. Seal each container to prevent evaporation of soil moisture. Affix labels to the containers bearing job designation, boring number, sample depth, and the blow count per 6-in. (0.15-m) increment. Protect the sample against extreme temperature changes. If there is a soil change within the sampler, make a jar for each stratum and note its location in the sampler barrel.

8. Report

8.1 Drilling information shall be recorded in the field and shall include the following:

8.1.1 Name and location of job,

8.1.2 Names of crew,

8.1.3 Type and make of drilling machine,

8.1.4 Weather conditions,

8.1.5 Date and time of start and finish of boring,

8.1.6 Boring number and location (station and coordinates, if available and applicable),

8.1.7 Surface elevation, if available,

8.1.8 Method of advancing and cleaning the boring,

8.1.9 Method of keeping boring open,

8.1.10 Depth of water surface and drilling depth at the time of a noted loss of drilling fluid, and time and date when reading or notation was made,

8.1.11 Location of strata changes,

8.1.12 Size of casing, depth of cased portion of boring,

8.1.13 Equipment and method of driving sampler,

8.1.14 Type sampler and length and inside diameter of barrel (note use of liners),

8.1.15 Size, type, and section length of the sampling rods, and

8.1.16 Remarks.

8.2 Data obtained for each sample shall be recorded in the field and shall include the following:

8.2.1 Sample depth and, if utilized, the sample number,

8.2.2 Description of soil,

8.2.3 Strata changes within sample,

8.2.4 Sampler penetration and recovery lengths, and

8.2.5 Number of blows per 6-in. (0.15-m) or partial increment.

9. Precision and Bias

9.1 *Precision*—A valid estimate of test precision has not been determined because it is too costly to conduct the necessary inter-laboratory (field) tests. Subcommittee D18.02 welcomes proposals to allow development of a valid precision statement.

9.2 *Bias*—Because there is no reference material for this test method, there can be no bias statement.

9.3 Variations in *N*-values of 100 % or more have been observed when using different standard penetration test apparatus and drillers for adjacent borings in the same soil formation. Current opinion, based on field experience, indicates that when using the same apparatus and driller, *N*-values in the same soil can be reproduced with a coefficient of variation of about 10 %.

9.4 The use of faulty equipment, such as an extremely massive or damaged anvil, or rusty cathead, a low speed cathead, an old, oily rope, or massive or poorly lubricated rope sheaves can significantly contribute to differences in *N*-values obtained between operator-drill rig systems.

9.5 The variability in *N*-values produced by different drill rigs and operators may be reduced by measuring that part of the hammer energy delivered into the drill rods from the sampler, and adjusting *N* on the basis of comparative tests. A method for energy measurement and *N*-value adjustment is given in Test Method D 4633.

10. Keywords

10.1 blow count; in-situ test; penetration resistance; split-barrel sampling; standard penetration test

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STANDARD OPERATING PROCEDURES

WELL, PIEZOMETER, AND VAPOR PROBE DESIGN AND INSTALLATION

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1.0 INTRODUCTION

This guideline is applicable to the design and installation of groundwater monitoring wells and piezometers at hazardous waste sites. Additionally, some of the information in this guideline is applicable to the design and installation of vapor extraction wells, air injection wells, and soil vapor monitoring probes. Specifics for all well and vapor probe design are included in the FSP in both text and figures. Each well must be designed to suit the hydrogeologic setting, the type of contaminants to be monitored, the overall purpose of the monitoring program, and other site-specific variables. As such, site-specific objectives for each monitoring well and its respective intended use must be clearly defined before the monitoring system is designed. Additionally, within a monitoring system, different monitoring wells may serve different purposes and thus require different types of construction. Therefore, during all phases of well design attention must be given to clear documentation of the basis for design decisions, the details of well construction, and the materials to be used.

2.0 WELL DESIGN

Consideration should be given to the following site-specific information before a groundwater monitoring system is designed:

- Purpose of the groundwater monitoring program (water quality, water levels, remediation, flow direction, and velocities);
- Surficial conditions, including topography, climate, drainage, site access;
- Known or anticipated hydrogeologic setting including geology (consolidated/unconsolidated), physical characteristics of the aquifer (porosity/permeability), type of aquifer (confined/unconfined), recharge/discharge conditions, aquifer thickness, and groundwater/surface water interrelationships;
- Borehole geophysical logs, if any;
- Known or anticipated contaminant chemical characteristics (chemistry, density, viscosity, reactivity, and concentration);
- Anthropogenic or tidal influences; and
- Regulatory requirements.

Common mistakes in groundwater monitoring system design include:

- Use of well casing or well screen materials that are incompatible with the hydrogeologic environment, and/or the anticipated contaminants, resulting in chemical alteration of the samples or failure of the well;
- Use of nonstandard well screen (field slotted or perforated) or incorrect slot size, resulting in well sedimentation and turbid groundwater samples;
- Improper length or placement of the well screen so that acquisition of accurate water level or water quality data from discrete zones is impossible;
- Improper selection and placement of filter pack materials resulting in well sedimentation, well screen plugging, or chemical alteration of the groundwater;
- Improper selection and placement of annular seal materials resulting in alteration of groundwater chemistry, plugging of the filter pack and/or well screen, or cross-contamination from geologic units that have been sealed off improperly; and
- Inadequate surface protection resulting in surface water entering the well.

Siting of monitoring wells should be performed after a preliminary estimation of the hydraulic gradients and groundwater flow direction. In most cases this may be done through review of background data and site terrain. Additionally, production wells in the area may be used to assess the local groundwater flow direction. If the groundwater flow direction cannot be determined by any of these methods, it may be practical to install piezometers in a preliminary phase to determine flow direction.

2.1 CASING DIAMETER AND SCREEN LENGTH

Monitoring well casing diameter is dependent on the purpose of the well and the amount and size of downhole equipment that must be accommodated. Additional criteria for selecting casing diameters include: drilling or well installation method used, anticipated depth of the well and associated strength requirements, ease of well development, volume of water required to be purged prior to sampling, rate of recovery of the well after purging, and cost.

Monitoring well casing diameters are generally 2 or 4 inches. Pumping tests or some types of borehole geophysical equipment may require wells 6 inches or larger in diameter. Four-inch-diameter wells are usually preferred due to their versatility. In smaller diameter wells, the volume of stagnant water to be purged prior to sampling is minimized, the cost of well construction is reduced, and the well stabilizes relatively

quickly. The quantities of potentially contaminated drill cuttings and development and purge water are also reduced.

The borehole diameter should be a minimum of 4 to 6 inches larger than the well casing and screen to allow for proper placement of annular materials.
In situations where groundwater flow lines are close to horizontal, screen lengths are typically 10 to 20 feet, with heterogeneous formations requiring shorter screen lengths. If non-aqueous phase liquids (NAPLs) that are lighter than water are anticipated, the well screen should extend above the water table so these liquids can be sampled. Consideration should be given to seasonal fluctuations in water levels when locating the well screen above the top of the water table. If dense NAPLs are anticipated, the screen interval should extend to the base of the aquifer. Well clusters may be necessary when contaminants both denser and lighter than water are anticipated in the same aquifer.

2.2 CASING AND SCREEN MATERIALS

Monitoring well casing is specified by diameter, thickness, and type of material. Casing thickness is referred to as "schedule." Polyvinyl chloride (PVC) is usually Schedule 40 (thinner wall), although Schedule 80 (thicker wall) is sometimes used. Steel casing is typically Schedule 5 or 10.

Selection of casing and screen material must be based on three primary characteristics: chemical interference potential, chemical resistance, and physical strength. The materials must not assimilate chemicals either by adsorption onto the material surface or absorption into the material matrix or pores; they must be durable enough to withstand potential chemical attacks either from natural chemical constituents or groundwater contaminants; and they must have the structural strength to withstand the forces exerted on them by the surrounding geologic materials and during installation. The three components of casing and screen structural strength are tensile strength, compressive (column) strength, and collapse strength.

Casing and screen materials generally available are Teflon, PVC, stainless steel, galvanized steel, carbon steel, and low-carbon steel. Teflon materials are extremely expensive and of comparatively low strength. Although relatively inert, recent studies have shown that Teflon is prone to sorption of selected organic compounds.

The two most commonly used materials are PVC and stainless steel. PVC is inexpensive, widely available, lightweight, and easy to work with. Many studies have been conducted concerning the effect of PVC on water quality data. Whereas adsorption of some chlorinated species to PVC was documented, the adsorption rate was found to be very slow. Because a sample is generally taken shortly after the purging of stagnant water in contact with the casing, the contaminants in the water will have minimal time to be influenced by sorption or leaching effects. Therefore, potential sample bias effects due to interactions with PVC appear to be negligible. The column strength of PVC may limit the depth of installation. Schedule 80 PVC may be used for deeper wells; however, the reduced inside diameter should be taken into account when designing the well.

Steel well materials are stronger, more rigid, and less temperature sensitive than PVC or Teflon. Stainless steel has the highest corrosion resistance of the various types of steel. Type 304 and Type 316 are the most commonly used stainless steels. Both are available in low-carbon forms, which are more easily welded than the normal carbon steel. Low-carbon steel is designated by an "L" after the number (e.g., Type 304L). Type 304 stainless steel is superior to Type 316 from a corrosion resistance and cost standpoint. Type 316 is preferred to Type 304 under reducing conditions. For either type of stainless steel, long-term exposure to corrosive conditions may result in chromium or nickel contamination of groundwater samples. Insoluble halogen and sulfur compounds may also form as a result of corrosion of stainless steel.

Threaded, flush-joint casing is preferred for monitoring well applications. Welded-joint steel casing may also be acceptable, but is typically more expensive and inconvenient. Glued PVC should never be used for monitoring wells; the glue may release organic contamination into the well. The casing should have a well cap that is vented to prevent the accumulation of gases and to allow water levels in the well to respond to barometric and hydraulic pressure changes.

The hydraulic efficiency of a well screen depends primarily upon the amount of open area available per unit length of screen. The two screen types commonly used for monitoring wells are machine-slotted, and continuous-slot wire-wound. Hand-slotted, drilled, or perforated casings should not be used as well screens. Slotted casing is manufactured from a variety of materials, including PVC and stainless steel.

Slot openings are designated by numbers that correspond to the widths of the openings in thousandths of an inch (e.g., number 10 slot refers to 0.010 inch slot size). The slots have

a consistent width for the entire wall thickness of the casing, which can result in clogging if irregularly shaped formation particles are brought through the screen during well development and sampling.

The continuous-slot, wire-wound screen has a greater area per opening per length and diameter than is available with any other screen type. The percentage of open area in continuous-slot screen is often more than twice that provided by standard slotted well screen. The triangular shaped wire makes these screens nonclogging. They are fabricated in PVC and a variety of metals.

If a monitoring well will also be used for hydraulic testing, the well screen open area should equal or exceed the formation's effective porosity so that the screen is not the limiting factor in formation hydraulic testing. In most cases, this amount of open area can only be achieved through the use of continuous-slot wire-wound well screen. In choosing between types of well screens, another factor is the speed and effectiveness of well development. Screens with a high percentage of open area greatly reduce the time and effort required for well development.

The bottom of the screen must be sealed by an endcap consisting of the same material as the screen. The use of a sediment sump or trap below the well screen is not appropriate for monitoring wells.

2.3 DECONTAMINATION OF CASING AND SCREEN MATERIALS

During the production of PVC casing, a wax layer can develop on the inner wall of the casing; protective coatings may also be added to enhance casing durability. Considerable quantities of oils and solvents are used during the manufacturing and machining of threads during the production of steel casing. All of these represent potential sources of chemical interference and must be removed with either a laboratory-grade nonphosphate solution or by steam cleaning prior to installation. Factory cleaning of casing and screen in a controlled environment by standard detergent washing, rinsing, and air-drying procedures is superior to any cleaning efforts attempted in the field. Factory cleaned and sealed casing and screen can be certified by the supplier.

2.4 FILTER PACK AND WELL SCREEN DESIGN

A properly designed monitoring well requires that a well screen be placed opposite the zone to be monitored and be surrounded by materials that are coarser and of greater hydraulic conductivity than the natural formation material. Naturally developed wells and wells with artificially introduced filter pack are the two basic types of well intake designs for unconsolidated or poorly consolidated materials.

2.4.1 Naturally Developed Wells

In naturally developed wells, the formation materials are allowed to collapse around the well screen. Naturally developed wells can be installed in which natural formation materials are relatively coarse grained, permeable, and of uniform grain size. It is essential that the grain-size distribution of the formation to be monitored is accurately determined by conducting a mechanical (sieve) analysis of samples taken from the interval to be screened. After sieving, a plot of grain size versus cumulative percentage of sample retained on each sieve is made. Well screen slot sizes are based on the grain-size distribution, specifically the effective size (the sieve size that retains 90 percent of the formation material, referred to as D10) and the uniformity coefficient (the ratio of the sieve size that retains 40 percent of the material or D60, to the effective size). A naturally developed well can be justified if the effective grain size is greater than 0.010 inch and the uniformity coefficient is greater than 3.0. The California Department of Toxic Substances Control (DTSC) recommends that an artificial filter pack be used if sieve analysis indicates that a screen slot size of 0.020 inches or less is required to retain 50 percent of the natural formation. The biggest drawback for naturally developed wells is the time required for well development to remove fine-grained formation material.

2.4.2 Artificially Filter-Packed Wells

Filter packs are installed to create a permeable envelope around the well screen. The use of an artificial filter pack in a fine-grained formation material allows the screen slot size to be considerably larger than if the screen were placed in the formation material without the filter pack. The selection of the filter pack grain size should be based on the grain size of the finest layer to be screened.

Filter pack grain size and well screen slot size should be determined by the grain size distribution of the formation material. The filter pack should be designed first. It is

recommended to use a filter pack grain size that is three to five times the average (D50) size of the formation materials. However, this method may be misleading in coarse, well graded formation materials. Another way to determine filter pack grain size is to take the D30 grain size of the formation materials and multiplying it by a factor of between 3 and 6, with 3 used if the formation is fine and uniform and 6 used if the formation is coarse and nonuniform. For both methods, the uniformity coefficient of the filter pack materials should be as close to 1.0 as possible to minimize particle size segregation during filter pack installation.

The filter pack should extend from the bottom of the well screen to approximately 2 to 5 feet above the top of the screen to account for settlement of the pack material during development and to act as a buffer between the well screen and the annular seal. A secondary filter pack is sometimes used to prevent annular seal materials from migrating into the primary filter pack. The secondary filter pack should extend at least 1 foot above the top of the primary filter pack. The D10 size of the secondary filter pack should be between one-third and one-fifth the D10 size of the primary filter pack.

Filter pack thickness must be sufficient to surround the well screen but thin enough to minimize resistance to the flow of fine-grained formation material and water into the well during development. A filter pack thickness of approximately 3 inches is generally suitable.

The materials comprising the filter pack should be as chemically inert as possible. It should be comprised of clean quartz sand or glass beads. Filter pack materials are usually supplied in 100-pound bags; these materials are washed, dried, and factory packaged.

The size of well intake openings can only be selected after the filter-pack grain size is specified. The slot size should be such that 90 percent to 100 percent of the filter-pack material is held back by the well screen.

The casing string should be installed in the center of the borehole. This will allow the filter-pack materials to evenly fill the annular space around the screen and ensure that annular seal materials fill the annular space evenly around the casing. If a hollow-stem auger or dual-tube rig is used, the auger or inner tube of the dual tube will adequately centralize the casing string. For other types of drilling, centralizers should be used to ensure the casing string is positioned in the center of the borehole. Centralizers are typically expandable metal or plastic that attach to the outside of the casing and are

adjustable along the length of the casing. Centralizers are generally attached immediately above the well screen and at 10- or 20-foot intervals along the casing to the surface.

Methods for filter pack emplacement include: 1) gravity (free-fall), 2) tremie pipe, 3) reverse circulation, and 4) backwashing. The latter two techniques are not commonly used for monitoring well construction, since they require the introduction into the borehole of water from a surface source.

Gravity emplacement is only possible in relatively shallow wells with an annular space of more than 2 inches, where the potential occurrence of bridging is minimized. Bridging can result in the occurrence of large unfilled voids in the filter pack or the failure of filter pack materials to reach their intended depth. Gravity emplacement may also cause filter pack gradation. Additionally, formation materials from the borehole wall can become incorporated into the filter pack, potentially contaminating it.

With the tremie emplacement method, the filter pack is poured or slurried into the annular space adjacent to the well screen through a rigid pipe, usually 1.5 inches in diameter. Initially the pipe is positioned so that its end is at the bottom of the annulus. If the filter pack is being installed in a temporarily cased borehole (hollow-stem auger or dual-tube percussion) the temporary casing is pulled to expose the screen as the filter-pack material builds up around the well screen. In unconsolidated formations the temporary casing should only be pulled out 1 to 2 feet at a time to prevent caving. In consolidated or well-cemented formations or in cohesive unconsolidated formations, the temporary casing may be raised well above the bottom of the borehole prior to filter pack emplacement. For deep wells and/or nonuniform filter pack materials, the filter pack may be pressure fed through a tremie pipe with a pump. Emplacement should be continuously monitored with a weighted measuring tape accurate to the nearest 0.1 foot to determine when the filter pack has reached the desired height.

2.5 ANNULAR SEAL

Proper annular seal formulation and placement results in the complete filling of the annular space and envelopes the entire length of the well casing to ensure that no vertical migration can occur within the borehole.

Annular seal materials may include bentonite, neat cement grout, or variations of both. Typically, a bentonite seal from 2 to 5 feet thick is emplaced immediately above the filter

pack. The use of bentonite as a sealing material depends on its efficient hydration following emplacement. Expansion of bentonite in water can be on the order of 8 to 10 times the volume of dry bentonite. This expansion causes the bentonite to provide a tight seal between the casing and the adjacent formation. Bentonite is available as pellets, granules, chips, chunks, or powder. Bentonite pellets expand in water at relatively slow rates, thus reducing the potential for bridging compared to chips, chunks, or granules. If the bentonite seal will be above the saturated zone, several gallons of clean water must be poured down the annulus to begin the hydration process. A minimum of 30 minutes should pass to allow for hydration before additional annular seal materials are placed above the bentonite.

Powdered bentonite is generally made into a grout slurry to allow emplacement as a bentonite seal. This grout slurry is prepared by mixing about 15 pounds of a high-solids, low-viscosity bentonite with 7 gallons of water to yield one cubic foot of grout. Once the grout is mixed, it should remain workable for 15 to 30 minutes. During this time the grout is pumped through a tremie pipe with a mud or grout pump. Once in place, the bentonite grout requires a minimum of 24 hours to strengthen. In water with a high total dissolved solids (TDS) content (>5000 ppm) or a high chloride content, the swelling of bentonite is inhibited.

A neat cement is commonly used to seal the remainder of the annulus. Neat cement is made up of one 94-pound bag of Portland cement and 6 gallons of water. The water used to mix the neat cement should be clean with a TDS <500 ppm. Bentonite powder is often added to neat cement to improve workability and reduce slurry weight and density. The proportion of bentonite by volume should be 3 to 8 percent.

The cement-bentonite grout should be mechanically blended in an aboveground rigid container and pumped through a tremie pipe to within a few inches of the bottom of the space to be sealed. This allows the grout to displace groundwater and loose formation materials up the hole. The end of the tremie pipe should always remain in the grout without allowing air spaces. After emplacement, the tremie pipe should be removed immediately. The grout should be emplaced in one continuous mass before initial setting of the cement or before the mixture loses its fluidity.

Cement is a highly alkaline substance (pH from 10 to 12) and introduces the possibility of altering the chemistry of the water it contacts. Thinner slurries may infiltrate an unprotected filter pack. After a borehole annulus is filled with grout a sample of water

may be obtained and the pH determined in the field. A pH reading of 12 or higher may indicate an invasion of cement grout into the well.

2.6 SURFACE COMPLETIONS

Two types of surface completions are common for groundwater monitoring wells: aboveground and flush-mounted. Aboveground completions are preferred wherever practical. The primary purpose of either type of completion is to prevent surface runoff from entering and infiltrating down the annulus of the well, and to protect the well from accidental damage or vandalism. The surface seal may be an extension of the annular seal installed above the filter pack, or a separate seal emplaced atop the annular seal.

For aboveground completions, a protective steel casing fitted with a locking cover is set into the uncured cement surface seal. Guard posts should be spaced around each well to afford additional protection.

In a flush-to-ground surface completion, a water-tight monitoring well is set into the cement surface seal before it has cured. This type of completion is used in high-traffic areas. A low, gently sloping mound of cement will discourage surface runoff. A locking well cap must be used to secure the inner well casing.

3.0 REFERENCES

National Water Well Association (NWWA), 1989. Handbook of Suggested Practices for the Design and Installation of Ground-Water Monitoring Wells, NWWA, Dublin, Ohio, 398 pp.

Nielson, David M. (ed.), 1991. Practical Handbook of Ground-Water Monitoring, Lewis Publishers, Chelsea, Michigan, 717 pp.

4.0 ATTACHMENTS

3 - Well Completion Form

4 - Monitoring Well and Piezometer Surface Completion Detail

5 - Soil Vapor Monitoring Probe Completion Detail

6 - ASTM Referenced SOP

QUALITY CONTROL | Graphic Standards Lead Professional | Technical Review Project Manager | Management Review Other

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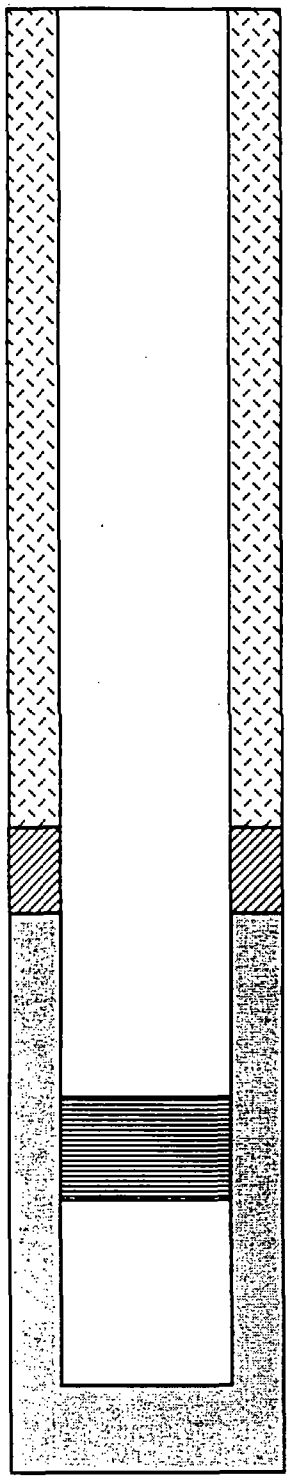


(NOT TO SCALE)

SOIL BORING/WELL NUMBER _____

GEOLOGIST _____

DATE CONSTRUCTION STARTED _____

DATE CONSTRUCTION COMPLETED _____

RELEVANT INFORMATION (Problems, corrective actions)

_____

_____

_____

_____

CASING SCHEDULE:

RISER TYPE _____

RISER DIAMETER _____

RISER LENGTH _____

SCREEN TYPE _____

SCREEN LENGTH _____

SCREEN DIAMETER _____

PROTECTIVE CASING TYPE , LENGTH, DIAMETER _____

_____ CEMENT GROUT INTERVAL

_____ TOP OF BENTONITE SEAL

BENTONITE TYPE _____

_____ TOP OF SAND PACK

SAND SIZE _____

_____ SCREENED INTERVAL
(Beginning and ending depth below ground surface)

SLOT SIZE _____

_____ USCS CLASSIFICATION OF FORMATION MATERIAL IN SCREENED INTERVAL

_____ DEPTH OF CASING
(Below ground surface)

_____ BOREHOLE DEPTH

ANNULAR VOLUME:

$V = \pi H (R_1^2 - R_2^2)$

WHERE:

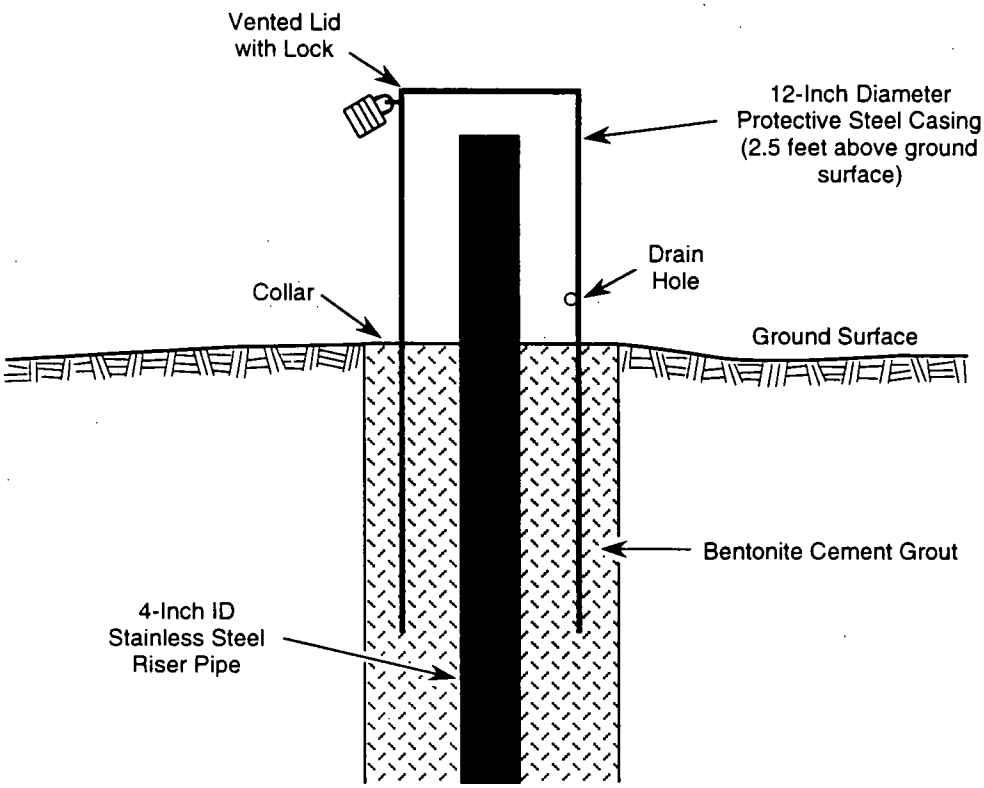
V = Annular Volume (ft^3)
$\pi = 3.142$
H = Length of Interval (ft)
R_1 = Borehole Radius (ft)
R_2 = Well Casing Radius (ft)

CALCULATIONS:

Appendix G
Attachment 3

Developed By	Drawn By	**WELL COMPLETION FORM**	Drawing Number 4077.0191
Approved By	Date 8/12/96		
Reference		TREATABILITY STUDY AMERICAN CHEMICAL SERVICE, INC. NPL SITE GRIFFITH, INDIANA	MONTGOMERY WATSON
Revisions			

QUALITY CONTROL | Graphic Standards / Lead Professional | Technical Review / Project Manager | Management Review / Other

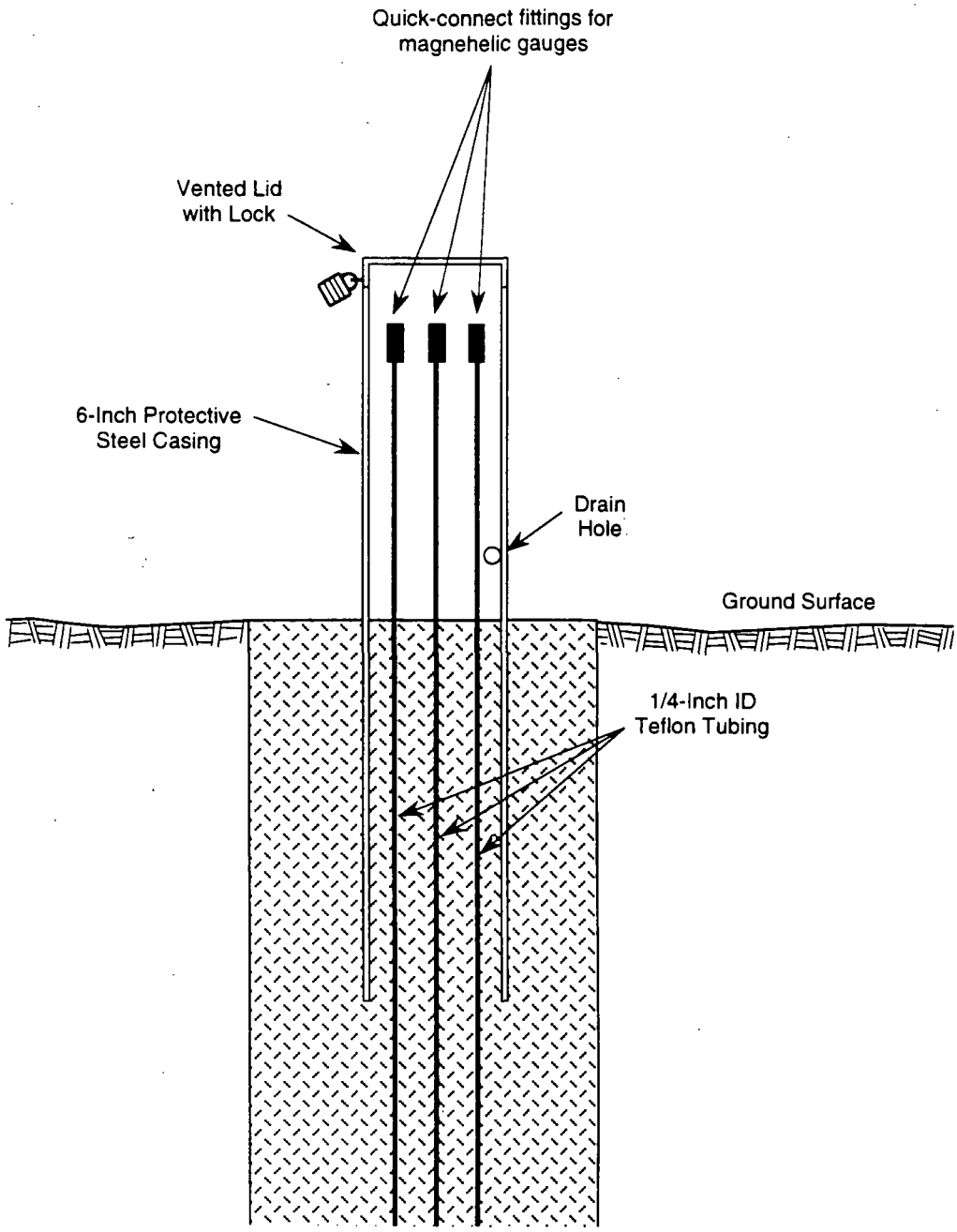


MONITORING WELL AND PIEZOMETER
(No Scale)

This document has been developed for a specific application and may not be used without the written approval of Montgomery Watson.

Appendix G
Attachment 4

Developed By	Drawn By	**Monitoring Well and Piezometer Surface Completion Detail**	Drawing Number 4077.0191
Approved By	Date *8/12/96*		
Reference		TREATABILITY STUDY AMERICAN CHEMICAL SERVICE, INC. NPL SITE GRIFFITH, INDIANA	MONTGOMERY WATSON
Revisions			



QUALITY CONTROL | Graphic Standards: Lead Professional | Technical Review: Project Manager | Management Review: Other

This document has been developed for a specific application and may not be used without the written approval of Montgomery Watson.

Apendix G
Attachment 5

Developed By	Drawn By	**SOIL VAPOR MONITORING PROBE COMPLETION DETAILS**	Drawing Number 4077.0191
Approved By	Date 8/12/96		
Reference		TREATABILITY STUDY AMERICAN CHEMICAL SERVICE, INC. NPL SITE GRIFFITH, INDIANA	MONTGOMERY WATSON
Revisions			

GROUNDWATER MONITORING WELL AND PIEZOMETER INSTALLATION CHECKLIST

Scope and Application: This method is applicable to the installation of groundwater monitoring wells and piezometers.

References: ASTM Standard D5092-90;
In Wisconsin: Chapter NR141,
Wisconsin Administrative Code;

I. PRE-FIELD CHECKLIST

A. Health and Safety Plan

B. Well construction plan

1. Well materials
2. Well depths
3. Conceptual hydrogeologic model
4. Objective for installing well. Why in that location? Why that depth?

C. Inform driller of the well construction plan

D. Electric water level indicator (where applicable)

E. Tape measure graduated in tenths and hundredths of a foot

F. Montgomery Watson standard locks

G. Well construction forms

H. Traffic cones

II. Checklist of Items to be Inspected

A. Before well installation

1. Measure static water level or estimate water level from wet soils (where applicable).

2. Measure borehole depth.

3. Is the borehole stabilized? A soil borehole should be cased with temporary drill casing or augers. Do not set a well through an open hole after pulling out the augers. Open boreholes in stable bedrock, within one aquifer, are permitted.

4. New well materials: no used well screen, riser, or caps.

5. Steam clean well materials inside and outside; wrap in plastic or store in a clean area; handle well materials with clean gloves.

6. Inspect screen and riser pipe inside and out for cleanliness, defects, gouges, cracks: reject any failed pieces.

7. Accurately measure length of screen piece including blank sections and well point.

8. Measure total length of slotted interval.

9. Prepare a sketch which accurately represents the screen piece showing lengths of blank interval, lengths of slotted interval, well point, etc. Measurements must be in tenths of feet.

10. Accurately measure length of each riser piece.

11. Count the number of riser pieces.

12. Determine the total length of the assembled well string.

13. Inspect filter pack material: proper gradation, proper material, contaminant free, sufficient quantity.

14. Inspect fine sand material: proper gradation, proper material, contaminant free, sufficient quantity.

15. Inspect bentonite: 100% pure Wyoming bentonite with no additives, proper size, sufficient quantity.

16. Optional: inspect portland cement, proper type and sufficient quantity. Caution: Use of Portland cement may result in grout contamination of the well (high pH) if improper seals and/or improper recipes are used. Consult with project hydrogeologist.

17. Inspect tremie pipe: proper size and sufficient quantity; steam cleaned.

18. Stick-up well protective pipe: metal casing 2in. dia greater than the well casing; minimum 5ft length with locking cap.

19. Flush mounted protective cover pipe: water tight metal casing 4in. dia greater than the well casing; minimum 12in. length; exterior flange or lugs; water tight, bolt down lid with the words `MONITORING WELL' on its outer surface.

20. Optional: (if necessary) place bentonite seal below filter pack using a tremie pipe; use this option when the borehole is greater than 5ft deeper than depth of the well. Use a 2in. dia well riser pipe as a tremie pipe set into the borehole below the well screen. Slowly place bentonite chips through the tremie pipe while checking for bridging. Bring the chips up to within 2ft of the well bottom. The tremie pipe allows placement of the seal without smearing bentonite at the screen interval and prevents losing the borehole should bridging occur.

B. During well installation

1. Determine depth of well placement as total length of assembled well string minus height of well string top above ground surface.

2. Riser pieces should have water tight joints: either neoprene gaskets or teflon tape. Do not use glue or solvent cement.

3. Accurately determine total well depth.

   a. Measure length of well riser pipe piece cut off from the total length of well string.

   b. Total well string length minus length of cut off piece equals total well depth (TD) measured from top of casing (TOC).

   c. The well top should stick up a minimum of 24in. aboveground surface.

4. Install a temporary well cap to prevent any materials from falling into the well.

5. Filter pack construction.

   a. Introduce a well graded sand in a controlled manner: slowly add filter sand while retracting augers or casing.

   b. Driller continuously uses a tape measure to check for bridging.

   c. Filter pack will extend from 6in. below the well bottom to 2ft above top of well screen.

   d. Record volume of sand placed along with manufacturer, brand name, and gradation; 50lbs of sand is approximately 0.5cf.

   e. Record depth to top of sand pack.

6. Collapsed formation (option): May be used when an artificial sand pack cannot be installed and when the collapsed formation is coarser than fine sand. The collapsed formation should be analyzed for grain size and specific gravity.

7. Filter pack seal construction.

   a. Introduce a well graded fine sand in a controlled manner.

   b. Driller continuously uses a tape measure to check for bridging.

   c. Place 2ft of fine sand; record the volume of fine sand placed along with manufacturer, brand name, and gradation; 50lbs of sand is approximately 0.5cf.

   d. Record the depth to the top of the fine sand pack.

   e. Check that the well is not being pulled up nor is it sinking as installation progresses.

   f. Wells having grouts or slurry as the annular space sealant will have a minimum 5 ft of bentonite seal placed above the fine sand.

      Bentonite Seal:

      (1) Use bentonite chips or pellets no larger than 3/8in. dia when placing the seal through water.

      (2) Granular bentonite may be used when the depth of placement is less than 25ft and when no standing water is in the borehole.

      (3) Seals above the water table will be placed and hydrated in 2ft lifts; use a tremie pipe to place either the bentonite or the water to prevent a thick cake of bentonite from forming in the augers.

      (4) Place 6in. of fine sand on top of the bentonite seal.

      (5) Record the type, size, and volume of sealant placed.

8. Annular space seal: all permanent monitoring wells will have an annular space seal which extends from the top of the filter pack seal to the bottom of the ground surface seal and will have a minimum 2ft length.

   a. For water table wells with the water table at 7ft or less below ground surface use granular bentonite only; place the bentonite in 2 ft lifts, hydrating each lift.

   b. Use thick bentonite slurry or bentonite-cement grout for placing annular space seal greater than 50ft deep. Caution: Use of Portland Cement may result in grout contamination of the well (high pH) if improper seals or improper recipes are used. Consult with the project hydrogeologist.

      (1) Bentonite slurry recipe: 2 lb of granular bentonite per gal of water, or as thick as the driller can pump it; Rule of thumb: Thick slurry tends to shear in the mud tub rather than flow.

      (2) Bentonite-cement grout recipe: It is important to closely follow this recipe; deviation from this recipe may result in grout contamination of the well. Mix 6 1/2 gal of water per 94 lb bag of Portland Type I cement then add 3 to 5 lb of bentonite powder. This will yield about 1 1/2 times the volume of water used. Carefully measure the amount of water: Too much water causes persistent pH problems in the well (grout contamination).

      (3) Tremie pump sealant from the bottom up using a side discharge tremie pipe. Pump the sealant until it flows full strength, unoiluted, up and out thru the top of the hole.

      (4) Allow a 12-hour period between installing slurry or grout and installing the protective casing to allow for settlement and curing. If a 12-hr waiting period is impractical, the slurry or grout should be bailed out down to the water table. The annular space should then be filled using bentonite chips, pellets, or granules as described below.

      (5) The top of the seal should not be higher than 5ft below ground surface - r remove excess grout by bailing it out before it sets up.

      (6) Avoid using bentonite slurry as annular space sealant in the unsaturated zone. Bentonite slurry will flow into the unsaturated zone leaving a void space in the unsaturated annular space. Pump slurry to the top of the borehole, let it settle for 12hours, then use "c" or "d" below.

   c. Use bentonite chips no larger than 3/8in. dia or bentonite pellets when there is less than 30ft of standing water in the borehole and the depth to the bottom of the annular space seal is less than 50ft (except when the depth to water table is less than 7ft use granular bentonite). Place pellets or chips slowly in a controlled manner. Check for bridging. Hydrate in the unsaturated zone.

   d. Use granular bentonite.

      (1) When there is no standing water in the borehole and the placement depth is less than 25ft.

      (2) The depth to the water table is less than 7ft below ground surface.

   e. Record type and volume of annular space seal.

9. Construct ground surface seal. Check for annular space seal settlement. If grout or slurry is used as the annual space seal, wait 24hr after seal installation before installing the surface seal.

a. Stick up well protective pipe.

(1) Measure the length of well protective pipe.

(2) Subtract well stick up height to get embedment depth of well protective pipe.

(3) If the well protective pipe embedment depth intersects the filter pack or filter pack seal, then shorten the length of the well protective pipe. The minimum embedment depth should not be less than the stick up height.

(4) The ground surface seal will start at least 5ft below ground surface.

(5) Place bentonite chips, pellets or granules up to 1ft below the well protective pipe embedment depth, then place 1ft of filter sand.

(6) Set the well protective pipe onto the firm bed of filter sand.

(7) Add granular bentonite around the outside of the protective pipe only and hydrate it in 2ft lifts to the surface.

(8) Concrete ground surface seals in regions where the ground freezes are not recommended. Frost heave will jack the concrete seal and the well protective pipe out of the ground. If it is necessary to construct a concrete ground surface seal in regions affected by freezing ground, the concrete should be at least 4-ft dia, 8-in. thick, with reinforced mesh poured onto 4-in. of compacted road gravel. The concrete should not be poured onto fine grained soils nor should it be in contact with bentonite. The concrete should be sloped radially away from the well casing. Concrete should not be in contact with the well casing. This may seem excessive, but anything less is very likely to be ruined by frost action.

(9) Do not place bentonite between the protective pipe and the well casing.

(10) If the monitoring well depth is such that both a minimum 2ft annular space seal and a minimum 5ft ground surface seal cannot both be placed, the ground surface seal may be shortened.

(11) Record the depth to the bottom of the ground surface seal, also record t the length and dia of the well protective pipe.

(12) The well protective pipe should stick up a minimum of 24in. above the ground surface and should always extend above the top of the well.

(13) The top of the well pipe must be within 4in. of the top of the well protective pipe.

(14) The well protective pipe should not extend into the annular space seal nor into the filter pack.

(15) The well protective pipe should be filled with filter sand to within 12in. of the top of the well.

(16) A weep hole may be drilled into the well protective pipe; a small vent hole should be cut or drilled into the well cap.

b. Flush mount wells.

(1) Install only in areas of high vehicular traffic.

(2) Do not install in areas subject to ponding or flooding and should not be installed directly down topographic slope from surficial contamination.

(3) Install through an impervious surface such as asphalt or concrete; if an impervious surface does not exist, one should be created such as a 4-ft dia reinforced concrete pad, 8in. thick poured onto 4in. of compacted road gravel (Really!).

(4) Granular bentonite should fill the borehole annular space from the top of the annular space seal to 3ft below ground surface, then a bed of filter sand should be placed.

(5) The flush mount well protective casing should rest on a firm bed of filter sand.

(6) Concrete should be placed around the outside of the flush mount well protective casing and it should be positioned slightly (1/8in.) above grade with the concrete sloping radially outward.

(7) There should be no more than 8in. between the top of the well casing and the top of the flush mount well protective casing.

(8) Flush mount wells should be installed with water tight locking well caps.

C. After well installation

1. Check for settlement of the ground surface seal; top off as necessary.

2. Label the protective casing with the well number.

3. Stick up wells: label the well cap inside and out with the well number.

4. Lubricate the well lock.

a. Do not use WD-40 nor penetrating oils.

b. Remove the lock away from the well and lubricate it with liquid graphite.

c. Wipe off excess lubricant, allow the lock to `dry', then return it to the well.

5. Flush mount wells.

a. Use a tape measure to accurately locate the well with reference to at least two permanent land marks (e.g., 20ft west and 11ft north of fire hydrant; 22ft due east of power pole).

6. Stick up wells in high traffic areas: consider placing bumper posts around the well.

a. Wood or steel, set in concrete or bentonite.

b. At least 8ft long with 4ft stick up.

c. Posts may be painted or flagged.

d. Do not paint the well protective casing.

7. Stick up wells in agricultural fields:

a. Securely attach a 10ft x 3/4in. PVC pipe (or equivalent) to the well protective casing using large hose clamps.

b. Locate the well by measuring distance from fence lines or landmarks.

8. Clean up the area: pick up trash, do not burn; pick up cuttings; use a broom, rake, or hose down the area.

III. FIELD DOCUMENTATION

A. Stick up Monitoring Well Construction Summary (See Attachment)

1. Use for stick up wells.

2. Readily adapts to Wisconsin Form 4400-113A.

3. Utilized for field and for inclusion in report.

4. Supplemented by Monitoring Well Construction Field QC Summary (See Attachment).

B. Flush Mount Monitoring Well Construction Summary (SeeAttachment)

1. Use for flush mount wells.

2. Readily adapts to Wisconsin Form 4400-113A.

3. Utilized for field and for inclusion in report.

4. Supplemented by Monitoring Well Construction Field QC Summary (See Attachment).

C. Waste Management

1. Monitor well construction summary (See Attachment).

2. Used for both field and final client issue.

3. Use in conjunction with one of Montgomery Watson's forms.

4. Supplemented by Well Installation QC Form.

IV. TYPICAL WELL DESIGN

A. Water table wells (See Attachment)

1. 10ft screen length (no more than 15ft).

GROUNDWATER MONITORING WELL AND PIEZOMETER INSTALLATION CHECKLIST

2. Measure static water level or estimate from wet soil sample.

3. Set screen to intersect water level.

   a. 7ft under, 3ft above at `dry' times; 8ft under 2ft above during `wet' times.

   b. All under water when the water table is less than 5 ft below ground.

   c. May need to have much less than 7ft under water for perched water table conditions to avoid poking a hole in the underlying confining layer. A shorter well screen may need to be installed in thin, shallow perched zones.

4. Construct filter pack and filter pack seal.

5. Construct annular space seal.

   a. Greater than 50ft: consult with project hydrogeologist.

   b. 50ft to 25ft: use bentonite pellets or chips in 2ft lifts; hydrate every lift; use a tremie pipe for water or chips to prevent a thick bentonite cake from forming in the augers or casing.

   c. 0 to 25ft: use granular bentonite or pellets or chips in 2ft lifts; hydrate every lift; use a tremie pipe for water or bentonite to prevent a thick bentonite cake from forming in the augers or casing.

6. Ground surface seal and well protective casing.

   a. Stick up wells with water table greater than 7ft deep.

      (1) With 8ft long well protective casing, start the ground surface seal at 6ft (less deep for shorter well protective casings); set the protective casing on a bed of filter sand, add a little more sand if necessary; place granular bentonite around the well protective casing and hydrate it in 2ft lifts; do not place bentonite inside the well protective casing; do not use concrete in areas subject to freezing temperatures.

   b. Stick up wells with water table less than 7ft deep:

      (1) Use shorter well protective casing (5ft minimum).

      (2) Start ground surface seal at 6in. below the bottom of the well protective casing; place 6in. of filter sand then set the well protective casing onto the sand; add granular bentonite or bentonite chips around the outside of the well protective casing and hydrate it in 2ft lifts; do not place bentonite inside the well protective casing; do not use concrete in areas subject to freezing temperatures.

   c. Flush mounted casing.

      (1) Start the ground surface seal at 5ft depth.

      (2) Place granular bentonite or bentonite chips in 2ft lifts and hydrate in place up to 2ft; from 2ft to 1ft add filter sand; place an old tyvek or trash bag over the filter sand, then use a chisel or other tool to enlarge the borehole to 4in. dia greater than the outside dia of the flush mount well box; make the walls of the hole vertical, not tapered; remove excess materials and the tyvek/trash bag;

place the flush mount well box onto the sand and center it; add or remove sand as necessary so the top of the flush mount well box is slightly above grade (< 1/8in.); mix concrete in a bucket or in a wheel barrel, do not mix it on the pavement, add just enough water to make it plastic but not runny; place concrete around the outside of the flush mount well box in rodded 6in. lifts; form up and puddle the concrete surface with a trowel so it slopes radially outward from the flush mount well box cover; lock the well and place bolt down cover; protect from freezing with temporary cardboard cover; protect from traffic using traffic cones.

B. Piezometers

1. Maximum 5ft screen length.

2. Depth depends on objectives specified in the well construction plan, and geology encountered.

3. May need to backfill the borehole to an appropriate depth using a bentonite seal below well screen. Use a tremie pipe.

4. The well screen and sand pack should be within one aquifer, not extended across a confining layer to connect two aquifers. The bentonite seal should be tied into a low permeability unit (if possible).

5. Construct filter pack and filter pack seal.

6. Construct annular space seal; calculate the volume of sealant needed to fill the hole from the top of the filter pack seal to the ground surface.

   a. Greater than 50ft deep with predominantly non-cohesive soils or granular bedrock.

      (1) Mix thick granular bentonite slurry and tremie-pump it to the top using a side discharge tremie pipe.

      (2) The drill casing or augers should be fully charged with thick slurry before starting to remove augers or casing.

      (3) Top off grout level from the surface as each section of auger or casing is removed.

      (4) Use a mix recipe such as: 2lbs of granular bentonite per gallon of water, or as thick as the driller can pump it; rule of thumb: Thick enough when it tends to shear in the mud tub rather than flow.

   b. Greater than 50ft deep with predominantly cohesive soils or tight bedrock.

      (1) Mix a batch of thick granular bentonite slurry as described in (4) above sufficient to fill the lower 25ft of the annular space.

      (2) Tremie pump the slurry from the bottom up using a side-discharge tremie pipe.

      (3) Mix bentonite-cement grout by closely following this recipe: 6_gallons water plus 94lbs portland Type 1 cement plus 3lbs bentonite powder to yield approximately 1_ times the gallons of water used. Carefully measure the water volume used; too much water causes pH problems in the well.

CAUTION: Use of portland cement may result in grout contamination of the well (high pH) if improper seals and/or improper recipes are used. Consult with the project hydrogeologist.

(4) Pull back the tremie pipe to 15ft above its former level and tremie pump until thick bentonite-cement grout flows out the top.

(5) The drill casing or augers should be fully charged with bentonite slurry and bentonite-cement grout before starting to remove augers or casing.

(6) Top off grout settlement from the surface as each section of augers or casing is removed.

(7) If the grout level hasn't settled after removing all casing and augers; then bail out grout until it is below 6ft.

c. Annular space seals less than 50ft deep.

(1) Use bentonite slurry or bentonite-cement grout in the saturated zone as described above, or

(2) Use bentonite pellets chips or granular bentonite as described in PartIV.A No.5. above

STANDARD OPERATING PROCEDURES

SOIL LOGGING

STANDARD OPERATING PROCEDURES

SOIL LOGGING

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1.0 INTRODUCTION

This SOP is applicable to logging soils at all sites requiring soil investigation by Montgomery Watson. The SOP is based on the Unified Soils Classification System (USCS) and the ASTM Standard D 2488 - 90 Standard Practice for Description and Identification of Soils (Visual-Manual Procedure) (ASTM, 1990). Variance from the logging procedures described herein shall be warranted only if specifically required by a particular client or regulatory agency. A solid working knowledge of this SOP is critical for Montgomery Watson field personnel to standardize logging procedures and to enable subsequent correlations between borings at a site, allowing for accurate and thorough site characterization.

The information described in this SOP is summarized in two soil logging field guides (attached). Laminated copies of these guides are available for all field personnel: use of the field guides is strongly recommended. Other field guidance references may also be used according to personal preference, however, such references must be based on the USCS. Note that many references (for example, AGI Data Sheet grain size scales) base soil classifications on the Wentworth Scale. Such scales may vary significantly from the USCS and will lead to inaccurate or inconsistent soil descriptions.

2.0 DEFINITIONS

Use of the USCS requires familiarity with the grain size ranges that define a particular type of soil, as well as several other physical characteristics. The grain size definitions and physical characteristics upon which soil descriptions are based are presented below. This information is also presented in tabular format on the field guides.

2.1 GRAIN SIZES

USCS grain sizes are based on U.S. standard sieve sizes, which are named as follows:

- standard sieves with larger openings are named according to the size of the openings in the sieve mesh. For example, a "3-in." sieve contains openings which are 3 inches square.
- standard sieves with smaller openings are given numbered designations that indicate the number of openings per inch. For example, a "No. 4" sieve contains 4 openings per inch.

The following grain size definitions are paraphrased from the ASTM Standard D 2488 - 90. Field personnel should familiarize themselves with the grain size definitions and refer to the appropriate field guide for a visual reference.

Boulders - Particles of rock that will not pass a 12-in. (300-mm) square opening.

Cobbles - Particles of rock that will pass a 12-in. (300-mm) square opening and be retained on a 3-in. (75-mm) sieve.

Gravel - Particles of rock that will pass a 3-in. (75-mm) sieve and be retained on a No. 4 (4.75-mm) sieve with the following subdivisions:

Coarse Gravel	Passes a 3-in. (75-mm) sieve and is retained on a 3/4-in. (19-mm) sieve
Fine Gravel	Passes a 3/4-in. (19-mm) sieve and is retained on a No. 4 (4.75-mm) sieve

Sand - Particles of rock that will pass a No. 4 (0.19 in. or 4.75-mm) sieve and be retained on a No. 200 (0.003 in. or 75-µm) sieve with the following subdivisions:

Coarse Sand	Passes a No. 4 (0.19 in. or 4.75-mm) sieve and is retained on a No. 10 (0.08 in. or 2-mm) sieve
Medium Sand	Passes a No. 10 (0.08 in. or 2-mm) sieve and is retained on a No. 40 (0.017 in. or 425-µm) sieve
Fine Sand	Passes a No. 40 (0.017 in. or 425-µm) sieve and is retained on a No. 200 (0.003 in. or 75-µm) sieve

Silt - Soil passing a No. 200 (0.003 in. or 75-µm) sieve that is nonplastic or very slightly plastic and that exhibits little or no strength when air dried. Individual silt particles are not visible to the naked eye.

Clay - Soil passing a No. 200 (0.003 in. or 75-µm) sieve that can be made to exhibit plasticity within a range of water contents and that exhibits considerable strength when air-dried. Individual clay particles are not visible to the naked eye.

2.2 PHYSICAL CHARACTERISTICS

The following physical characteristics are used in the USCS classification for fine-grained soils. A brief definition of each physical characteristic is presented below. Tables 1 through 4 present descriptions of field tests that may be performed to estimate these properties in a field sample. However, with the exception of plasticity, the tests are generally too time-consuming to perform regularly in the field. A determination of the

type of fine-grained soil present in the sample can generally be made on the basis of plasticity, as described in Section 4.1.2.

Dry Strength - The ease with which a dry lump of soil crushes between the fingers (Table 1).

Dilatancy Reaction - The speed with which water appears in a moist pat of soil when shaking in the hand, and disappears while squeezing (Table 2).

Toughness - The strength of a soil, moistened near its plastic limit, when rolled into a 1/8 in. diameter thread (Table 3).

Plasticity - The extent to which a soil may be rolled into a 1/8 inch. thread, and re-rolled when drier than the plastic limit (Table 4).

3.0 RESPONSIBILITIES

This section presents a brief definition of field roles, and the responsibilities generally associated with them. This list is not intended to be comprehensive; additional personnel may be involved in other aspects of the project. Project team member information is usually included in project-specific plans (i.e., work plan, field sampling plan, quality assurance plan, etc.), and field personnel should always consult the appropriate documents to determine project-specific roles and responsibilities. In addition, one person may serve in more than one role on any given project.

Project Manager - Defines objectives of field work. Selects site-specific monitoring well design and installation methods with input from the Project Hydrogeologist and Field Team Leader. Oversees and prepares subcontracts.

Project Hydrogeologist - Selects site-specific drilling/sampling options. Helps prepare technical provisions of drilling subcontracts. Reviews boring logs.

Field Team Leader - Implements selected drilling program. May also review boring logs.

Drilling Rig Geologist - Records the boring log. Supervises drilling subcontractor.

Quality Manager - Performs field and logging process audits.

4.0 SOIL LOGGING PROCEDURES

The following aspects of a project must be considered before sampling and soil logging commences. This information is generally summarized in a project-specific work plan or field sampling plan, which must be thoroughly reviewed by all field personnel prior to the initiation of work.

- Purpose of the soil logging (e.g., initial investigation, subsequent investigation, remediation);
- Known or anticipated hydrogeologic setting including lithology (consolidated/unconsolidated, depositional environment, presence of fill material), physical characteristics of the aquifer (porosity/permeability), type of aquifer (confined/unconfined), recharge/discharge conditions, aquifer thickness and ground water/surface water interrelationships;
- Drilling conditions;
- Previous soil boring or borehole geophysical logs;
- Soil sampling and geotechnical testing program;
- Characteristics of potential chemical release(s) (chemistry, density, viscosity, reactivity, and concentration);
- Health and Safety protection requirements; and
- Regulatory requirements.

The procedures used to determine the correct soil sample classification are described below. These procedures are presented in tabular and flow chart form on the field guides.

4.1 FIELD CLASSIFICATION OF SOILS

The following soil classification procedures are based on the ASTM Standard D 2488 - 90 for visual-manual identification of soils (ASTM, 1990). The flow chart attached to this SOP can be used to assign the appropriate soil group name and symbol. When naming soils, the proper USCS soil group name is given, followed by the group symbol. For clarity, it is recommended that the group symbol be placed in parentheses after the written soil group name.

Soil identification using the visual-manual procedures is based on naming the portion of the soil sample that will pass a 3-in. (75-mm) sieve. Therefore, before classifying a soil,

any particles larger than 3 inches (cobbles and boulders) should be removed, if possible. Estimate and note the percentage of cobbles and boulders.

Using the remaining soil, the next step of the procedure is to estimate the percentages, by dry weight, of the gravel, sand, and fine fractions (particles passing a No. 200 sieve). The percentages shall be estimated to the closest 5%. In general, the soil is *fine-grained* (e.g., a silt or a clay) if it contains 50% or more fines, and *coarse-grained* (e.g., a sand or a gravel) if it contains less than 50% fines. If one of the components is present but estimated to be less than 5%, its presence is indicated by the term *trace*. For example, 'trace of fines' would be added as additional information following the formal USCS soil description.

4.1.1 Procedure for Identifying Coarse-Grained Soils (contain less than 50% fines)

If it has been determined that the soil contains less than 50% fines, the soil is a *gravel* if the percentage of gravel is estimated to be more than the percentage of sand. The soil is a *sand* if the percentage of gravel is estimated to be equal to or less than the percentage of sand.

If the soil is predominantly sand or gravel but contains an estimated 15% or more of the other coarse-grained constituent, the words "with gravel" or "with sand" shall be added to the group name. For example: "gravel with sand (GP)." If the sample contains any cobbles or boulders, the words "with cobbles" or "with cobbles and boulders" shall be added to group name. For example: "silty gravel with cobbles (GM)."

5% or less fines. The soil is a 'clean gravel' or 'clean sand' if the percentage of fines is estimated to be 5% or less. 'Clean' is not a formal USCS name, but rather a general descriptor for implying little to no fines. Clean sands and gravels are given the USCS designation as either *well-graded* or *poorly-graded*, as described below.

Identify the soil as a *well-graded gravel* (GW), or as a *well-graded sand* (SW), if it has a wide range of particle sizes and substantial amounts of the intermediate particle sizes. Identify the soil as a *poorly-graded gravel* (GP) or as a *poorly-graded sand* (SP) if it consists predominantly of one grain size (uniformly graded), or has a wide range of sizes with some intermediate sizes obviously missing (gap- or skip-graded).

NOTE: When using the USCS, keep in mind the difference between grading and sorting. The term grading is used to indicate the range of particles contained in the sample. For example, a poorly-graded sand containing predominantly one grain size would be considered well-sorted, and vice-versa. One notable exception to this general rule is a skip-graded (bimodally distributed) sample: a sand containing two distinct grain sizes would be considered both poorly-sorted and poorly-graded. The USCS uses only the *GRADING* descriptor in soil naming, not the sorting descriptor.

≥ 15% fines. The soil is a *silty* or *clayey gravel* or a *silty* or *clayey sand* if the percentage of fines is estimated to be 15% or more. For example, identify the soil as *clayey gravel* (GC) or a *clayey sand* (SC) if the fines are clayey. Identify the soil as a *silty gravel* (GM) or a *silty sand* (SM) if the fines are silty. The coarse grained descriptor "poorly-graded" or "well-graded" is not included in the soil name, but rather, should be included as additional information following the formal USCS soil description.

≥5% but <15% fines. If the soil is estimated to contain greater than 5% and less than 15% fines, give the soil a dual identification using two group symbols. The first group symbol shall correspond to a clean gravel or sand (GW, GP, SW, SP) and the second symbol shall correspond to a clayey/silty gravel or sand (GC, GM, SC, SM). The group name shall correspond to the first group symbol, and include the words "poorly-graded" or "well-graded", plus the words "with clay" or "with silt" to indicate the character of the fines. For example, "poorly-graded gravel with silt (GP-GM)".

4.1.2 Procedure for Identifying Fine-Grained Soils (contain 50% or more fines)

The USCS classifies inorganic fine-grained soils according to their degree of plasticity (no or low plasticity - indicated with an "L", or high plasticity - indicated with an "H") and other physical characteristics (defined in Section 2.2/Tables 1 through 4). As indicated in Section 2.2, the field tests used to determine dry strength, dilatancy, and toughness are generally too time consuming to be performed on a routine basis. Field personnel should be familiar with the definitions of the physical characteristics and the concepts of the field tests; however, field classifications will generally be based primarily on plasticity. NOTE: if precise engineering properties are necessary for the project (i.e., construction, modeling, etc.) geotechnical samples should be collected for laboratory testing. The results of the laboratory tests should be compared to the field logging results.

Lean clay (CL) - soil has medium to high dry strength, no or slow dilatancy, and medium toughness and plasticity.

Fat clay (CH) soil has high to very high dry strength, no dilatancy, and high toughness and plasticity.

Silt (ML) - the soil has no to low dry strength, slow to rapid dilatancy, and low toughness and plasticity, or is nonplastic.

Elastic silt (MH) - the soil has low to medium dry strength, no to slow dilatancy, and low to medium toughness and plasticity; will air dry more quickly than lean clay and have a smooth, silky feel when dry.

Organic soil (OL or OH) - the soil contains enough organic particles to influence the soil properties. Organic soils usually have a dark brown to black color and may have an organic odor. Often, organic soils will change color, for example, from black to brown, when exposed to the air. Organic soils normally will not have a high toughness or plasticity.

Other Modifiers for use with Fine-Grained Soils:

15% to 25% coarse-grained material. If the soil is estimated to have 15% to 25% sand or gravel, or both, the words "with sand" or "with gravel" (whichever is predominant) shall be added to the group name. For example: "lean clay with sand (CL)" or "silt with gravel (ML)". If the percentage of sand is equal to the percentage of gravel, use "with sand."

≥30% coarse-grained material. If the soil is estimated to have 30% or more sand or gravel, or both, the words "sandy" or "gravelly" shall be added to the group name. Add the word "sandy" if there appears to be the same or more sand than gravel. Add the word "gravelly" if there appears to be more gravel than sand. For example: "sandy silt (ML)", or "gravelly fat clay (CH)".

4.1.3 Procedure for Identifying Borderline Soils

To indicate that the soil may fall into one of two possible basic groups, a borderline symbol may be used with the two symbols separated by a slash. For example, a soil containing an estimated 50% silt and 50% fine grained sand may be assigned a borderline symbol "SM/ML". Borderline symbols should not be used indiscriminately. Every

effort shall be made to first place the soil into a single group and then to estimate percentages following the USCS soil description.

4.2 DESCRIPTIVE INFORMATION FOR SOILS

After the soil name and symbol are assigned, the soil color, consistency/density, and moisture content shall be described in that order. Other information is presented later in the description, as applicable.

4.2.1 Color

Describe the color. Color is an important property in identifying organic soils, and may also be useful in identifying materials of similar geologic or depositional origin in a given location. The Munsell Soil Color Charts should be used, if possible.

When using the Munsell Soil Color Charts, first attempt to assign the soil a general color, such as brown, gray, red, etc. Then go to the correct area in the charts and assign the applicable color name and Munsell symbol. The ability to detect minor color differences varies among people, and the chance of finding a perfect color match in the charts is rare. Keeping this in mind should help field personnel avoid spending unnecessary time and confusion going through the chart pages. In addition, attempting to describe detail beyond the reasonable accuracy of field observations could lead to making poorer soil descriptions than by expressing the dominant colors simply (Munsell Soil Color Chart, 1992).

If the color charts are not being used or are unavailable, again attempt to assign general colors to soils. Comparing a particular soil sample to samples from different locations in the borehole will help keep the eye "calibrated". For example, by holding two soils together, it may become evident that one is obviously greenish-brown, while another is reddish.

4.2.2 Consistency/Density

For intact fine-grained soil, describe consistency as very soft, soft, medium stiff, stiff, very stiff, or hard, based on the blows per foot using a 140 pound hammer dropped 30" (Table 5). If blow counts are not available, perform the field test described in Table 6 to determine consistency.

For coarse-grained soils, describe density based on blows per foot as very loose, loose, medium dense, dense, and very dense (Table 5). If blow counts are not available, attempt to estimate the soil density by observation, since a practical field test is not available. Be sure to clearly indicate on the field boring log if blow counts could not be obtained.

4.2.3 Moisture

Describe the moisture condition of the soil as dry (absence of moisture, dusty, dry to the touch), moist (damp but no visible water), or wet (visible free water, saturated).

4.2.4 Grain Size

Describe the maximum particle size found in the sample in accordance with the following information:

Sand Size -	If the maximum particle size is a sand size, describe as fine, medium, or coarse. (See Section 2 for sand size definitions.)
Gravel Size -	If the maximum particle size is a gravel size, describe the diameter of the maximum particle size in inches.
Cobble or Boulder Size -	If the maximum particle size is a cobble or boulder size, describe the maximum dimension of the largest particle.

For gravel and sand components, describe the range of particle sizes within each component. For example, "about 20% fine to coarse gravel, about 40% fine to coarse sand".

4.2.5 Odor

Due to health and safety concerns, NEVER intentionally smell the soil. This could result in exposure to volatile contaminants that may be present in the soil. If, however, an odor is noticed, it should be described if organic or unusual. Soils containing a significant amount of organic material usually have a distinctive odor of decaying vegetation (sometimes a hydrogen sulfide ["rotten egg"] smell). If the odor is unusual (petroleum product, chemical, etc.), it shall be described. Organic vapor readings from a PID or similar instrument should be noted on the field boring log. The project-specific health

and safety plan should then be consulted to determine the appropriate level of protection necessary for the continuation of field work.

4.2.6 Cementation

Describe the cementation of intact coarse-grained soils as weak, moderate, or strong, in accordance with the following criteria:

Weak - Crumbles or breaks with handling or little finger pressure
Moderate - Crumbles or breaks with considerable finger pressure
Strong - Will not crumble or break with finger pressure

The presence of calcium carbonate may be confirmed on the basis of effervescence with dilute hydrochloric acid, HCl, if calcium carbonate or caliche is believed to be present in the soil. Proper health and safety precautions must be followed when mixing, handling, storing, or transporting HCl. For further information, see I/HW Health and Safety Procedure 630.24, "Procedure for Hydrochloric Acid Handling for Soil Logging."

4.2.7 Angularity

Describe the angularity of the sand (coarse sizes only), gravel, cobbles, and boulders, as angular, subangular, subrounded, or rounded in accordance with the following criteria:

Angular - Particles have sharp edges and relatively planar sides with unpolished surfaces

Subangular - Particles are similar to angular description but have rounded edges

Subrounded - Particles have nearly plane sides but have well-rounded corners and edges

Rounded - Particles have smoothly curved sides and no edges

A range of angularity may be stated, such as "subrounded to rounded."

4.2.8 Structure

Describe the structure of intact soils in accordance with the criteria in Table 7.

4.2.9 Lithology

Describe the lithology (rock or mineral type) of the sand, gravel, cobbles, and boulders, if possible. It may be difficult to determine the lithology of fine and medium-grained sand or particles that have undergone alteration.

4.2.10 Additional Comments

Additional comments may include the presence of roots or other vegetation, fossils or organic debris, staining, mottling, or oxidation; difficulty in drilling, and caving or sloughing of the borehole walls. Also, when drilling in an area known or suspected to contain imported fill material, every effort should be made to identify the contact between fill and native soils. If a soil is suspected to be fill, this should be clearly indicated on the log following the soil description. Stratigraphic units and their contacts should be noted wherever possible.

4.2.11 Bedrock Descriptions

If the soil boring penetrates bedrock, the boring log form should indicate the rock type, color, weathering, fracturing, competency, mineralogy, age (if known), and any other miscellaneous information available. Definitions of these terms are not included in this SOP, because only a small percentage of drilling activities conducted by Montgomery Watson penetrate bedrock. If bedrock drilling is planned, the field team leader, with the concurrence of the project manager, should make arrangements to provide the field team with appropriate definitions and indicate the types with information that should be collected.

4.3 ADDITIONAL BORING LOG INFORMATION

In addition to soil descriptions, there are several other items that should be included on all Montgomery Watson boring log forms. The boring log form included in Section 6 should be used unless a different form is required by the client. Information in the log heading should be complete and accurate. The following information should be included, at a minimum:

- Boring or monitoring well number
- Project name and job number

- Site name
- Name of individual who logged the boring
- Name of boring log form reviewer
- Drilling contractor
- Drill rig type and method of drilling (for example, "CME 75, hollow stem auger")
- Name of drilling company
- Name of driller and helper
- Borehole diameter and drill bit type
- Type of soil sampler (for example, Modified California, continuous core, etc.)
- Time and date that drilling started and finished
- Time and date that the well was completed or the soil boring backfilled, as appropriate
- Method of borehole abandonment
- Sketch map of boring or well location with estimated distances to major site features such as property lines or buildings, and north arrow

Soil sample information should include the depth interval that was sampled, the blow counts per six inches, the amount of soil recovered, and the portion submitted for analysis or testing, if any. The sample identification number may also be noted on the log.

The degree to which soil samples are collected during a field effort depends on the overall scope and purpose of the investigation, which should be clearly defined before the field effort commences. Additional soil samples may need to be collected if, for example, soils are very heterogeneous or unexpected conditions such as perched water zones or zones of contamination are encountered.

If groundwater is encountered during drilling, the depth to water and the time and date of the observation should be recorded. If the first water encountered is a perched zone, the depth, time, and date that any additional groundwater zones are encountered should also be recorded. Depth to water after drilling, the measuring point, and the date and time of the measurement(s) must be noted. Additional measurements of depth to groundwater, including depth and time, may be beneficial.

If a monitoring well is installed, the construction details such as casing material type, screen length and slot size should be noted on the boring log form. The annulus fill material (sand pack, bentonite, grout, etc.) shall also be recorded.

If the soil boring is abandoned, the backfill material used (e.g., grout, bentonite, etc.) and volume used, should be recorded on the boring log form.

5.0 OTHER APPLICABLE SOPS

Several other Montgomery Watson SOPs contain information related to soil boring and logging activities. The following is a list of these SOPs:

Drilling Methods
Monitoring Well Design and Installation
Soil Sampling
Trenching and Test Pitting

6.0 REFERENCES

ASTM, 1990, Standard D 2488 - 90 Standard Practice for Description and Identification of Soils (Visual-Manual Procedure)

Macbeth, 1992, Munsell Soil Color Charts

TABLE 1

CRITERIA FOR DESCRIBING DRY STRENGTH

Description	Criteria
None	The dry specimen crumbles into powder with mere pressure of handling
Low	The dry specimen crumbles into powder with some finger pressure
Medium	The dry specimen breaks into pieces or crumbles with considerable finger pressure
High	The dry specimen cannot be broken with finger pressure. Specimen will break into pieces between thumb and a hard surface
Very High	The dry specimen cannot be broken between the thumb and a hard surface

TABLE 2

CRITERIA FOR DESCRIBING DILATANCY

Description	Criteria
None	No visible change in the specimen
Slow	Water appears slowly on the surface of the specimen during shaking and does not disappear or disappears slowly upon squeezing
Rapid	Water appears quickly on the surface of the specimen during shaking and disappears quickly upon squeezing

TABLE 3

CRITERIA FOR DESCRIBING TOUGHNESS

Description	Criteria
Low	Only slight pressure is required to roll the thread near the plastic limit. The thread and the lump are weak and soft
Medium	Medium pressure is required to roll the thread to near the plastic limit. The thread and the lump have medium stiffness
High	Considerable pressure is required to roll the thread to near the plastic limit. The thread and the lump have very high stiffness.

TABLE 4

CRITERIA FOR DESCRIBING PLASTICITY

Description	Criteria
Nonplastic	A 1/8-in. (3-mm) thread cannot be rolled at any water content
Low	The thread can barely be rolled and the lump cannot be formed when drier than the plastic limit
Medium	The thread is easy to roll and not much time is required to reach the plastic limit. The thread cannot be rerolled after reaching the plastic limit. The lump crumbles when drier than the plastic limit.
High	It takes considerable time rolling and kneading to reach the plastic limit. The thread can be rerolled several times after reaching the plastic limit. The lump can be formed without crumbling when drier than the plastic limit.

TABLE 5

DENSITY/CONSISTENCY BASED UPON BLOW COUNTS

Density (Sand and Gravel) Blows/ft*				Consistency (Silt and Clay) Blows/ft*			
Term	1.4" ID	2.0" ID	2.5" ID	Term	1.4" ID	2.0" ID	2.5" ID
very loose	0-4	0-5	0-7	very soft	0-2	0-2	0-2
loose	4-10	5-12	7-18	soft	2-4	2-4	2-4
medium dense	10-29	12-37	18-51	medium stiff	4-8	4-9	4-9
dense	29-47	37-60	51-86	stiff	8-15	9-17	9-18
very dense	>47	>60	>86	very stiff	15-30	17-39	18-42
				hard	30-60	39-78	42-85
				very hard	>60	>78	>85

* 140 lb. hammer dropped 30 inches

TABLE 6

CRITERIA FOR DESCRIBING CONSISTENCY

Description	Criteria
Very soft	Thumb will penetrate soil more than 1 in. (25 mm)
Soft	Thumb will penetrate soil about 1 in. (25 mm)
Firm	Thumb will indent soil about 1/4 in. (6 mm)
Hard	Thumb will not indent soil but readily indented with thumbnail
Very Hard	Thumbnail will not indent soil

TABLE 7

CRITERIA FOR DESCRIBING STRUCTURE

Description	Criteria
Stratified	Alternating layers of varying material or color with layers at least 6 mm thick; note thickness
Laminated	Alternating layers of varying material or color with the layers less than 6 mm thick; note thickness
Fissured	Breaks along definite planes of fracture with little resistance to fracturing
Slickensided	Fracture planes appear polished or glossy, sometimes striated
Blocky	Cohesive soil that can be broken down into small angular lumps which resist further breakdown
Lensed	Inclusion of small pockets of different soils, such as small lenses of sand scattered through a mass of clay; note thickness
Homogeneous	Same color and appearance throughout