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REPORT

FINAL OPERABLE UNIT 3 PRE-DESIGN INVESTIGATION WORK PLAN

Nease Chemical Site, Columbiana and Mahoning Counties, Ohio

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August 31, 2010

Project No.: 933-6154-004
Via e-mail and FedEx

Mr. Dion Novak
USEPA Region V (SR-6J)
77 West Jackson Boulevard
Chicago, IL 60604

RE: FINAL PRE-DESIGN INVESTIGATION WORK PLAN
OPERABLE UNIT 3, NEASE CHEMICAL SITE, SALEM, OHIO

Dear Dion,

On behalf of RÜTGERS Organics Corporation (ROC), we are pleased to submit the Final Pre-Design Investigation (PDI) Work Plan for Operable Unit 3 of the Nease Chemical Site located in Columbiana and Mahoning Counties, Ohio. Copies have also been sent directly to the Ohio Environmental Protection Agency (Ohio EPA) in accordance with your request.

This PDI Work Plan is being submitted in accordance with the requirements of an Administrative Order on Consent (AOC) between ROC and USEPA with an effective date of June 30, 2009. This version of the Work Plan includes revisions to the previous Work Plan (submitted on June 16, 2010) based on the Agencies' comments, which were provided to ROC on August 17, 2010. A separate document is also enclosed that provides a written response to each comment, as requested.

As noted in previous communications, ROC intends to begin the field investigation on September 8, 2010. Field activities are anticipated to last 3 to 4 weeks, including fish tissue sampling, which will be conducted the week of September 13. Your final concurrence with the Work Plan prior to beginning the field effort will be appreciated.

If you should have any questions during your review of this Work Plan, please do not hesitate to contact Dr. Rainer Domalski at ROC (814/231-9200) or the undersigned (856/793-2005).

Very truly yours,

GOLDER ASSOCIATES INC.

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Enclosure

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1.0 INTRODUCTION

This Pre-Design Investigation Work Plan (PDI Work Plan) has been prepared by Golder Associates Inc. (Golder), on behalf of RÜTGERS Organics Corporation (ROC) for Operable Unit 3 (OU-3) at the Nease Chemical Site, Columbiana and Mahoning Counties, Ohio (Site). OU-3 is comprised of contaminated portions of Feeder Creek (located at the former Nease Chemical Facility) and Middle Fork Little Beaver Creek (MFLBC), including associated floodplain soils. The United States Environmental Protection Agency (USEPA) selected the remedy for OU-3 in a Record of Decision (ROD) dated September 24, 2008. Administratively, the PDI Work Plan is submitted in accordance with the requirements of an Administrative Order on Consent (AOC) between ROC and USEPA with an effective date of June 30, 2009. The AOC and accompanying Statement of Work (SOW) establishes the scope and schedule of activities leading to the design of the OU-3 remedy.

ROC previously completed a Remedial Investigation (RI), an Endangerment Assessment (EA), and a Feasibility Study for OU-3 (FS). The RI Report was submitted in January 1996 and was approved by USEPA and the Ohio Environmental Protection Agency (Ohio EPA) (referred to collectively as "the Agencies") on June 19, 1996. The final EA was submitted to the Agencies in April 2004 (with errata pages in September, 2004), and included the Human Health Risk Assessment and the Ecological Risk Assessment. The Agencies issued final approval of the EA on August 30, 2004. The FS was submitted on June 5, 2008 and approved by the Agencies on June 30, 2008. The RI/FS, including the EA, formed the basis for USEPA's remedy selection.

1.1 Site Location and Description

Figure 1 shows the location of the Site (including the former manufacturing facility and the MFLBC). The former Nease Chemical facility included a manufacturing area west of the Conrail Railroad tracks and wastewater ponds on both sides of the railroad tracks. Historically, Crane-Deming owned adjacent property between the railroad tracks and Allen Road. In 1998 ROC took ownership of this property, including the production building, with Crane-Deming continuing to lease and operate its pump manufacturing facility on the property. ROC subsequently sold a portion of the property north of the railroad tracks, including the former Crane Deming building, and MAC Trailer Company now manufactures trailers at this location.

Feeder Creek, which flows from the former manufacturing facility to the MFLBC, originates close to the railroad tracks and merges with several drainage ditches en route to its confluence with the MFLBC at Allen Road. Feeder Creek is the main route for surface water drainage from the former Nease facility to the MFLBC. As such, it likely represented the primary transport route for Site-related contaminants to enter the MFLBC system.

The MFLBC flows northeast from its source southwest of the former Nease facility, passing to the east of the Nease facility. The confluence of Feeder Creek and the MFLBC is at about MFLBC River Mile (RM)

37.6. The former Nease facility is on a topographic high and the ground generally slopes towards the creek. The MFLBC flows north out of the City of Salem through Mahoning County, turns to the east, and then flows south through Columbiana County until it joins with the West Fork Little Beaver Creek and North Fork Little Beaver Creek to form Little Beaver Creek. Little Beaver Creek flows south to the Ohio River at East Liverpool. The MFLBC extends approximately 40.6 river miles with an average gradient of 11.8 feet per mile (Ohio EPA, 2005), and drains a total area of approximately 496 square miles (RNC, 1996). The MFLBC is divided into two different use classifications according to the Ohio Revised Code (ORC) 3745-1-15. The MFLBC is classified as Warmwater Habitat from the headwater to the spillway at Lisbon (River Mile 12.5). From RM 12.5 to the mouth is classified as Exceptional Warmwater Habitat. All waters of the MFLBC are designated for agriculture, industry, and primary contact uses, but none are designated for "drink" use. As discussed in the ROD, the area where additional characterization and targeted remediation is required is the stream reach from RM 37.6 to RM 31.0.

The area surrounding the former Nease facility includes residential properties along State Route 14 and other industrial/commercial facilities east and northeast along Allen Road. The ROC property and areas to the east and northeast are zoned for industrial purposes. The properties bordering the MFLBC include residential, recreational, agricultural, and industrial/commercial uses. As shown in the aerial photograph on Figure 1, land use along the creek from RM 37.6 through RM 31.0 is primarily agricultural. There are two dairy farms within the target reach: one near RM 33.0 and another near RM 35.0. Colonial Villa, a residential area, is located between RM 35.0 and RM 36.0. There is also a commercial facility between RM 32.0 and RM 33.0 on State Route (SR) 45.

2.0 OVERALL OBJECTIVES

The selected remedy for OU-3 specifies response actions that will address chemically- contaminated sediment and floodplain soil in Feeder Creek and the MFLBC. The selected remedy consists of removal of Feeder Creek sediment and targeted removal of MFLBC sediment and floodplain soil with on-Site consolidation beneath a soil cover that is part of the remedy for Operable Unit 2 (OU-2). Targeted removals will be designed to achieve the sediment and floodplain soil surface weighted average concentration (SWAC) preliminary remedial goals (PRGs) specified in the ROD. The sediment SWAC PRG selected by USEPA is 0.5 mg/kg mirex, but, as described in the ROD, this PRG may be modified to be as high as 0.75 mg/kg during detailed design to prevent disturbance of areas of high quality habitat. The sediment PRG will be evaluated over an exposure area within the stream covering 1 river mile. The floodplain soil SWAC PRG selected by USEPA is 1.0 mg/kg, to be evaluated over an exposure unit of 1 acre. Similar to sediment, some areas of the floodplain are considered high-quality habitat, so the results of the PDI will be used to determine how to achieve the PRG while protecting habitat to the extent possible. The Remedial Design (RD) will be a phased process and will be conducted such that pertinent information will be taken into account as it becomes available.

During the development of this PDI Work Plan, potential dynamic field activities were considered (Triad approach), and to the extent appropriate, have been incorporated into this Work Plan. This PDI includes components that will require concurrence with the Agencies before proceeding, such as the selection of a laboratory for fish tissue analyses, and the selection of archived samples for analysis to meet PDI objectives. As such, a critical component of the timely and successful completion of the PDI will be the on-going discussions between the Agencies and ROC during the implementation of the PDI; however, based on a long history of cooperation and collaboration between ROC and the Agencies, ROC anticipates that such concurrence can be achieved without significant difficulty.

The following summarizes the overall objectives of the PDI as presented in the SOW:

- Sediment Conditions
  - Detailed mapping of sediment bodies from RM 37.6 through 31.0
  - Refinement of the extent and distribution of mirex contamination in MFLBC sediment between RM 37.6 through 31.0 to delineate targeted areas that require removal to meet the remediation goal
  - Identification of smaller sub-areas of elevated concentrations to evaluate whether a maximum, "do not exceed" mirex criterion shall be established in the RD
  - Identification of areas of high quality habitat
- Floodplain Soil Conditions
  - Refinement of the extent and distribution of mirex contamination in floodplain surface soils along the MFLBC from RM 37.6 through 31.0 to delineate targeted areas that require removal to meet the remediation goal

- Identification of smaller sub-areas of elevated concentrations to evaluate whether a maximum, "do not exceed" mirex criterion shall be established in the RD
- Physical characterization of the areas targeted for remediation
- Identification of areas of high quality habitat

- Fish Tissue Conditions
  - Sampling and analysis of both whole body and fillet fish samples for mirex and lipids to provide a baseline sampling event consistent with the anticipated long-term monitoring program
- Surface Features
  - Wetlands and Floodplain Assessments

3.0 DESIGN OBJECTIVES AND PDI SCOPE

3.1 Site Reconnaissance

3.1.1 Overview

As specified in the Statement of Work (SOW), Site reconnaissance is required as a preliminary step of the PDI, and a Work Plan Memorandum describing the planned reconnaissance activities was submitted to the Agencies on August 24, 2009. The Agencies provided comments via e-mail on August 31, 2009 and ROC provided responses to these comments on August 31, 2009. The Agencies approved the Work Plan Memorandum (as modified by the responses to comments) on September 11, 2009.

In order to facilitate dynamic decision-making and efficient data collection in the PDI, the Site Reconnaissance described in the Work Plan Memorandum included some portions of the PDI scope required by the SOW in addition to the basic requirements specified for the Site Reconnaissance.

The reconnaissance included the following tasks, as outlined in the PDI Reconnaissance Work Plan Memorandum (see Appendix A):

- Detailed mapping of the extent of fine-grained sediment bodies from RM 37.6 through 31.0[1] using a handheld global positioning system (GPS) device.
- Detailed description of fine-grained sediment through collection of cores and/or grab samples of sediment within the mapped sediment bodies for visual classification.
- Description of general sediment type in areas adjacent to fine-grained sediment bodies, including at least one description per 0.1 river mile (for segments that included fine-grained sediment).
- Laboratory grain size analyses of each significant fine-grained sediment "type" encountered.
- Photographic documentation of the stream channel and banks at each 0.1 river mile.
- Mapping of obstructions within the stream that might interfere with investigation or remediation activities (including photographic documentation).
- Identification of areas of high quality habitat.
- Discussions with riparian area property owners[2] to discuss the upcoming PDI work and associated access requirements.

3.1.2 Results

A Trimble GeoXH handheld GPS device running ESRI's ArcPad software version 8.0 with possible GPS horizontal accuracy of 1 ft or better was used to collect spatial data and other observations in the field. For each location measurement collected with the handheld GPS unit, the estimated accuracy is recorded

[1] As agreed with Ohio EPA's on-site representative, one segment of the stream from RM 36.4 to RM 36.7 was not mapped based on the objections of a nearby riparian area property owner. Access agreements for portions of RM 36.4 to 36.7 have since been signed by the property owners, so areas with signed agreements will be mapped and sampled as appropriate during the PDI activities outlined in this Work Plan.
[2] These discussions were coordinated and primarily conducted by Ohio EPA.

by the device. Accuracy can be influenced by time of day (number and locations of available satellites) and by obstructions such as bridges and the forest canopy, so in some cases, the theoretically possible accuracy was not achieved during the Reconnaissance work. The rated accuracy of the device is also influenced by the exact position of the satellites it uses. The positions of satellites may shift slightly from the predicted positions, so the GPS device accounts for these possible shifts when determining the estimated accuracy of the data. In order to reduce the uncertainty related to the exact positions of satellites, there are services that collect data on the satellite locations and by using these data, it is possible to revise the location information collected by the GPS in the field, and in many cases, the confidence in the GPS data (and therefore the accuracy) is significantly increased by using known satellite positions. This process is referred to as post-processing. Post-processing of the data was conducted using Trimble GPS Analyst's Differential Correction tool and the results indicate that approximately 91% of the location data collected were accurate to within 1 meter with about 56% being accurate to within 0.5 meter. The accuracy of the collected locations is important because the GPS location data will be used for identifying sampling locations during the full PDI and will also be used for detailed design (e.g., for identifying locations for staging areas, which will impact the design of temporary roads, etc. and may affect contractor cost estimates).

The detailed mapping of fine-grained sediment bodies showed that between RM 31.0 and approximately RM 36.3, the majority of sediment is located in small pockets close to the banks of the stream in areas that would be expected to be depositional given the stream morphology. These are areas where water velocities are slower (such as on the outsides of bends and in deep pools), allowing fine particles to settle out of suspension. In addition, fine-grained sediment is limited to a thickness of 1 or 2 inches in most places, and in some areas, fine-grained sediment is found only in the interstices between larger cobbles. From RM 36.3 to RM 37.7, fine-grained sediment is trapped by submerged aquatic vegetation (*elodea* or similar species) that covers up to 70% of the streambed in some areas. These plants cause the water velocity to slow, allowing fine-grained sediment to settle out and collect near the roots of the plants. These plants were typically found growing in coarser materials, such as sands and gravels, so the fine-grained sediment in these areas is restricted to a thickness of 1 to 2 inches at the base of the vegetation. The frequency of occurrence of this aquatic vegetation throughout this segment of the stream appeared to be higher than in previous investigations suggesting that the growth may be a relatively recent occurrence, potentially as a result of increased nutrient loading, or other environmental factors. Maps showing the locations where fine-grained sediment was identified are provided as Figures 2 through 6.

In addition to the sediment mapping activities, the locations of obstructions in the stream were also recorded and photographed to aid in the development of future sampling and remediation plans. A total of 51 obstructions (typically fallen trees blocking the stream) were encountered.

In general, at least four photographs were taken in each 0.1 RM segment to provide information about the stream habitat quality and the floodplain conditions. These photographs were also used to document

potential access points. The locations where photographs were taken were recorded using the handheld GPS.

3.2 MFLBC Sediment Conditions

Further characterization of sediment conditions is needed to support the detailed design of the sediment remedy. The sediment investigation objectives of the PDI include:

- Detailed mapping of sediment bodies from RM 37.6 through 31.0 (already completed reconnaissance task, see Section 3.1) to refine the understanding of sediment body locations and volumes, which will be used in detailed design, and to identify areas where sediment samples should be collected for calculation of the SWAC.
- Refinement of the extent and distribution of mirex contamination in MFLBC sediment from RM 37.6 through 31.0 to delineate targeted areas that require removal to meet the remediation goal, so as to provide refined volume estimates for design and construction costing purposes.
- Identification of smaller sub-areas of elevated concentrations to evaluate whether a maximum, "do not exceed" mirex criterion shall be established in the RD.

As specified in the ROD for OU-3, the sediment remedy will be designed to achieve a SWAC of 0.5 mg/kg over 1 mile stream reaches between RM 31.0 and RM 37.6 via targeted removal (see Figure 1). In accordance with the ROD, this SWAC remediation goal may be adjusted up to 0.75 mg/kg in circumstances where high quality habitat would be protected (i.e., not disturbed by remedial activities) by leaving certain areas of elevated concentrations in place[3]. The adjusted remediation goal of 0.75 mg/kg was selected in the ROD because it is within the range of protective PRGs determined in the FS process. The primary purpose of the PDI will be to collect sufficient data so that a reliable SWAC can be calculated for each 1 RM area and compared against the PRG to determine the target areas for sediment removal.

Sediment samples will be collected as horizontal composites similar to the previous sediment sampling event in 2005. This allows for a greater density of sampling throughout the target reach and is more representative of actual exposure. As specified in the ROD, the sediment remediation will target "surface" sediments, or the upper 6 inches of sediment, which is generally considered to be the biologically active zone; however, recognizing that removal of shallow sediment will expose deeper sediment, additional samples will also be collected from the 6 inch to 12 inch depth horizon, if fine-grained sediment is present at that depth, and archived for possible analysis pending the results of analyses on the shallow samples. Deeper sediment samples would be analyzed if surface sample mirex concentrations indicate that removal of sediment in this area will be necessary to achieve the SWAC remediation goal. As discussed above, fine-grained sediment throughout the target remediation area is typically 2 inches thick or less, so very few "deep" samples (i.e., 6 to 12 inches) are anticipated. All samples will be collected as a horizontal composite over the sampling area.

[3] Certain, localized areas of elevated mirex (i.e., greater than the remediation goal) will only be left in place to avoid disturbance of habitat if the overall SWAC within that river mile reach can achieve the modified remediation goal of 0.75 mg/kg without removing that sediment.

Because mirex adheres most strongly to fine-grained, organic-rich sediments, sediment sampling will be focused primarily on collecting samples from areas where silts and clays dominate. To further confirm that elevated mirex concentrations are restricted to areas of fine-grained sediment, additional samples will be collected from coarser-grained sediments (i.e., sand-dominated) in several locations (one per river mile) adjacent to pockets of fine-grained sediment that are sampled in the PDI. These coarse-grained sediment samples will be collected as surface samples (0-6 inches) and will be collected as five-point composites within the area adjacent to the fine-grained sediment deposit. The selection of coarse-grained sediment sampling locations will be a field decision based on the presence of significant quantities of fine-grained sediment in the area. Previous mirex concentrations in sediment will also be considered in selecting sampling locations. The SWAC calculated for each RM will likely be somewhat biased towards fine-grained sediment as a result of the sampling strategy, but this is consistent with the RI sampling data used to calculate the PRG.

There are currently no available field testing methods for mirex, so the approach described above is designed to expedite the investigation by collecting all the samples at once while providing an iterative analysis approach to meet the data requirements for detailed design.

Based on the results of fine-grained sediment mapping during the Reconnaissance (see Figures 2 through 6), it is anticipated that up to 71 primary sediment samples will be collected (65 from the 0 to 6 inch depth horizon plus an estimated six[4] from the 6 to 12 inch depth horizon). The final number of samples, however, will be determined in the field based on the thickness of fine-grained sediment encountered[5]. Each sample will be collected as a composite of at least 8 to 10 points across each 0.1 RM segment where possible (small areas of sediment may be an exception). This sampling approach draws on the concepts of multi-incremental sampling since the exposure unit for this Site is 1 RM (*i.e.*, 80 to 100 discrete locations will be sampled per exposure unit) but sample manipulation (*e.g.*, drying, sieving, grinding) will not be conducted since the goal is to collect a sample that is representative of potential exposures. In some cases, it is appropriate to collect separate samples from each side of the stream where this may affect the remedy implementation. For example, if the stream is too wide, or if there are significant obstructions, it will not be possible to remove sediment from both sides of the stream from just one staging area. By collecting separate composite sediment samples from opposite sides of the stream, this situation may be avoided if, based on mirex concentrations in the two samples, one side does not need to be removed to achieve the SWAC remediation goal. A summary of the anticipated

[4] Table 2 includes anticipated locations for four "deep" samples; however, an allowance for up to six samples has been made in the Work Plan.

[5] If fine-grained sediment is only present in the 0 to 6 inch horizon, then only one sample will be collected, whereas if fine-grained sediment is present deeper than 6 inches, a second, 6 to 12 inch sample will be collected. Gravel and/or cobbles underlying fine-grained sediment will not be sampled, regardless of depth.

number of samples for each 0.1 RM segment is shown in Table 2[6]. The final sample locations and number of samples collected may also be affected by access limitations.

Sediment samples will be analyzed for mirex, grain size distribution, total organic carbon (TOC), and total solids. Initially only the surficial fine-grained sediment samples will be analyzed at the laboratory while deeper samples and coarser-grained samples will be archived pending the results of the shallow analyses. Coarse-grained and "deep" samples will be analyzed if nearby or overlying fine-grained sediments have concentrations exceeding the PRG.

A description of the specific investigation procedures that will be implemented to assess the extent of mirex impacts to sediment is presented in Section 4.1.2. See Figure 7 for a map of the overall sampling plan for OU-3.

3.3 MFLBC Floodplain Soil Conditions

Further characterization of floodplain soil conditions is needed to support the detailed design of the floodplain soil remedy, which involves targeted removal and backfilling to meet a SWAC PRG. The primary objectives of the floodplain soil aspects of the PDI include:

- Refinement of the extent and distribution of mirex contamination in floodplain surface soils along the MFLBC from RM 37.6 through RM 31.0 to delineate targeted areas that require removal to meet the remediation goal.
- Identification of smaller sub-areas of elevated concentrations to evaluate whether a maximum, "do not exceed" mirex criterion shall be established in the RD.
- Physical characterization of the areas targeted for remediation.

3.3.1 Extent of Mirex Impacts to Floodplain Surface Soil

The primary objectives of the floodplain surface soil remedy are to mitigate potential ecological exposures to mirex at concentrations over 1.0 mg/kg and to be protective of human health exposures via beef and milk ingestion as described in the ROD. The remedial component identified in the ROD to mitigate these exposures is targeted removal and backfilling with clean soil. Therefore, in order to complete the design of targeted excavations within the floodplain, it is necessary to determine the extent of mirex impacts to floodplain surface soil above the PRG.

GIS data layers of the 100-yr floodplain were downloaded for Mahoning and Columbiana counties from the Ohio Department of Natural Resources website. These floodplain layers are digitized versions of Federal Emergency Management Agency (FEMA) Flood Insurance Rate Maps (FIRMs) from 1977 and 1973 (for Mahoning and Columbiana Counties, respectively). FEMA and Ohio Soil Conservation Department personnel have indicated that no changes have been made on the maps of the MFLBC floodplain in more recent years. The GIS 100-yr floodplain layer was overlaid on the detailed topography

[6] Table 2 does not provide the locations of anticipated coarser-grained sediment samples. The locations of these samples will be based on field conditions.

available for the MFLBC from an aerial flyover performed in 1991 as part of the Remedial Investigation. Initial sample areas have been developed based on the detailed topography and aerial photography and are shown on Figures 2 and 4. The proposed sampling locations shown on Figures 2 and 4 were selected based on the results of previous investigations. As specified in the ROD, "there are three primary sections where removal likely will be necessary: between about RM 35.4 to RM 34.8; near RM 33.3; and near RM 32.9. The PDI will include further delineation of the extent of floodplain areas to be removed to ensure the remediation goal is met." Because the original RI conducted for the Site was a multi-phased effort designed to identify all areas of the floodplain likely to have accumulated mirex (based on physical conditions and/or sampling results from earlier phases), PDI floodplain soil sampling is only needed to refine the boundaries of the remediation areas as specified in the ROD (see above) and to identify specific hot-spots within those areas that need to be removed to achieve the SWAC PRG. Therefore, PDI floodplain soil sampling will be conducted in those areas where the most recent soil samples have shown mirex concentrations above the PRG, and in the adjacent floodplain extending halfway to the nearest previous sample location that was below the PRG in both the upstream and downstream directions. If the results of the PDI indicate that the extent of mirex above the PRG within the targeted areas has not been adequately delineated, additional sampling will likely be required in conjunction with the initial steps of the detailed design (e.g., the 30% design).

Compliance with the Site-specific PRG of 1.0 mg/kg, expressed as a SWAC, will be evaluated across an exposure unit equal to one acre as described in the ROD. For purposes of the detailed design, a higher density of sampling is required so that a targeted removal approach can be developed. As such, horizontal composite soil samples will be collected from ¼-acre areas, homogenized, and archived for possible analysis. Each 1-acre exposure area will be composed of 4 analytical samples (these are the ¼ acre samples) so that, if necessary, an area as small as ¼ acre (representing about 200 cubic yards of material within the upper 6 inches) can be identified that is resulting in an exceedance of the SWAC remediation goal. In order to reduce the total number of samples analyzed, the ¼-acre composite samples will be combined into samples representing ½-acre areas. These ½-acre samples will be analyzed first and then 1-acre composite area SWACs will be calculated. For areas where this initial SWAC exceeds the remediation goal, a decision will be made whether certain ¼-acre samples should be analyzed to further refine the area requiring removal. For example, if one of the ½-acre samples is much lower than the PRG, while the other ½-acre sample is much higher than the PRG, then ¼-acre samples that make up the higher concentration area will be analyzed to determine if that ½-acre area concentration is being driven primarily by just a portion of the area. In this way, a targeted removal approach consistent with the overall approach described in the ROD can be designed. The sampling strategy targets the areas listed in the ROD as likely requiring remediation.

As specified in the ROD, the floodplain soil remedy will focus on surface soil (i.e., 0 to 6 inches) so floodplain soil samples will be collected from the 0 to 6 inch depth horizon. RI data indicates that mirex

concentration is generally inversely related to distance from (or elevation above) the stream bank. Therefore, the ½-acre samples will be composed of ¼ acre composite areas arranged parallel to the creek bank so that one of the ¼ acre samples will be closer to the stream and the other will be farther away. An example figure showing the proposed sampling approach is provided in Appendix B.

Soil from at least five discrete locations will be collected and composited from each ¼ acre sampling area, so that each 1-acre exposure unit will contain 20 or more sub-samples. This sampling approach draws on the concepts of multi-incremental sampling since at least 20 discrete locations will be sampled per exposure unit for this Site (1 acre) but sample manipulation (*e.g.*, drying, sieving, grinding) will not be conducted since the goal is to collect a sample that is representative of potential exposures to in-place soil.

The final remedy for floodplain soil will likely involve backfilling in most areas excavated so as to maintain the current floodplain profile, and given that historic data have shown decreasing mirex concentrations with depth, there should not be a need to characterize soil deeper than 6 inches. However, if, based on the detailed design, some areas will be left without backfilling, post-excavation sampling will be specified in the design to confirm that remaining surface soil mirex concentrations are below the PRG.

It is anticipated that up to 273 floodplain soil samples will be collected (91 from ½-acre composite areas, and 182 from the ¼-acre composite sub-areas). The final number of samples, however, may be adjusted based on field conditions. Samples collected from ½-acre composite areas in and immediately adjacent to previously sampled areas that exceeded the PRG will be analyzed upon receipt at the laboratory. Other composite samples from the ½-acre areas will initially be archived. These samples will then be analyzed sequentially beginning with those bordering the initial samples, followed by additional adjacent samples until a concentration below the PRG is measured across the entire floodplain width. The final sample locations and number of samples collected may also be affected by access limitations. The ¼-acre composite area samples will be archived for possible analysis pending the results of the initial samples. Floodplain soil samples will be analyzed for mirex, grain size, and TOC.

The typical maximum holding time specified for analysis of pesticides is 14 days; however, according to a USEPA reevaluation of sample holding times (USEPA, 2005), there is no significant reduction in the concentrations of a variety of pesticides stored at 4°C over a period of 217 days. Therefore, archiving some soil samples at a temperature of 4°C or lower and protecting them from ultraviolet radiation will produce reliable results when analyzed within approximately 200 days of collection. The traditional default holding times will be met for the first set of composite soil samples (those in and immediately adjacent to areas that are known to exceed the PRG).

A description of the specific investigation procedures that will be implemented to assess the extent of mirex impacts to floodplain surface soil is presented in Section 4.2.1. See Figure 7 for a map of the overall sampling plan for OU-3.

3.3.2 Physical Characterization

The riparian zone along the MFLBC is generally forested, which protects the stream from runoff and provides valuable shading and habitat opportunities for wildlife. However, the trees present an obstacle for removal of floodplain soil. In addition, areas of the floodplain may need to be used as staging locations for the targeted sediment removal remedy. In order to adequately design the remedial activities, it is important to characterize the physical condition of these areas. A description of the investigation procedures that will be used to assess the physical conditions of the floodplain is provided in Section 4.2.2.

3.4 MFLBC Fish Tissue Conditions

The SOW requires "Sampling and analysis of both whole body and fillet fish samples for mirex and lipids to provide a baseline sampling event consistent with the anticipated O&M monitoring program." The fish tissue monitoring program was discussed in the approved FS and is based on Ohio EPA's "'Indicator' Fish Species Recommendations" as provided in a memorandum dated January 22, 2008 (included herein as Appendix C). Ohio EPA recommends that up to three fish species should be targeted: common carp, yellow bullhead, and/or white sucker. White sucker and common carp have historically been collected throughout the PDI area, so these two species will likely be the most prevalent during the PDI and future sampling efforts. However, in order to maintain consistency and because the species that will be available at all stations will not be known until all stations have been sampled, there should be some flexibility in sample collection to allow for decisions regarding which fish to analyze to be made at the end of the sampling effort. As such, all species and sizes caught at the different sampling stations during the PDI will be retained until decisions have been made regarding analysis. Each fish will be prepared and wrapped separately and each will be marked with a unique identifier so that when final analysis decisions are made, the laboratory can be notified as to which samples should be homogenized/composited. As recommended by Ohio EPA based on their experience with sediment remediation in Ohio (see Appendix C), post-remediation monitoring should begin no sooner than 5 years after construction completion, so it is important to collect only fish that have been in the MFLBC for less than 5 years. For consistency, fish collected during the PDI should match those that will be targeted in the post-remediation long-term sampling, so it is anticipated that fish between 3 and 5 years old will be sampled during the PDI. The age of the fish will be determined based on the length of the fish as described in Ohio EPA's memorandum (see Appendix C).

Given the equipment requirements for fish tissue sampling, it is only feasible to collect fish where a sample staging/preparation area can be set up nearby. Therefore, consistent with previous sampling efforts, fish tissue sampling will be conducted near road crossings. There are five locations within the

target reach that will serve as sampling points for fish tissue collection: RM 37.5, RM 36.7, RM 35.4, RM 33.3, and RM 32.0 (see Figures 2 through 6). In addition, fish tissue samples will be collected from one upstream (RM 38.4) and one downstream location (RM 12.5). The final sample locations and number of samples collected may also be affected by access limitations. Fish have been collected from all of the proposed locations in at least one previous fish tissue sampling event for the Site. Most recently, in 2005, the target species (common carp, white sucker, and/or yellow bullhead) were collected at four of the proposed PDI sampling locations: RM 36.7, RM 35.4, RM 33.3, and RM 12.5. Although the intent of the baseline sampling event is not explicitly to confirm previous sampling results, it may nevertheless be useful during long-term monitoring to use all available data sets of sufficient quality in evaluating trends.

In order to provide data relevant to human health and wildlife exposures to fish, both fillet and whole body samples will be analyzed. It is not possible to analyze both the whole body and the fillet from the same fish, so fillets will be analyzed and the offal[7] from each sample will also be analyzed so that the total whole body concentration can then be calculated mathematically using the two independent concentrations. This will provide baseline data for both wildlife (whole body fish tissue results) and human health (fish fillet results) exposures. Collection of fillet and whole-body data from the same fish may make it possible to reduce the sampling program in the future (e.g. if a reliable ratio can be determined, then only fillets may be collected in the future). At each sampling location, therefore, four samples (one fillet and one offal sample for each of two species) will be submitted for laboratory analysis for a total of 28 samples. The whole body fish tissue concentrations will be calculated using the formula below:

$$WBC = \frac{(FC \times FM) + (OC \times OM)}{(FM + OM)}$$

where:

WBC = the whole body concentration of the target analyte (lipid or mirex) (mg/kg or %)

FC = the concentration of the target analyte in the fillet sample (mg/kg or %)

FM = the total mass of the fillet sample (kg)

OC = the concentration of the target analyte in the offal sample (mg/kg or %)

OM = the total mass of the offal sample (kg)

Because the calculation of whole body concentrations uses the mass of offal and fillet samples, it is important to accurately weigh both samples for each fish. Both the fillet and offal samples will be weighed initially in the field, and will subsequently be weighed upon receipt at the laboratory prior to homogenization. A description of the specific investigation procedures that will be implemented for the

[7] In this work plan, offal is being used to refer to all parts of the fish after the fillet has been removed. This includes any scales and/or skin that have been removed, as well as bones, organs, etc. The total mass of fillet and offal collected will be recorded in the field and again at the laboratory so that a mass-weighted whole body concentration can then be calculated.

baseline fish tissue mirex monitoring event is presented in Section 4.3. See Figure 7 for a map of the overall sampling plan for OU-3.

3.5 Surface Features

3.5.1 Wetlands and Floodplain

Given the targeted nature of the remedy, it is not necessary to delineate wetlands and conduct a floodplain assessment throughout the entire reach of the MFLBC that is included in the PDI. The purpose of delineating wetlands and floodplains along the stream is to allow for appropriate consideration of these location-specific requirements in the detailed design. Therefore, wetland delineation and floodplain assessment activities will be conducted in a focused manner in conjunction with the early steps of the remedial design (e.g., during preparation of the 30% design) once sediment and floodplain soil contamination have been further characterized. A description of the methods that will be used for these assessments is provided in Section 4.4.1.

3.5.2 Habitat (Aquatic and Terrestrial)

A general characterization of habitat type and quality within the stream channel and in the floodplain is necessary for the detailed design since the objectives of the remedy include protection of areas of high-quality habitat. The methods for identifying areas of high quality habitat are discussed in Section 4.4.2.

4.0 FIELD PROCEDURES

This section presents the scope of work for the various PDI components outlined in Section 3.0.

4.1 MFLBC Sediment Conditions

Equipment

Sediment sampling equipment will be constructed of inert materials such as stainless steel or Teflon. Dedicated equipment will be used when possible and if non-dedicated equipment is used, proper decontamination procedures will be followed to minimize the potential for cross-contamination. The following equipment may be required:

- Stainless Steel spoon or similar for grab sampling;
- Plastic 5-gallon bucket with bottom removed;
- Stainless steel trays and/or bowls;
- Rubber chest-high waders;
- Stainless steel spoons for homogenization;
- Latex and/or nitrile gloves;
- Sediment field information forms;
- Chain-of-Custody (COC) forms;
- Digital camera;
- Cooler with ice; and,
- Trimble GeoXH handheld mobile GIS/GPS device (or similar).

Procedure

To obtain a representative measure of chemical contamination within the sediment at each sampling site, a reach of approximately one tenth of a river mile will be walked from downstream to upstream, with equal volume sediment sub-samples taken randomly from at least 8 to 10 locations within the 0.1 RM segment (the locations of sub-samples will be recorded using a handheld GPS device). Each sub-sample will be collected using a stainless steel grab sampling device (e.g. spoon), a stainless steel bowl, and a plastic bucket with the bottom removed. At each sub-sample location, the bucket will be used to reduce the loss of fine-grained sediment from the sample. The bucket will be placed into the water with the top and bottom openings in line with the water flow direction and downstream of the sampling location. When near the sediment, the bucket will be quickly tipped upright such that it blocks the sampling location from the overlying water. After waiting at least 2 minutes to allow particles to settle, the sample will be collected using a stainless steel spoon or other grab sampling device and placed into a stainless steel bowl. A photograph will be taken of each sub-sample and a physical description of the sediment type will be recorded. The same spoon (or similar sampling device) and tray (or bowl) can be used for all sub-samples at one sample site, but a new, decontaminated spoon and tray will be used at each new 0.1 river mile sample site. The bucket will be rinsed with stream water to remove the majority of adhered sediment

particles between samples. Once collected, all sub-samples from each depth horizon will be composited together and well-mixed to provide an average sediment sample for the sampling site.

The overall goal of sediment collection is to collect sufficient material over the sample area to account for both compositional and distributional heterogeneity. Because much more sediment sample material will likely be collected than could be practically forwarded to the laboratory, the samples will be processed to provide a smaller but representative sample of material for shipment to the laboratory. Once homogenized, the entire sample from a site will be laid out in the tray or bowl and small spoon samples will be taken randomly across the mix until the analytical sample jar is filled. Excess material will be returned to the last sub-sampling location. When practical, sediment sample jars will be placed immediately on ice (to maintain 4°C) in a cooler. In some sampling areas, it will not be feasible to bring a cooler to the sediment sample location. In those cases, the samples will be held until the next opportunity to access the cooler (not to exceed 8 hours). Sediment samples will be maintained at 4°C until they are shipped to the approved laboratory. All sediment samples submitted for mirex analysis (immediate or archived) will be protected from ultraviolet radiation by using amber bottles, or by wrapping the clear glass sampling containers in aluminum foil. Initial samples will be analyzed promptly. As discussed in the approved work plan for the PDI for Operable Unit 2 (Golder, 2006), according to a USEPA reevaluation of sample holding times (USEPA, 2005), there is no significant reduction in the concentrations of a variety of pesticides, including mirex, stored at 4°C over a period of 217 days. Therefore, archiving some samples (*e.g.*, deeper samples and samples of coarse-grained material) at a temperature of 4°C or lower and protecting them from ultraviolet radiation will produce reliable results when analyzed within approximately 200 days of collection. Collected sediment samples will be transported under Chain of Custody to TestAmerica, Inc. of North Canton, Ohio for analysis as described in Section 5.1 of this Work Plan and analyzed within the holding times provided in Table 3. One field duplicate and one matrix spike/matrix spike duplicate (MS/MSD) will be collected for every twenty primary samples for quality assurance (QA) and quality control (QC) purposes. Sediment data will be reported on a dry weight basis.

The following information will be recorded on for each area sampled:

- Date;
- Sediment composite area ID;
- Approximate River Mile (to the nearest 0.1 RM);
- Start time for sampling;
- Weather conditions;
- Sampling device; and,
- Pertinent observations regarding sample characteristics (sediment type, color, odor, etc.).

Dedicated equipment will be used, or if decontamination is required, the decontamination procedures described in Appendix D will be used.

4.2 MFLBC Floodplain Soil Conditions

4.2.1 Extent of Mirex Impacts to Floodplain Surface Soil

Equipment

The following equipment items may be used for the collection of soil samples for mirex analysis:

- Latex and/or nitrile gloves;
- Cut resistant gloves (if cutting liners);
- Chain of Custody (COC) form;
- Field notebook;
- Stainless steel soil sampling device (hand held split-spoon sampler driven with a slide hammer, step probe, trowel, and/or hand-held bucket auger);
- Dedicated plastic liners (when appropriate for sample device);
- Spring retractable utility knife;
- Stainless steel mixing bowls (or trays) and spoons;
- Appropriate labeled sample bottles;
- Cooler with ice; and
- Trimble GeoXH handheld mobile GIS/GPS device (or similar).

Dedicated equipment will be used, or if decontamination is required, the decontamination procedures described in Appendix D will be used.

Sample Collection

Composite sampling techniques will be used to obtain a representative measure of chemical exposure associated with floodplain soil at each location shown in Figures 2 through 4. Locations of individual sub-samples that will be used to prepare composites are also shown in Figures 2 and 4 and will be determined in the field in consultation with Agency personnel so as to represent areas where exposure may occur and where mirex deposition could be present (based on the surface morphology and avoiding areas where there is evidence of disturbance). Soil samples will be described in the field in accordance with the Unified Soil Classification System (USCS) (ASTM D 2487), based on visual inspection. At each ¼ acre sampling area, between five and ten equal volume sub-samples will be collected using decontaminated stainless steel hand-held bucket augers, split-spoon samplers, step probes, or trowels and placed in a decontaminated stainless steel bowl. The method will be selected based on initial field reconnaissance, sample point accessibility, and current site surface conditions, and a combination of methods may ultimately be used. At each sub-sample location the sampling equipment will be advanced to a depth of 6-inches below ground surface after any surface vegetation and associated root matter is removed. Each sub-sample location will be measured using a handheld GPS device.

The sampling device (when appropriate) will be lined with a dedicated plastic liner or a decontaminated stainless steel liner for each sampling location and will be between 1 and 2 inches in diameter. The

plastic liner (if used) will be opened by slicing it with a decontaminated stainless steel spring retractable utility knife. Large debris items (such as rocks, twigs, and large roots) will be removed prior to homogenization.

After removing large debris items, the sub-sample will be thoroughly homogenized (mixed) using a decontaminated stainless steel spoon. Following homogenization, equal volume aliquots of each discrete sample will be transferred to another dedicated or decontaminated stainless steel bowl for the preparation of the composite sample as discussed below. Equal volumes will be measured using a dedicated or decontaminated measuring cup (½ cup aliquots). If any of the discrete sampling locations have insufficient recovery to yield the ½ cup volume, the samplers will choose a new location immediately adjacent to the original discrete sampling location and collect a new sample as previously described. Homogenizing the sub-samples of the composite separately before combining into one sample will reduce the effect of heterogeneities in the soil such that representative concentrations for the area are measured. Note that equipment need only be dedicated or decontaminated for each composite sampling area, so augers, bowls, spoons, etc. may be reused between sub-sample locations without decontamination. To reduce the sampling materials needed, sub-sample locations may be collected sequentially in the same bowl.

After all of the discrete sample aliquots have been added to the composite sample bowl, the material will be thoroughly homogenized using a dedicated or decontaminated stainless steel spoon. The sub-samples of the two ¼ acre portions of a ½ acre sampling area will be collected in separate bowls and will be homogenized after all sub-samples have been collected. Appropriate sample containers will be filled from the homogenized composite sample and packaged for shipment to the approved laboratory for analysis of mirex. After the ¼ acre samples have been homogenized and placed in sample containers, equal volume aliquots of the two will be combined in another dedicated or decontaminated stainless steel bowl, homogenized, and then placed in another appropriate sample container. Samples will be labeled appropriately so that it is clear which samples are to be analyzed immediately and which should be archived for possible future analysis. One field duplicate and one MS/MSD will be collected for every twenty primary samples for QA/QC purposes. For field duplicate samples, two separate sample containers will be filled from the same homogenized sample and one will be labeled as a duplicate. For MS/MSD samples, triple volume will be collected from the homogenized sample and the containers will all be labeled with the primary sample ID. The chain of custody will clearly indicate that the sample should be used for MS/MSD analysis.

All soil samples submitted for mirex analysis (immediate or archived) will be protected from ultraviolet radiation either by the use of amber glass bottles or by wrapping clear glass sampling containers in aluminum foil. Following collection of the samples, all excess soil will be returned to the borehole and the ground surface will be reinstated to a similar condition as the surrounding area. Soil samples will be

transported under Chain of Custody to TestAmerica, Inc. of North Canton, Ohio for analysis. Soil data will be reported on a dry weight basis.

The following information will be recorded for each area sampled:

- Date;
- Soil Core ID;
- Soil composite area ID;
- Approximate River Mile (to the nearest 0.1 RM);
- Start time for sampling;
- Weather conditions;
- Sampling device; and,
- Pertinent observations regarding sample characteristics (soil type, color, odor, etc.).

4.2.2 Physical Characterization

As discussed in Section 3.3.2, an assessment of the physical conditions of the floodplain soil areas targeted for potential removal will be conducted for use in the detailed design when determining the methods and extent of remediation in these areas.

Within each 0.1 RM, at least one photograph will be taken in each cardinal direction (north, south, east, west) and a qualitative assessment of the physical layout of the area will be recorded in the field notebook or in the mobile GIS device. The description will identify whether the area is open (meadow), farmland, sparsely forested, heavily vegetated/overgrown, etc., and the presence of mature trees will be noted. The ease of access for sampling and for potential remedial activities will be evaluated and recorded for each area, as well as specific potential access points where applicable.

4.3 MFLBC Fish Tissue Conditions

Equipment

Fish tissue sampling equipment will be constructed of inert materials such as stainless steel. Proper equipment decontamination procedures will be followed to minimize the potential for cross-contamination. The following equipment may be required:

- Stainless steel trays;
- Backpack or boat-mounted electroshocker;
- Rubber chest-high waders;
- Stainless steel fillet knife;
- Latex and/or nitrile gloves;
- Cut resistant gloves;
- 5-gallon buckets or other suitable containers (to temporarily store fish);
- Fish field information forms;

- Chain-of-Custody (COC) forms;
- Digital camera;
- Cooler with dry ice; and,
- Trimble GeoXH handheld mobile GIS/GPS device (or similar).

Procedure

Electroshocking

Electroshocking will be conducted by using a backpack or boat-mounted electroshocker moving from downstream to upstream so as to limit the disturbance of sediment and fish prior to collection. Backpack electroshocking may not be appropriate in areas of the stream that are too wide or deep, so other equipment, such as a boat-mounted T&J generator or Smith-Root 2.5 GPP unit will be considered for use. Staff conducting electroshocking will have the proper training and certification for the use of the device. During electroshocking, fish of the target species will be collected into a 5-gallon plastic bucket (or similar suitable container) filled with stream water for temporary containment. Once the entire stream area has been walked, the fish will be taken to the sample preparation area.

Health and safety precautions will be taken as discussed in the Health and Safety Plan (HSP) included in Appendix E. Backpack electroshocking is only possible in wadeable areas (where the water level is below the bottom of the backpack unit). In deeper portions of the stream, if encountered, a field decision will be made regarding whether to employ a vessel-based electroshocker to collect additional fish. If the area where the backpack unit cannot be used represents only a small portion of the target sampling area, it may not be necessary to collect fish from the deeper water area to produce a representative sample.

Sample Preparation

When possible, composite samples (by species) will be collected using a minimum of three fish[8] within an appropriate size range for that species (see Appendix C). Fish samples will be filleted in the field after the length and weight are measured and recorded. Fillet samples and the remaining offal will be wrapped separately in clean aluminum foil, placed in a sealed plastic bag, and stored on dry ice. Common carp, yellow bullhead, and white sucker will all be filleted with the skin off. Decontaminated stainless steel fillet knives will be used for each sample. Decontamination will include an initial water rub/rinse, cleaning with soapy water, rinse with distilled water, and final rinse with acetone. Fillet boards will be covered with clean aluminum foil for each sample. Latex/nitrile outer gloves will be worn while processing fillet samples, and will be changed between each sample. Fillet samples will be rinsed in river water prior to placing on clean aluminum foil. A minimum of 200 grams of fillet material is required for each sample. Sampling protocols will follow those listed in the Ohio EPA Fish Tissue Guidance Manual (Ohio EPA, 2004); however, it is not necessary to decontaminate aluminum foil which is used directly from the roll. All

[8] Although a minimum of three fish is optimal, final decisions regarding which samples to analyze will be determined once all sampling stations have been sampled, as indicated in Section 3.4 of the text. In some cases, a sample may be analyzed even if it does not comprise tissue from three fish.

samples will be placed on dry ice and shipped to TestAmerica of North Canton, OH for preparation/homogenization under chain of custody. Prior to preparation/homogenization of the samples the laboratory will weigh the fillet and offal samples separately to obtain total sample mass values for later use in calculating the whole-body mirex and lipid concentrations. Should sufficient tissue be available, one laboratory-prepared duplicate and one MS/MSD for fillet samples and one laboratory-prepared duplicate and one MS/MSD of the "offal" will be analyzed for QA/QC purposes. Duplicates and MS/MSDs will be obtained post-homogenization so that the samples analyzed are representative. Fish tissue data will be reported on an "as received" basis.

The selected laboratory for the PDI, TestAmerica, of North Canton, Ohio has not previously reported fish tissue data for this project. In order to establish the qualification of TestAmerica to analyze Mirex in the tissue matrix, a demonstration of method applicability (DMA) is proposed as described in Section 5.1.

4.4 Surface Features

4.4.1 Wetland and Floodplain Assessments

Wetland delineation will be conducted in accordance with the procedures outlined in the U.S. Army Corps of Engineers Wetlands Delineation Manual, Technical Report (1987). In February 2006, the Ohio EPA proposed Draft Rules for Wetland/Stream Mitigation. The proposed changes will be taken into consideration during the wetland delineation. Wetland delineation will not be conducted during the sampling phase of the PDI, since the extent of remediation is not yet known. Once samples have been collected and analyzed and the extent of mirex impacts above the PRG in sediment and floodplain soil has been determined, then wetland delineation will be conducted in the appropriate areas. To the extent necessary, floodplain determination for the detailed design will be based on published FEMA Flood Insurance Rate Maps (FIRMs).

4.4.2 Habitat (Aquatic and Terrestrial)

The PDI field activities will include identification of areas of high-quality aquatic and terrestrial (floodplain) habitat. During the PDI Reconnaissance field work, a general description of the habitat in the area of each fine-grained sediment body was recorded. This information will be supplemented during the fish and sediment sampling activities so that the areas of highest quality habitat can be identified. In general, the areas where habitat quality is highest have little fine-grained sediment, or the sediment is present only in small pockets along the banks. The procedure for identifying areas of high quality aquatic habitat was outlined in the PDI Reconnaissance Work Plan memorandum (see Appendix A) and is based upon the following parameters:

- General habitat type (riffle, run, pool, or glide).
- Predominant substrate type(s) (e.g., boulders, cobbles, sand, etc.), including identification of whether there is extensive siltation/embeddedness.

- Type(s) and extensiveness of instream cover (e.g., woody debris, overhanging vegetation, root mats, etc.).
- Channel morphology, including sinuosity, presence of channelization, and stability.
- Riparian zone quality (e.g., forest, swamp, urban, agricultural, etc.) and presence/absence of bank erosion.
- Depth of pools, pool/glide width compared to riffle/run width, and current velocity.

In general, areas of highest-quality habitat will have a mixture of riffle/run and pool/glide areas with deep pool areas that are generally wider than riffle/run areas. In addition, they have predominantly larger substrate materials (boulders, cobbles, gravel, sand), little siltation, significant and diverse instream cover, high stream sinuosity, and a wide riparian zone of high quality (such as forest or swamp) with little bank erosion.

For terrestrial (floodplain) habitat, the general description of the floodplain's physical condition (see Section 4.2.2) will be used to identify areas of potentially high-quality habitat. For example, a forested area is considered higher quality habitat while a meadow used for grazing cows is lower.

4.5 Health and Safety Plan

The Health and Safety Plan (HASP) presented in Appendix E will be followed during the implementation of the PDI field activities.

4.6 Investigation Derived Waste

Investigation Derived Waste (IDW) will be grouped in the following categories:

- Spent Decontamination Liquids; and,
- Personal Protective Equipment (PPE).

Spent decontamination liquids will be disposed of with water from the on-Site leachate collection systems (LCS-1 or LCS-2 depending on the level of impact). The leachate collected by this system is treated in the on-Site treatment plant or is shipped off-Site for disposal.

Personal Protective Equipment (PPE) will be containerized in DOT-approved 55 gallon drums and labeled as "PPE". Once a drum has been filled, it will be sealed, dated, and numbered and recorded in the field notebook.

Drums of PPE will be disposed of at an appropriate non-hazardous solid waste facility in accordance with all applicable state and federal regulations.

4.7 Field Sampling Procedures

Appendix D provides general field sampling procedures associated with the PDI activities discussed in this Section. These procedures include the following:

- Sampling Equipment Decontamination
- Field Documentation
  - Field Notebooks
  - Photographic Documentation
  - Correspondence/Communications
  - Change in Procedures
- Sample Handling, Custody, and Shipment
  - Sampling Handling
  - Sample Custody
  - Sample Packaging and Shipment
- QA/QC Sampling Requirements
  - Field Duplicates
  - MS/MSD
  - Internal QC Samples

5.0 QUALITY ASSURANCE

In general, the PDI work will be conducted in accordance with the approved QAPP for the Site. As specified in the SOW, the existing QAPP was evaluated against the needs of the PDI sampling effort and necessary modifications identified. The following sections provide an update to some of the analytical method and quality assurance (QA) requirements specified in the original QAPP and supersede the corresponding provision of the original QAPP. The original QAPP has been included in Appendix F.

The overall QA objective for this project is to develop and implement procedures for field sampling, sample custody, laboratory analysis, and data reporting that will provide results that are of known quality and that can be evaluated using known standards. Specific procedures for sampling, chain-of-custody, laboratory analysis, reporting of data, data verification, data quality assessment, and data usability evaluations to achieve the overall QA objective are described in other sections of this document.

Two types of data will be generated: definitive data and screening data. The majority of the data will be definitive analytical data that are suitable for final decision-making. Definitive data are usually generated at fixed environmental analytical laboratories, but can be obtained in the field if suitable calibration and documentation procedures are followed. Examples of definitive data to be generated are sediment mirex concentration and grain size distributions.

Screening data are data that are suitable for interim decision-making. Screening data may require confirmation by definitive data prior to final decision-making. Screening data are also usable to support definitive data. Examples of screening data anticipated are field descriptions of grain size.

5.1 Analytical Method Requirements

Samples collected as part of the PDI will be analyzed for mirex, and physical characteristics, such as TOC/lipids and grain size (for soil and sediment) as described in Table 1. The selected contract laboratory for this PDI, TestAmerica, Inc., will implement the project-required analytical methodology following established laboratory Standard Operating Procedures (SOPs). These laboratory SOPs for sample preparation, cleanup, and analysis are based on *Test Method for Evaluating Solid Waste, Physical/Chemical Methods (USEPA SW846), Final Update IIIB; June 2005;* Ohio EPA SOPs, and other applicable voluntary consensus body methods (*e.g.,* ASTM, etc.). The specific methods for analysis are included in Table 3. Samples will be preserved as specified in Table 3 and sample analyses will be performed by the laboratory within the technical holding times provided on Table 3. Field and Laboratory QC samples will be analyzed at the frequencies specified in Table 4.

TestAmerica participated in a demonstration of method comparability in March 2007 (February 28, 2008 Memo). As the data reported by TestAmerica was in good agreement with the data reported by the Ohio EPA Division of Environmental Services (DES) laboratory, TestAmerica was approved by USEPA and

Ohio EPA to report mirex data for soil and sediment samples and has been reporting mirex data for solid samples since that time.

The scope for this work plan proposes the first fish tissue sampling since TestAmerica became the preferred contract laboratory for the site. As the selected contract laboratory has not previously provided fish tissue data for this project, a demonstration of method comparability for fish tissue will be performed so that the Agencies can approve TestAmerica for fish tissue analysis for OU-3. After the fish tissue samples are collected as described in Section 4.3.2, they will be transported to TestAmerica. TestAmerica will homogenize the samples and will then preserve the homogenates at -20°C. TestAmerica will split the homogenates for the sample locations selected for analysis by both TestAmerica and Ohio EPA, and provide material to Ohio EPA. TestAmerica and Ohio EPA will proceed with analysis of three duplicate samples of fillet tissue and three duplicate samples of "offal." The selection of these six samples will be performed in the field with concurrence from Agency personnel. It is anticipated that one fillet and one offal sample from each of the three species targeted for sampling (common carp, yellow bullhead, and white sucker) will be included in this demonstration of method comparability. The reported data will proceed through the data quality assessment described in Section 5.3.1. As part of the data usability assessment, the results reported by TestAmerica, will be compared to those reported by Ohio EPA. The findings of the split sample analysis will be communicated to the Agencies in a timely fashion along with a recommendation for the laboratory to be used for the analysis of the remaining tissue samples. Once there is concurrence on the laboratory to use for the remaining samples, the laboratory will be notified and the analyses will be initiated so that the PDI data evaluation can be completed.

5.2 Reporting Requirements

The method detection limit (MDL) is the minimum concentration of a substance that can be identified, measured, and reported with a 99% confidence that the substance concentration is greater than zero, for a specific matrix containing the substance. The MDLs are determined as outlined in 40 CRF Part 136. The quantitation limit (QL) is defined as the level of measurement that can be reliably achieved within specified limits of precision and accuracy during routine laboratory operations. The QLs are generally 2-5 times greater than the MDLs, and for organic analyses are defined by the lowest concentration standard used during initial calibration.

The laboratory will report all detections above the method detection limit (MDL). Values above the MDL and below the QL will be qualified as estimated ("J" qualifier) and these data will be deemed useable for the purpose of this PDI. A summary of the MDLs and QLs for the target analytes is presented in Table 5. Target analytes, MDLs and QLs were derived from analyte-specific methodology, site-specific history, and intended data usages and may change based on sample volume, matrix effects, laboratory method, or dilutions.

The task of reporting laboratory data begins after the laboratory analysis activities have been concluded. The laboratory QA officer or designee must perform a final review of the report summaries and case narratives to determine whether the report meets project requirements. In addition to the record of chain-of-custody, the laboratory report format shall consist of the following:

Case Narrative

- Date of issuance
- Laboratory analysis performed
- Any deviations from intended analytical strategy
- Laboratory batch number
- Numbers of samples and respective matrices
- QC procedures utilized and also references to the acceptance criteria
- Laboratory report contents
- Project name and number
- Condition of samples 'as-received'
- Discussion of whether or not sample holding times were met
- Discussion of technical problems or other observations which may have created analytical difficulties
- Discussion of any laboratory QC checks which failed to meet project criteria
- Signature of the Laboratory QA Officer

Chemistry Data Package

- Case narrative for each analyzed batch of samples
- Summary page indicating dates of analyses for samples and laboratory QC checks
- Cross referencing of laboratory sample to project sample identification numbers
- Description of data qualifiers to be used
- Sample preparation and analyses for samples (including instrument run logs)
- Sample results (Analytical results will be reported as estimated **J** when detected above the MDL and below the QL.)
- Raw data for sample results and laboratory QC samples (including Laboratory Control Samples (LCS), MS/MSD, surrogates, interference check samples, serial dilutions, and method blanks)
- Results of (dated) initial calibration and continuing calibration checks, gas chromatography – mass spectrometry (GC/MS) tuning results, and gas chromatography – electron capture device (GC/ECD instrument) performance checks

All chemical data will also be submitted in an electronic data deliverable (EDD) to follow the EQuIS standard 4-file format (EFW2FSample, EFW2FLabTST, EFW2FLabRES, and EFW2FLabBCH), or the USEPA Region 5 EDD format.

5.3 Data Review, Validation, and Verification Requirements

5.3.1 Data Quality Assessment

One hundred percent of the definitive analytical chemistry data will undergo a data quality assessment (DQA) by the project chemist or designee following the review levels defined in Guidance for Labeling Externally Validated Laboratory Analytical Data for Superfund Use (EPA-540-R-08-005, January 2009). All data will undergo Stage I and Stage 2A reviews, which will entail reviewing the data for:

- sample receipt conditions;
- sample characteristics (e.g. percent moisture);
- compliance of analytical methods with the project specifications;
- compliance of compounds reported with the project specifications;
- sample holding times;
- frequency of Quality Control (QC) samples;
- method blanks;
- surrogate recoveries;
- Laboratory Control Sample (LCS) recoveries;
- MS/MSD recoveries; and,
- field quality control samples such as rinsate blanks and field duplicates.

The specific criteria measurement performance criteria (MPC) used to evaluate the quality control (QC) parameters during the DQA are those criteria identified in Tables 6 through 8. The QC criteria are derived from USEPA methodology and laboratory historical performance, and are subject to change based on periodic laboratory re-evaluation as specified in the analytical methods. Data will be qualified as estimated (J/UJ) or rejected (R) if necessary based on the results of the data evaluations.

Data qualification, if required, will follow the guidance from USEPA Contract Laboratory Program (CLP) National Functional Guidelines for Superfund Organic Methods (SOM) Data Review (EPA-540-R-08-01, June 2008), where applicable to the analytical methodologies. While the referenced USEPA National Functional Guidelines for Organic Data Review will be used as guidance during the data review and validation procedures, the documents were written for CLP Statement of Work (SOW) analyses. Since samples will be analyzed by USEPA SW-846 and ASTM methods, the USEPA National Functional Guidelines will be used where applicable to the analytical methodologies used for analysis. Where the USEPA National Functional Guidelines and method specific criteria differ, professional judgment will be used to determine specific qualifications.

5.3.2 Data Validation

If results of the DQA indicate additional data review is warranted, approximately 10% of the data will undergo a Stage 3 data validation by the project chemist or designee as defined by Guidance for Labeling Externally Validated Laboratory Analytical Data for Superfund Use (EPA-540-R-08-005, January 2009).

The data validation is a more comprehensive examination of the data than the DQA. In addition to the sample-related QC reviewed in Stage 2A, the method specific instrument performance checks (e.g. mass spectrometry tunes, pesticide breakdown checks), initial calibration data, and continuing calibration data will be evaluated relative to the specific QC criteria presented in analytical methods and the Tables 6 to 8. Select analytical results may be recalculated, such as surrogate recoveries, analytical response factors, target analyte concentrations. As in the DQA, the data will be qualified as estimated (J/UJ) or rejected (R) based on the findings of the data validations following the guidance from USEPA CLP National Functional Guidelines for SOM Data Review (June 2008), where applicable to the analytical methodologies.

5.4 Reconciliation with User Requirements

After the data have been reviewed for compliance with the analytical methods and the MPC as specified in Sections 5.2 and 5.3, the project chemist or designee will provide a preliminary summary of potential data usability concerns to the Golder Project Manager or Task Leader. The data obtained will then be both qualitatively and quantitatively assessed for data usability on a project-wide, matrix-specific, parameter-specific, and/or unit-specific basis cooperatively by the project chemist and the task leader, or their designees. This section provides performance acceptance criteria expressed as precision, accuracy, representativeness, completeness, comparability, and sensitivity (PARCCS). Where possible, acceptance criteria are specified to help delineate minimum acceptability levels for use of data in the overall decision-making process.

5.4.1 Precision

Precision refers to the degree to which repeated measurements are similar to one another. It measures the agreement (reproducibility) among individual measurements, obtained under prescribed similar conditions. Measurements that are precise are in close agreement with one another.

Field precision is assessed through the collection and measurement of field duplicates, which will be collected at an approximate rate of 1 duplicate per 20 analytical samples collected. A field duplicate sample is defined as two or more representative portions taken from the same sampling location, homogenized, split and submitted for identical analyses. The field duplicate sample is submitted to the laboratory blind (i.e. submitted as an individual sample and not identified as a field duplicate) so as to impartially represent field precision.

Precision in the laboratory is assessed through the calculation of relative percent differences (RPD) between sample results. The RPD is calculated according to the following formula.

$$RPD = \frac{2 \times |\text{Amount in Sample 1} - \text{Amount in Sample 2}|}{(\text{Amount in Sample 1} + \text{Amount in Sample 2})} \times 100$$

General precision control limits are provided in Tables 6 and 7. For GC/ECD and GC/MS organic analyses, laboratory precision shall be accessed through the analysis of matrix spike/matrix spike duplicate (MS/MSD) and field duplicate samples.. For non GC analyses, such as TOC, laboratory

precision shall be accessed through the analysis of laboratory duplicate pairs and field duplicate pairs. If the RPDs for field or laboratory duplicates are not within evaluation criteria, both results will be qualified as estimated values ("J" qualified).

5.4.2 Accuracy

Accuracy is the degree of agreement between an observed value and an accepted reference or true value. The accuracy measurement is generally determined by the percent recovery (%R) of a known value. Accuracy as %R is determined by the following equation:

$$\% R = \frac{(\text{Amount in Spiked Sample} - \text{Amount in Sample}) \times 100}{\text{Known amount added}}$$

Accuracy in the field is assessed through the use of field and trip blanks to assess the potential of cross contamination. In addition, field accuracy is assessed by the adherence to all sample handling, preservation, and holding time criteria.

Laboratory accuracy is assessed through the analysis of MS/MSD, standard reference materials (SRM), LCS, surrogate compounds, and the determination of the %R for these measurements. General accuracy control limits are provided in Tables 6, 7, and 8. Where accuracy criteria are not met, data will be qualified as either estimated (minor deviation from accuracy criteria) or rejected (major deviation from accuracy criteria). Data qualified as rejected should not be used for decision-making purposes.

5.4.3 Representativeness

Representativeness expresses the degree to which data accurately and precisely represent a characteristic of a population, a parameter variation at a sampling point, a process condition, or an environmental condition within a defined spatial and/or temporal boundary.

Representativeness is dependent upon the proper design of the sampling program and will be satisfied by ensuring that the PDI Work Plan is followed and that proper sampling techniques are used. The sampling program was designed to provide data representative of Site conditions. During development of this program, consideration was given to historical activities, existing analytical data, physical setting and processes. Using the proper analytical procedures, appropriate methods, meeting sample holding times and meeting QC criteria for each parameter, ensures representativeness in the laboratory. An additional assessment of representativeness will be made through field duplicates. While field duplicates are used to assess precision, they also indicate sample homogeneity and therefore the representativeness of the site.

5.4.4 Completeness

Completeness is a measure of the amount of valid data obtained from a measurement system compared to the amount of data that was expected under normal conditions. Data is considered valid and complete

if all QC elements have met the criteria established in this Work Plan. Qualified data may be considered usable and will be considered complete on a case by case basis.

Following completion of the analytical testing, the percent completeness will be calculated by the following equation:

$$\textit{Completeness} = \frac{\textit{(number of valid measurements) x 100}}{\textit{(number of measurements planned)}}$$

The laboratory completeness goal for this project is 90% or greater. Field measurements not collected from a specified location, or samples not collected due to environmental conditions, will be identified in the investigation report. The evaluation of field program adequacy to meet project goals will be assessed qualitatively in the report. Data qualified by the laboratory or data reviewer as estimated is usable and therefore considered complete; however, data qualified as rejected are not usable and do not count toward completeness goals.

5.4.5 Comparability

Comparability is an expression of the confidence with which one data set can be compared to another. Comparability of data is achieved by ensuring Site-wide sample collection and analyses follow the same protocol.

Comparability depends upon the proper design of the sampling program and will be satisfied by following the PDI Work Plan, SOPs, and using proper sampling techniques. The field manager will routinely oversee field activities and verify compliance to the PDI Work Plan and SOPs. Analytical data are comparable when similar sampling and analytical methods are used as identified in Section 5.1. Appropriate laboratory personnel will review and have a working knowledge of the laboratory SOPs to be used during the analysis of samples for the investigation. Additionally, the laboratory QA manager will review all data generated, verify compliance with method requirements, and ensure QA objectives are met.

Data comparability will also be evaluated through the analysis of split samples by the two independent laboratories (TestAmerica and Ohio EPA). Sample comparability for split samples will be assessed analogous to the evaluation of field duplicate samples.

5.4.6 Sensitivity

Sensitivity is defined as the capability of a method or instrument to discriminate between measurement responses representing different levels of a variable of interest. Two measurement responses of interest in assessing sensitivity are the MDL and the QL.

The sensitivity for field measurements will be determined, in part, by the limitations of field instrumentation as described in the manufacturer's manual and specific field measurement SOPs. Other factors that will influence sensitivity include matrix and environmental conditions.

The MDL and QL goals for field investigations are identified in Table 5. The laboratory will verify the QLs defined by a point on the calibration curve which is below the stated QL. Additionally the laboratory will provide MDL studies for each compound upon request by the USEPA Quality Assurance Review (QAR) and/or Golder Associates QA officer.

5.5 Data Usability Summary

The data obtained will then be both qualitatively and quantitatively assessed for data usability on a project-wide, matrix-specific, parameter-specific, and/or unit-specific basis cooperatively by the Data QA/QC Task Leader or Project Manager or their designees. Factors to be considered in this overall assessment of field and laboratory data will include, but not be limited to the following:

- Were samples obtained using the methodologies proposed in the OU-3 PDI Work Plan or in accordance with modified methods developed during the course of the work?
- Were proposed analyses performed according to the methods identified in this Work Plan or in accordance with modified methods selected during the course of the work?
- Were samples obtained from the proposed sampling locations?
- Do analytical results exhibit elevated detection limits due to matrix interference or contaminants present at high concentrations?
- Were target analytes not expected to be present at the facility, or a given unit, identified?
- Were field and laboratory data validated according to the validation protocols, including project-specific QC objectives as proposed in this Work Plan?
- Which chemical data sets were found to be unusable based on the data quality assessment and data validation results?
- Which chemical data were found to be usable based on the data quality assessment and data validation results?
- What effects do qualifiers, applied as a result of data quality assessment and data validation, have on the ability to implement project decisions?
- Can valid conclusions be drawn for all matrices at each unit and/or area under investigation?
- Were issues requiring corrective action fully resolved?
- For cases where the proposed procedures and/or requirements have not been met, has the effect of these issues on the project objectives been evaluated?

The usability assessment will be presented in the Technical memo summarizing the PDI findings.

6.0 PROJECT ORGANIZATION, REPORTING, AND SCHEDULE

6.1 Project Organization

The key personnel selected for the project have broad experience on a variety of Comprehensive Environmental Response, Compensation, and Liability Act (CERCLA) projects, including multiple remedial design investigations, treatability studies, and/or detailed design. Key team members include the following:

Golder Associates Project Director

The Project Director for the PDI will be Mr. P. Stephen Finn, C. Eng., a Principal of Golder Associates. Mr. Finn will be responsible for the overall project direction and technical evaluation of the PDI results. He will review all major technical evaluations and reports and will also be responsible for ensuring the appropriate resources are made available to ensure project execution in a timely manner.

Golder Associates Project Manager

The Project Manager for the PDI will be Mr. Kevin McCullen, a Senior Project Geologist. Mr. McCullen will be responsible for the day to day coordination of all PDI activities. He will be responsible for overseeing implementation of the field activities, scheduling, and integration of the various technical disciplines which will be required during the PDI.

Task Leaders

Mr. Andrew Joslyn, E.I.T., a Project Environmental Engineer will be the task manager for the OU-3 investigation. Ms. Julie Lehrman, CHMM, a Senior Project Environmental Chemist will be the task manager for the data quality assessment and data validation.

Field personnel

Subject to schedule coordination, it is envisioned that the field team will comprise personnel from Golder Associates and Ohio EPA.

6.2 PDI Reporting

PDI field progress will be documented in monthly progress reports to the USEPA. PDI results will be reported in a PDI Technical Memorandum as required by the SOW.

6.3 Project Schedule

Based on stream flow data obtained from a United States Geological Survey gauging station located on Little Beaver Creek (downstream of MFLBC), the highest stream flow can be expected in the spring (April-May) and stream flow in the summer is highly variable based on periodic storm events. Sampling during

high stream flow events can cause both logistical and data quality issues, so the most favorable time to conduct sampling in MFLBC is in the late summer or early fall. Therefore, PDI field work is planned between September 1 and September 30, 2010, which is within Ohio EPA's preferred sampling period for fish tissue and sediment collection. The PDI Technical Memorandum will be submitted 60 days after receipt of all analytical data. The Technical Memorandum will summarize the results of all samples that were immediately analyzed and will present recommendations for moving forward with the analyses of archived samples. The actual schedule for completion of the PDI will be dependent upon factors beyond the control of ROC including:

- Agencies review and approval time for project deliverables (a 30-day Agency review has been incorporated into the schedule);
- Access to private properties; and,
- Subcontractor availability, means and methods, and weather.

The schedule will be refined throughout the PDI and the Agencies will be kept informed as results are received from the analyses of archived samples. A preliminary schedule is provided as Figure 8.

7.0 REFERENCES

Golder, 2005. Feasibility Study for Operable Unit 2 Nease Chemical Company Salem Ohio. Golder Associates, February 2005.

Golder, 2006. PDI Work Plan for Operable Unit 2 Nease Chemical Company Salem Ohio. Golder Associates, September 2006.

Ohio EPA 2004. State of Ohio Cooperative Fish Tissue Monitoring Program Fish Collection Guidance Manual.

RNC 1990. RI/FS Work Plan, Nease Site, Salem Ohio.

USEPA, 1999. Memorandum: Issuance of Final Guidance: Ecological Risk Assessment and Risk Management Principals for Superfund Sites. OSWER Directive 9285.7-28P.

USEPA, 2005. Sample Holding Time Reevaluation. EPA/600/R-05/124. October 2005.

Table 1
Sampling Objectives and Scope
Rütgers Organics Corporation
Nease Site Operable Unit 3, Ohio

Medium	Sampling Objective	Approximate Sampling Area[1]	Depth Interval or Fish Sample Type	Number of Composite Samples[1]	Lab Analysis: Immediate or Archived	Proposed Analyses
Sediment[2]	Calculate surface weighted average concentration for each 1 RM to represent current exposure.	RM 31.0 - 37.6 (fine-grained sediment)	0-6"	65	Immediate	Mirex TOC Grain Size Total Solids
	Determine potentially exposed SWAC for future exposure.		6-12"	6	Archived	
	Determine whether high concentrations of mirex are restricted to fine-grained sediment	RM 31.0 - 37.6 (coarse-grained sediment)	0-6"	7	Archived	
Floodplain Soil[3]	Determine extent of floodplain soil PRG exceedance (1/2 acre composites)	RM 32.9, 33.3, 34.9-35.3, 35.5	0-6"	44	Immediate[4]	Mirex TOC Grain Size Total Solids
		RM 32.8, 33.0-33.1, 34.6-34.8, and 35.5-35.7	0-6"	47	Archived[4]	
	Refine the Targeted Removal Approach to meet SWAC PRG (1/4 acre composites)	RM 32.8-33.1, 33.3, 34.6-34.8, and 35.5-35.7	0-6"	182	Archived	
Fish[5]	Provide baseline data for pre-remedy condition.	RM 12.5, 32.0, 33.3, 35.4, 36.7, 37.5, and 38.4	Fillet Offal	14 14	Immediate/ Archived[6]	Mirex Lipid

Notes:

(1) Sample locations subject to change based on access. River mile designations are approximate. The stream has been segmented into 0.1 RM lengths and river mile designations refer to the entire 0.1 RM segment starting with the given river mile point and extending downstream to the next river mile point. For example, the 0.1 RM segment from RM 35.4 to RM 35.3 has been assigned a RM value of 35.4, representing the upstream end of the segment. For specific sample locations for floodplain soil refer to Figures 2 through 4.

(2) See Section 3.2 of the text for a detailed description of sampling location rationale.

(3) See Section 3.3.1 of the text for a detailed description of sampling location rationale.

(4) Samples from 1/2 acre floodplain areas within and immediately adjacent to former sample locations that exceeded the PRG will be analyzed immediately while other floodplain soil samples will be archived. The laboratory will be instructed to analyze samples from farther away if the first samples exceed the PRG. The 1/2 acre area samples will be used to calculate the SWAC within each 1 acre area of the floodplain sampled. This will determine the extent of floodplain soil PRG exceedance. Based on the results, the 1/4-acre samples would then be analyzed to determine whether specific areas within each acre could be removed to reduce the overall concentration of that 1 acre to the PRG or lower. These samples will therefore be used to refine the targeted removal approach to meet the SWAC PRG.

(5) See Section 3.4 of the text for a detailed description of sampling location rationale.

(6) All fish samples will be stored at -20 degrees C. A subset of fillet and offal samples will be homogenized, and split between Test America and Ohio EPA for comparitive analyses. The rest of the samples will be analyzed following qualification of Test America for fish tissue mirex analysis.

Table 2
Anticipated Composite Fine-Grained Sediment Sample Locations
Rütgers Organics Corporation
Nease Site Operable Unit 3, Ohio

River Mile[1]	Anticipated Number of Composite Samples[2]	Notes[3]
31.1	2	one sample from each side along stream bank
31.2	1	
31.3	2	one sample from 0-6" and one sample from 6-12"
31.4	1	
31.5	-	no fine-grained sediment identified in this segment
31.6	-	no fine-grained sediment identified in this segment
31.7	1	
31.8	2	one sample from 0-6" and one sample from 6-12"
31.9	2	one sample from 0-6" and one sample from 6-12"
32.0	1	
32.1	-	no fine-grained sediment identified in this segment
32.2	1	
32.3	2	one sample from each side along stream bank
32.4	-	no fine-grained sediment identified in this segment
32.5	-	no fine-grained sediment identified in this segment
32.6	-	no fine-grained sediment identified in this segment
32.7	-	no fine-grained sediment identified in this segment
32.8	-	no fine-grained sediment identified in this segment
32.9	1	
33.0	1	
33.1	2	one sample from each side along stream bank

Table 2
Anticipated Composite Fine-Grained Sediment Sample Locations
Rütgers Organics Corporation
Nease Site Operable Unit 3, Ohio

River Mile[1]	Anticipated Number of Composite Samples[2]	Notes[3]
33.2	1	
33.3	-	no fine-grained sediment identified in this segment
33.4	-	no fine-grained sediment identified in this segment
33.5	1	Although fine-grained sediment was not identified in this segment during the Reconnaissance, one sample will be collected for confirmation purposes based on the detection of mirex in the 2005 sampling event.
33.6	1	
33.7	-	no fine-grained sediment identified in this segment
33.8	1	
33.9	1	
34.0	-	no fine-grained sediment identified in this segment
34.1	-	no fine-grained sediment identified in this segment
34.2	1	
34.3	1	
34.4	2	one sample from each side along stream bank
34.5	1	
34.6	2	one sample from each side along stream bank
34.7	1	
34.8	1	
34.9	1	
35.0	1	

Table 2
Anticipated Composite Fine-Grained Sediment Sample Locations
Rütgers Organics Corporation
Nease Site Operable Unit 3, Ohio

River Mile[1]	Anticipated Number of Composite Samples[2]	Notes[3]
35.1	3	one sample from each side along stream bank, plus one deep sample from 6-12"
35.2	1	
35.3	1	
35.4	-	no fine-grained sediment identified in this segment
35.5	-	no fine-grained sediment identified in this segment
35.6	-	no fine-grained sediment identified in this segment
35.7	2	one sample from each side along stream bank
35.8	1	
35.9	1	
36.0	2	one sample from each side along stream bank
36.1	1	
36.2	1	
36.3	2	one sample from each side along stream bank
36.4	2	one sample from each side along stream bank
36.5	1	
36.6	-	no fine-grained sediment identified in this segment
36.7	2	one sample from each side along stream bank
36.8	2	one sample from each side along stream bank
36.9	2	one sample from each side along stream bank
37.0	2	one sample from each side along stream bank
37.1	1	

Table 2
Anticipated Composite Fine-Grained Sediment Sample Locations
Rütgers Organics Corporation
Nease Site Operable Unit 3, Ohio

River Mile[1]	Anticipated Number of Composite Samples[2]	Notes[3]
37.2	2	one sample from each side along stream bank
37.3	2	one sample from each side along stream bank
37.4	1	
37.5	1	
37.6	1	

Notes:

(1) The stream has been segmented into 0.1 RM lengths and river mile designations refer to the entire 0.1 RM segment starting with the given river mile point and extending downstream to the next river mile point. For example, the 0.1 RM segment from RM 35.4 to RM 35.3 has been assigned a RM value of 35.4, representing the upstream end of the segment.

(2) This table shows only the anticipated locations of fine-grained sediment samples. In addition to these samples, one sample from coarser-grained sediment (i.e., sand-dominated) will be collected from each river mile. For more information, see Section 3.2 of the text. Also note that this table lists four locations where fine-grained sediment was encountered at depths greater than 6 inches during the Reconnaissance. The PDI work plan allows for collection of up to 6 samples to account for the fact that there was a portion of the stream that could not be mapped during the Reconnaissance.

(3) In 0.1 river mile segments where there were distinct sediment bodies on both sides of the stream, these sediment bodies will be sampled separately such that one composite sample from along one bank is collected, and a separate composite sample along the other bank is also collected. For all other segments, the sediment bodies appear on only one side of the stream or the other, so only one composite sample will be collected. Please see Section 3.2 of the text for additional detail.

Table 3
Analytical Methods, Sample Containers, Sample Volume, Preservation
And Analytical Hold Times for Soil, Sediment and Tissue Samples
Rütgers Organics Corporation
Nease Site, Salem, Ohio

PARAMETER	METHODOLOGY	CONTAINER	MINIMUM SAMPLE	PRESERVATION [1]	HOLD TIME [2]
SOIL					
Mirex	SW-846 Method 8081A	8 oz G	50 g	Cool 4° C, protect from light [3]	200 days [4]
Total Organic Carbon	Walkley-Black	8 oz G	50 g	Cool 4° C	28 days
Moisture Content	ASTM D2216	8 oz G	5 g	Method	Method
SEDIMENT					
Mirex	SW-846 Method 8081A	8 oz G	50 g	Cool 4° C, protect from light	200 days [4]
Total Organic Carbon	Walkley-Black	8 oz G	50 g	Cool 4° C	28 days
Moisture Content	ASTM D2216	8 oz G	5 g	Method	Method
Grain Size	ASTM D422	1 gal P bag	-	Method	Method
TISSUE					
Mirex	SW-846 Method 8081A	See Note 5	50 g	Cool -20 °C, protect from light [4]	1 year [6]
Percent Lipids		See Note 5	-	Cool -20 °C	Method

Notes:

1. Sample preservation is performed by sampler immediately upon sample collection.
2. Hold time based upon day of sample collection not Verified Time of Sample Receipt.
3. Mirex samples will be protected from UV radiation by the use of amber glass bottles, or by covering sample containers with aluminum foil.
4. The traditional holding time for pesticide analysis in solid samples requires extraction of the sample within 14 days of collection; and analysis of the extract within 45 days of creation. When possible, mirex samples will adhere to these holding times. However, as discussed in the approved work plan for the PDI for Operable Unit 2 (Golder, 2006), according to a USEPA reevaluation of sample holding times (USEPA, 2005), there is no significant reduction in the concentrations of a variety of pesticides, including mirex, stored at 4°C over a period of 217 days. Therefore, the project specific holding time for archived samples is extraction within 200 days of collection.
5. Tissue samples will be wrapped in aluminum foil and packaged within clean plastic bags.
6. The holding time for tissue samples at -20 °C is 1 year. Tissue samples will be extracted for Mirex within 14 days of thawing and extracts will be analyzed within 45 days of extract creation analogous to the holding times for other solid samples.

P indicates that Plastic should be used.
G indicates that Glass should be used.

Golder Associates

Table 4
Quality Control Sample Frequency
Rütgers Organics Corporation
Nease Site Operable Unit 3, Ohio

PARAMETER	METHODOLOGY	Field Duplicate Frequency (1)	MS/MSD Frequency (1)
SOIL			
Mirex	SW-846 Method 8081A	1 per 20 samples	1 per 20 samples
Total Organic Carbon	Walkley-Black	1 per 20 samples	1 per 20 samples
Moisture Content	ASTM D2216	1 per 20 samples	1 per 20 samples
SEDIMENT			
Mirex	SW-846 Method 8081A	1 per 20 samples	1 per 20 samples
Total Organic Carbon	Walkley-Black	1 per 20 samples	1 per 20 samples
Moisture Content	ASTM D2216	1 per 20 samples	1 per 20 samples
Grain Size	ASTM D422	1 per 20 samples	1 per 20 samples
TISSUE			
Mirex	SW-846 Method 8081A	1 per 20 samples	1 per 20 samples
Percent Lipids	Gravimetric	1 per 20 samples	1 per 20 samples

Notes:

1. Field duplicates and MS/MSD will not be collected for archived samples, as the locations for analysis are not known. The laboratory will randomly pick locations for duplicate and MS/MSD analysis upon sample activation.

Table 5
Method Detection and Laboratory Quantitation Limits
Rütgers Organics Corporation
Nease Site Operable Unit 3, Ohio

PARAMETERS	MDL	QL	UNITS
Soil/Sediment			
Mirex	2.2	3.3	ug/kg
Total Organic Carbon (1)	0.25	1	mg/kg
Total Solids (2)	10	10	%
Grain Size Distribution	NA	NA	-
Tissue			
Mirex	2.2	3.3	ug/kg
% lipids	0.030	0.1	%

Notes:
MDL and QL assume 30g sample extracted, 100% solids, and sample extracts analyzed without dilution.
Sample specific MDL and RL may be adjusted proportional to sample size, moisture content, matrix effects and concentration of target and non-target compounds
SWAC = Surface Weighted Average Concentration
MDL = method detection limit
QL = quantitation limit
ug/kg = micrograms per kilogram (parts per billion)
mg/kg = milligrams per kilogram (parts per million)

(1) TOC = Walkely Black Method

(2) The MDL for Total Solids cannot be determined, so it is set equal to the QL. The QL is set to 10% because below this point, the sample is no longer considered a "solid" matrix. The accuracy of the total solids measurements is 0.1%, so results reported above 10% will be reported to the nearest 0.1% value.

Table 6
Laboratory Control Spike Recovery Limits
Rütgers Organics Corporation
Nease Site Operable Unit 3, Ohio

PARAMETERS	LCL	UCL	RPD
Soil/Sediment			
Mirex	50	150	30
Total Organic Carbon (1)	51	128	20
Total Solids	NA	NA	NA
Grain Size Distribution	NA	NA	NA
Tissue			
Mirex	50.0	150	30
% lipids	NA	NA	NA

Notes:
LCL = lower control limit
UCL = upper control limit
RPD = relative percent difference

1) TOC = Walkley Black

Golder Associates

Table 7

Field Quality Control Sample Recovery and Precision Limits

Rütgers Organics Corporation

Nease Site Operable Unit 3, Ohio

	MATRIX SPIKE/MATRIX SPIKE DUPLICATE			FIELD DUPLICATE
PARAMETERS	**LCL**	**UCL**	**RPD**	**RPD (2)**
Soil/Sediment				
Mirex	50	150	50	50
Total Organic Carbon (1)	51	128	50	50
Total Solids	NA	NA	NA	20
Grain Size Distribution	NA	NA	NA	NA
Tissue				
Mirex	50.0	150	50	50
% lipids	NA	NA	NA	20

Notes:

LCL = lower control limit

UCL = upper control limit

RPD = relative percent difference

1) Total Organic Carbon will be analyzed following the Walkley Black method

2) If either result is non-detect or below the quantitation limit, the results will be assessed against the alternate precision limit of ± quantitation limit

Golder Associates

Table 8

Surrogate Spike Recovery Limits

Rütgers Organics Corporation

Nease Site Operable Unit 3, Ohio

PARAMETERS	LCL	UCL
Soil/Sediment		
Decachlorobiphenyl	18	145
Tetrachloro-m-xylene	31	131
Tissue		
Decachlorobiphenyl	10	199
Tetrachloro-m-xylene	10	199

Notes:

LCL = lower control limit

UCL = upper control limit

Surrogate recoveries will not be calculated for samples analyzed at more than a 20 x dilution.

Surrogate recoveries for samples analyzed with between a 5x and 20x dilution may be affected by dilution effects.

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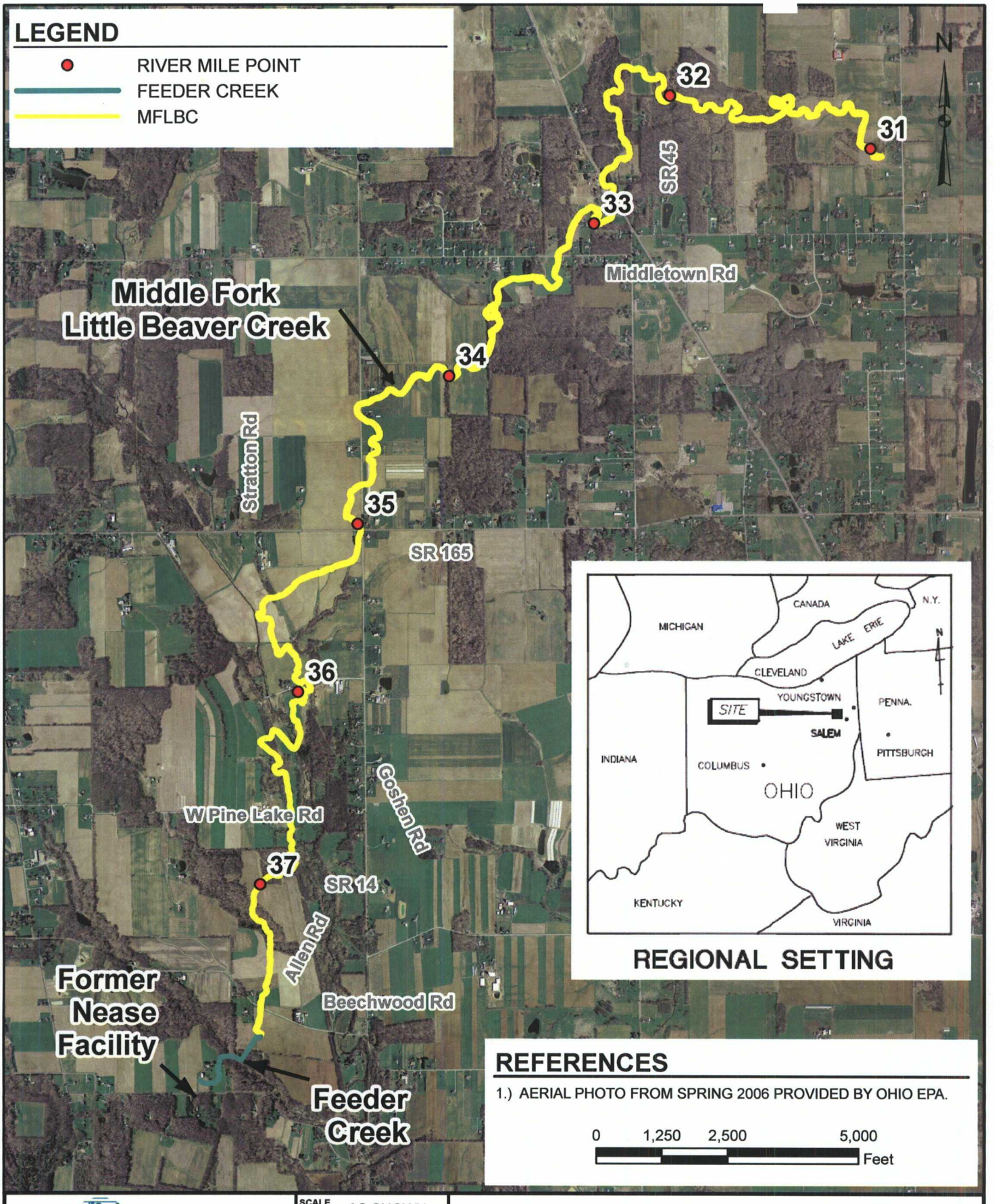


REFERENCES

1.) AERIAL PHOTO FROM SPRING 2006 PROVIDED BY OHIO EPA.

0 1,250 2,500 5,000 Feet

Golder Associates
Philadelphia USA

SCALE	AS SHOWN
DATE	3/17/2010
DESIGN	PSF
GIS	APJ
CHECK	APJ
REVIEW	PSF
FILE No.	9336154S003
PROJECT No.	9336154
REV.	0

OPERABLE UNIT 3 LOCATION MAP

RÜTGERS ORGANICS CORPORATION

FIGURE 1

Map Document: V:\Projects\1993\933-6154\REPORT_FILES\S_OU3_PDI_Workplan\MXD\9336154S003.mxd / Modified 3/17/2010 7:58:18 AM / Plotted 3/17/2010 7:58:31 AM by AMorales

LEGEND

- RI FLOODPLAIN SOIL SAMPLE LOCATION WITH MIREX > 1.0 MG/KG
- RI FLOODPLAIN SOIL SAMPLE LOCATION WITH MIREX < 1.0 MG/KG
- OBSTRUCTION
- RIVER MILE POINT
- FINE GRAIN SEDIMENT LOCATION
- OU-3 PDI PROPOSED FLOODPLAIN SAMPLING AREA
- 1/2 ACRE FLOODPLAIN SAMPLING AREA FOR IMMEDIATE ANALYSIS
- 2006 COMPOSITE FLOODPLAIN SOIL SAMPLE LOCATION
- MIDDLE FORK LITTLE BEAVER CREEK

NOTE

1.) FOR PREVIOUS FLOODPLAIN SOIL MIREX CONCENTRATIONS REFER TO THE OU-3 FEASIBILITY STUDY.

2.) FLOODPLAIN SAMPLING AREAS SHOWN ARE 1/2 ACRE AREAS. ALL 1/2 ACRE AREAS WILL ALSO BE SPLIT INTO TWO 1/4 ACRE SAMPLES THAT WILL BE ARCHIVED.

REFERENCE

1.) AERIAL FROM SPRING 2006 PROVIDED BY OHIO EPA.

400 200 0 400 Feet

Golder Associates
Philadelphia USA

FILE No.	9336154S008
PROJECT No.	933-6154 REV. 1
SCALE	AS SHOWN
DATE	9/1/2010
DESIGN	APJ
GIS	AM
CHECK	APJ
REVIEW	PSF

FINE GRAINED SEDIMENT AREAS AND PROPOSED FLOODPLAIN SAMPLING RIVER MILE 31.0-33.1

RÜTGERS ORGANICS CORPORATION

FIGURE 2

Map Document: V:\Projects\1993\933-6154\REPORT_FILES\S_OU3_PDI_Workplan\MXD\9336154S008-rev1.mxd / Modified 9/1/2010 1:52:39 PM / Plotted 9/1/2010 1:53:06 PM by AMorales

LEGEND

- RI FLOODPLAIN SOIL SAMPLE LOCATION WITH MIREX > 1.0 MG/KG
- RI FLOODPLAIN SOIL SAMPLE LOCATION WITH MIREX < 1.0 MG/KG
- OBSTRUCTION
- RIVER MILE POINT
- FINE GRAIN SEDIMENT LOCATION
- OU-3 PDI PROPOSED FLOODPLAIN SAMPLING AREA
- 1/2 ACRE FLOODPLAIN SAMPLING AREA FOR IMMEDIATE ANALYSIS
- 2006 COMPOSITE FLOODPLAIN SOIL SAMPLE LOCATION
- MIDDLE FORK LITTLE BEAVER CREEK

NOTE

1.) FOR PREVIOUS FLOODPLAIN SOIL MIREX CONCENTRATIONS REFER TO THE OU-3 FEASIBILITY STUDY.

2.) FLOODPLAIN SAMPLING AREAS SHOWN ARE 1/2 ACRE AREAS. ALL 1/2 ACRE AREAS WILL ALSO BE SPLIT INTO TWO 1/4 ACRE SAMPLES THAT WILL BE ARCHIVED.

REFERENCE

1.) AERIAL FROM SPRING 2006 PROVIDED BY OHIO EPA.

400 200 0 400 Feet

Golder Associates
Philadelphia USA

SCALE	AS SHOWN
DATE	9/1/2010
DESIGN	APJ
GIS	AM
CHECK	APJ
REVIEW	PSF
FILE No.	9336154S009
PROJECT No.	933-6154
REV.	1

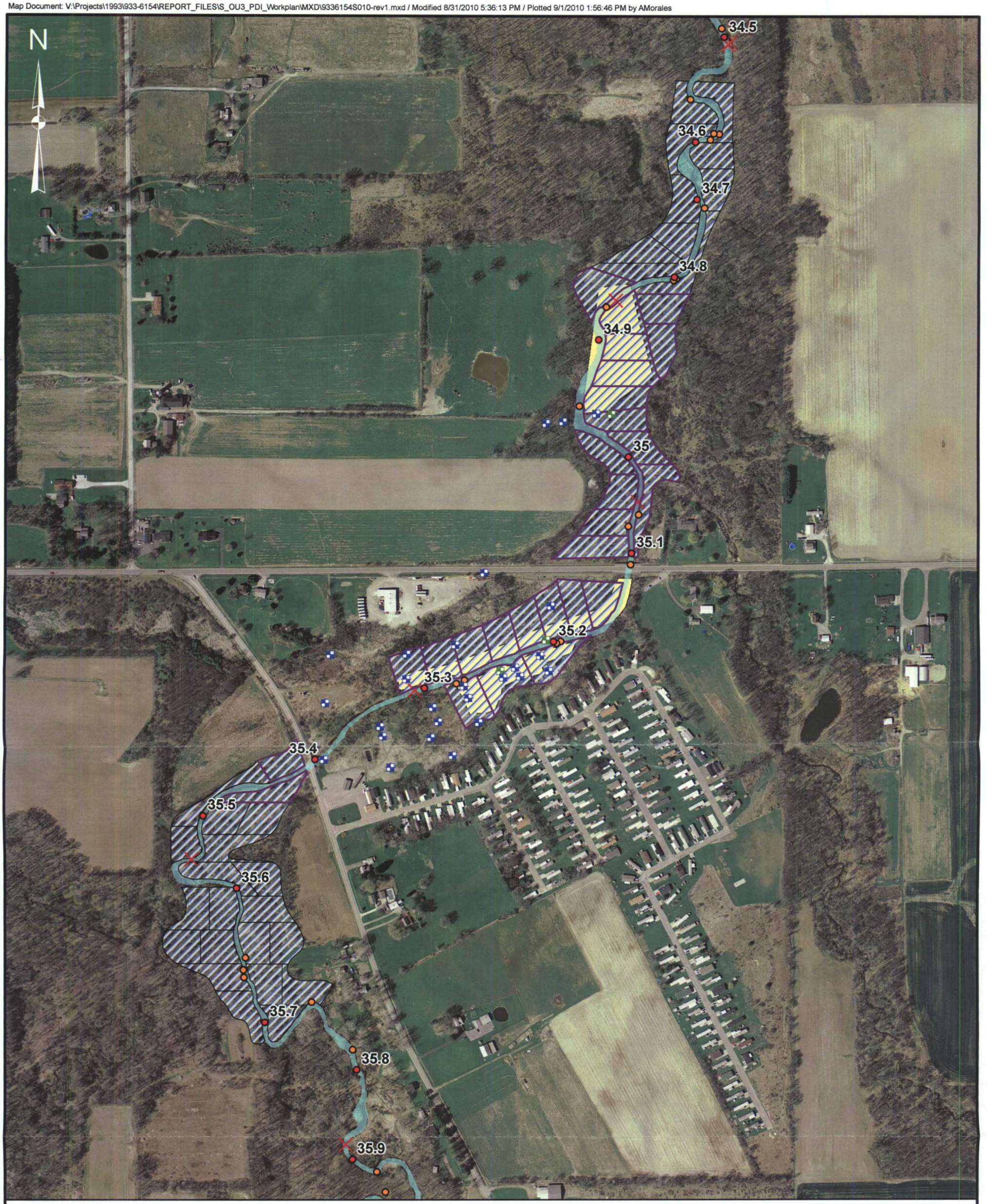
FINE GRAINED SEDIMENT AREAS AND PROPOSED FLOODPLAIN SAMPLING RIVER MILE 33.2-34.4

RÜTGERS ORGANICS CORPORATION

FIGURE 3

Map Document: V:\Projects\19930\933-6154\REPORT_FILES\S_OU3_PDI_Workplan\MXD\9336154S009-rev1.mxd / Modified 9/1/2010 1:54:39 PM / Plotted 9/1/2010 1:55:03 PM by AMorales

Map Document: V:\Projects\1993\933-6154\REPORT_FILES\S_OU3_PDI_Workplan\MXD\9336154S010-rev1.mxd / Modified 8/31/2010 5:36:13 PM / Plotted 9/1/2010 1:56:46 PM by AMorales



LEGEND

- RI FLOODPLAIN SOIL SAMPLE LOCATION WITH MIREX > 1.0 MG/KG
- RI FLOODPLAIN SOIL SAMPLE LOCATION WITH MIREX < 1.0 MG/KG
- OBSTRUCTION
- RIVER MILE POINT
- FINE GRAIN SEDIMENT LOCATION
- 1/2 ACRE FLOODPLAIN SAMPLING AREA FOR IMMEDIATE ANALYSIS
- OU-3 PDI PROPOSED FLOODPLAIN SAMPLING AREA
- MIDDLE FORK LITTLE BEAVER CREEK
- 2006 COMPOSITE FLOODPLAIN SOIL SAMPLE LOCATION

NOTE

1.) FOR PREVIOUS FLOODPLAIN SOIL MIREX CONCENTRATIONS REFER TO THE OU-3 FEASIBILITY STUDY.

2.) FLOODPLAIN SAMPLING AREAS SHOWN ARE 1/2 ACRE AREAS. ALL 1/2 ACRE AREAS WILL ALSO BE SPLIT INTO TWO 1/4 ACRE SAMPLES THAT WILL BE ARCHIVED.

REFERENCE

1.) AERIAL FROM SPRING 2006 PROVIDED BY OHIO EPA.

400 200 0 400 Feet

FIGURE 4

REVIEW	PSF	9/1/2010
CHECK	APJ	9/1/2010
GIS	AM	9/1/2010
DESIGN	APJ	9/1/2010
REV. 1	SCALE: AS SHOWN	
FILE No.	9336154S010	
PROJECT No.	933-6154	

TITLE

FINE GRAINED SEDIMENT AREAS AND PROPOSED FLOODPLAIN SAMPLING RIVER MILE 34.5-35.9

PROJECT

RÜTGERS ORGANICS CORPORATION

Golder Associates
Philadelphia USA

Map Document: V:\Projects\1993\933-6154\REPORT_FILES\S_OU3_PDI_Workplan\MXD\9336154S011-rev1.mxd / Modified 9/1/2010 1:57:47 PM / Plotted 9/1/2010 1:58:09 PM by AMorales

LEGEND

- RI FLOODPLAIN SOIL SAMPLE LOCATION
- OBSTRUCTION
- RIVER MILE
- FINE GRAIN SEDIMENT LOCATION
- MIDDLE FORK LITTLE BEAVER CREEK

NOTE

1.) FOR PREVIOUS FLOODPLAIN SOIL MIREX CONCENTRATIONS REFER TO THE OU-3 FEASIBILITY STUDY.

REFERENCE

1.) AERIAL FROM SPRING 2006 PROVIDED BY OHIO EPA.

400 200 0 400 Feet

FIGURE 5

REVIEW	PSF	9/1/2010
CHECK	APJ	9/1/2010
GIS	AM	9/1/2010
DESIGN	APJ	9/1/2010
REV. 1	SCALE: AS SHOWN	
FILE No.	9336154S011	
PROJECT No.	933-6154	

TITLE

FINE GRAINED SEDIMENT AREAS
RIVER MILE 36.0-37.3

PROJECT

RÜTGERS ORGANICS CORPORATION

Golder Associates
Philadelphia USA

Map Document: V:\Projects\1993\933-6154\REPORT_FILES\S_OU3_PDI_Workplan\MXD\9336154S012-rev1.mxd / Modified 9/1/2010 1:58:59 PM / Plotted 9/1/2010 1:59:16 PM by AMorales

LEGEND

- OBSTRUCTION
- RIVER MILE
- FINE GRAIN SEDIMENT LOCATION
- MIDDLE FORK LITTLE BEAVER CREEK
- 2006 COMPOSITE FLOODPLAIN SOIL SAMPLE LOCATION

NOTE

1.) FOR PREVIOUS FLOODPLAIN SOIL MIREX CONCENTRATIONS REFER TO THE OU-3 FEASIBILITY STUDY.

REFERENCE

1.) AERIAL FROM SPRING 2006 PROVIDED BY OHIO EPA.

400 200 0 400 Feet

FIGURE 6

REVIEW	PSF	9/1/2010
CHECK	APJ	9/1/2010
GIS	AM	9/1/2010
DESIGN	APJ	9/1/2010
REV. 1	SCALE: AS SHOWN	
FILE No.	9336154S012	
PROJECT No.	933-6154	

TITLE

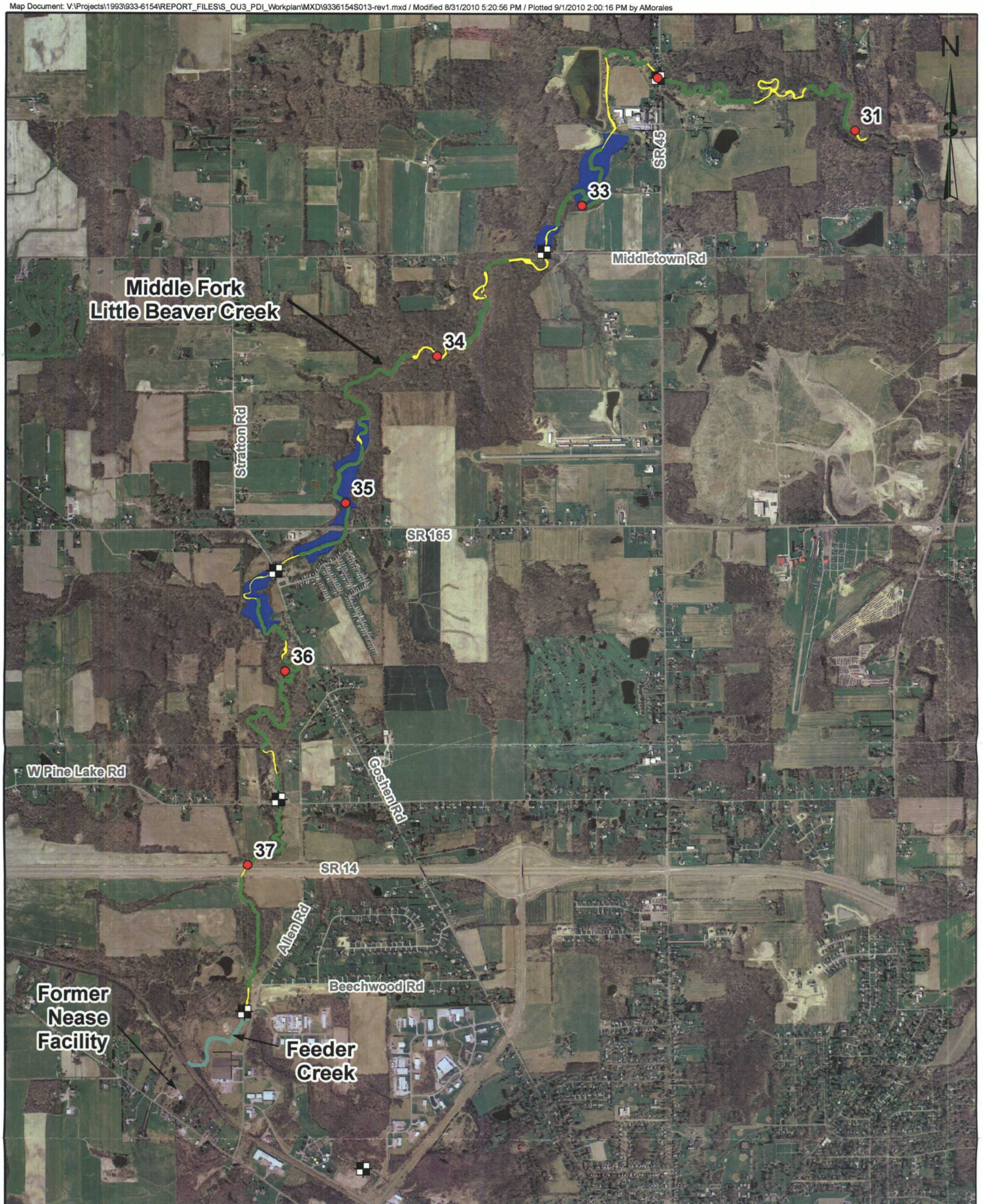
FINE GRAINED SEDIMENT AREAS RIVER MILE 37.4-38.4

PROJECT

RÜTGERS ORGANICS CORPORATION

Golder Associates
Philadelphia USA

Map Document: V:\Projects\1993\933-6154\REPORT_FILES\S_OU3_PDI_Workplan\MXD\9336154S013-rev1.mxd / Modified 8/31/2010 5:20:56 PM / Plotted 9/1/2010 2:00:16 PM by AMorales



LEGEND

- RIVER MILE POINT
- PROPOSED FISH TISSUE SAMPLE LOCATION
- PROPOSED SEDIMENT SAMPLING AREA
- PROPOSED FLOODPLAIN SAMPLING AREA
- FEEDER CREEK
- MFLBC

NOTES

1.) FISH TISSUE SAMPLES WILL ALSO BE COLLECTED DOWNSTREAM AT RM 12.5 NEAR LISBON DAM.

REFERENCES

1.) AERIAL PHOTOGRAPH FROM SPRING 2006 PROVIDED BY OHIO EPA.

1,800 900 0 1,800 Feet

FIGURE 7

REVIEW	PSF	9/1/2010
CHECK	APJ	9/1/2010
GIS	AM	9/1/2010
DESIGN	APJ	9/1/2010
REV. 1	SCALE: AS SHOWN	
FILE No.	9336154S013	
PROJECT No.	933-6154	

TITLE

PROPOSED OVERALL PDI SAMPLING PROGRAM

PROJECT

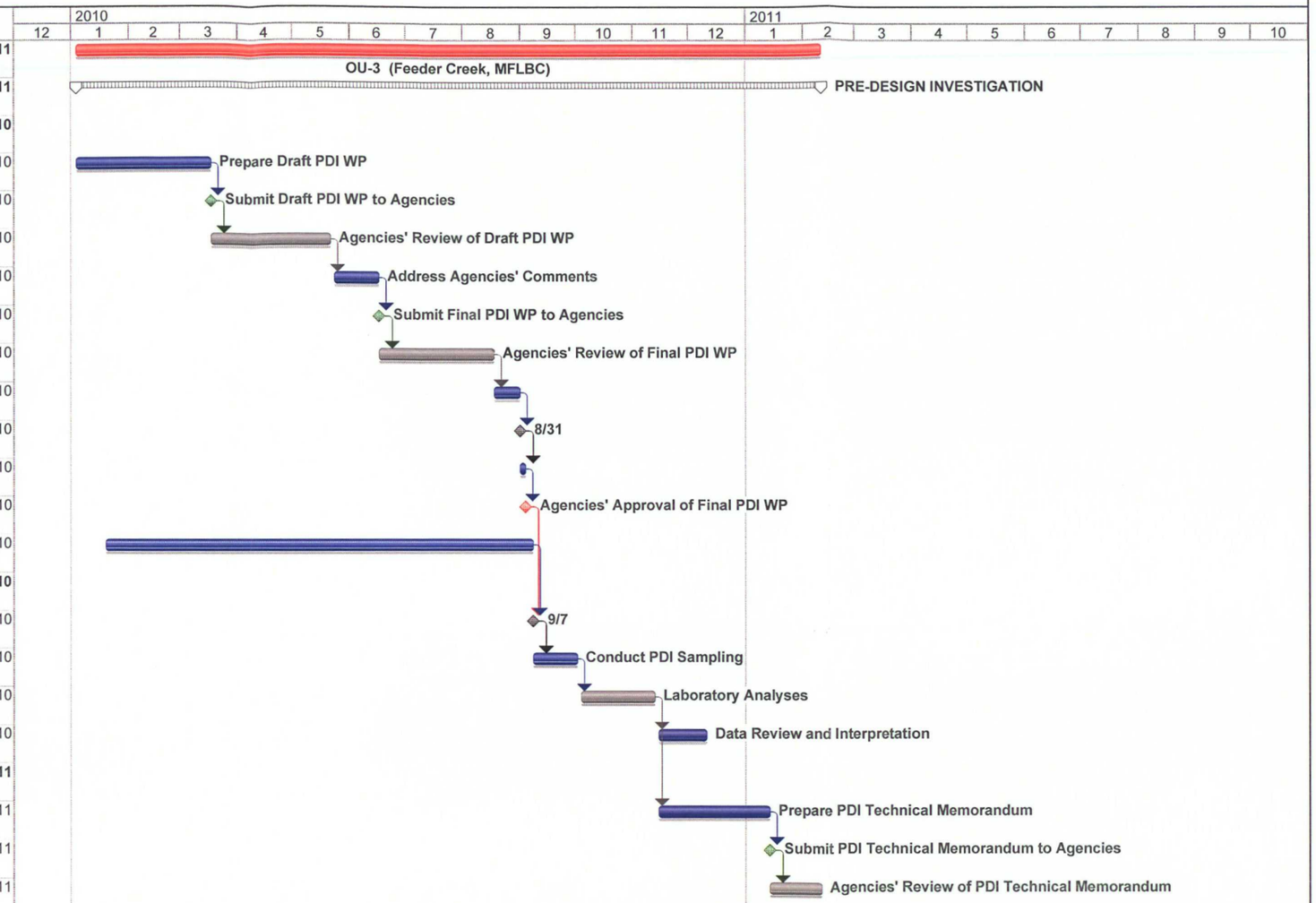
RÜTGERS ORGANICS CORPORATION

Golder Associates
Philadelphia USA

August 2010

Figure 8
Preliminary OU-3 Pre-Design Investigation Schedule
Rütgers Organics Corporation
Nease Site Operable Unit 3, Ohio

ID	Task Name	Duration	Start	Finish
1	**OU-3 (Feeder Creek, MFLBC)**	**289 days**	**Mon 1/4/10**	**Thu 2/10/11**
2	**PRE-DESIGN INVESTIGATION**	**289 days**	**Mon 1/4/10**	**Thu 2/10/11**
3	**PDI Work Plan**	**177 days**	**Mon 1/4/10**	**Tue 9/7/10**
4	Prepare Draft PDI WP	53 days	Mon 1/4/10	Wed 3/17/10
5	Submit Draft PDI WP to Agencies	0 days	Wed 3/17/10	Wed 3/17/10
6	Agencies' Review of Draft PDI WP	47 days	Thu 3/18/10	Fri 5/21/10
7	Address Agencies' Comments	18 days	Mon 5/24/10	Wed 6/16/10
8	Submit Final PDI WP to Agencies	0 days	Wed 6/16/10	Wed 6/16/10
9	Agencies' Review of Final PDI WP	44 days	Thu 6/17/10	Tue 8/17/10
10	Adress Agencies' Comments on Final PDI WP	10 days	Wed 8/18/10	Tue 8/31/10
11	Submit Revised Final PDI WP	0 days	Tue 8/31/10	Tue 8/31/10
12	Agencies' Review of Revised Final PDI WP	3 days	Wed 9/1/10	Fri 9/3/10
13	Agencies' Approval of Final PDI WP	0 days	Fri 9/3/10	Fri 9/3/10
14	Obtain Access Agreements	165 days	Wed 1/20/10	Tue 9/7/10
15	**PDI Field Work**	**68 days**	**Tue 9/7/10**	**Fri 12/10/10**
16	Mobilization	0 days	Tue 9/7/10	Tue 9/7/10
17	Conduct PDI Sampling	18 days	Wed 9/8/10	Fri 10/1/10
18	Laboratory Analyses	30 days	Mon 10/4/10	Fri 11/12/10
19	Data Review and Interpretation	20 days	Mon 11/15/10	Fri 12/10/10
20	**PDI Technical Memorandum**	**64 days**	**Mon 11/15/10**	**Thu 2/10/11**
21	Prepare PDI Technical Memorandum	44 days	Mon 11/15/10	Thu 1/13/11
22	Submit PDI Technical Memorandum to Agencies	0 days	Thu 1/13/11	Thu 1/13/11
23	Agencies' Review of PDI Technical Memorandum	20 days	Fri 1/14/11	Thu 2/10/11



Golder Associates

NEASE CHEMICAL SITE, OH
OU-3 PRE-DESIGN INVESTIGATION

APPENDIX A
PDI RECONNAISSANCE WORK PLAN MEMORANDUM

MEMORANDUM

TO: Mary Logan, USEPA

August 24, 2009
Project No.: 933-6154

cc: Sheila Abraham, Ohio EPA
Rainer Domalski, ROC

FR: Andrew Joslyn / P. Stephen Finn, Golder Associates

RE: **PDI RECONNAISSANCE WORK PLAN**
RIVER MILE 31 TO 37.6, MIDDLE FORK LITTLE BEAVER CREEK, OH

This PDI reconnaissance work plan memorandum is being submitted on behalf of RÜTGERS Organics Corporation (ROC) for Operable Unit 3 (OU-3) of the Nease Chemical Superfund Site, located in Columbiana and Mahoning Counties, Ohio (Site).

Background:

The Nease facility, a former chemical manufacturing plant, is located about 2 ½ miles northwest of the town of Salem, in Columbiana County. The Site consists of three operable units, with Operable Unit 3 (OU-3) addressing Feeder Creek and contaminated sediment and floodplain soil impacted by mirex in portions of the Middle Fork of Little Beaver Creek (MFLBC). The USEPA issued a Record of Decision (ROD) for OU-3 in September 2008. As specified in the ROD, the selected remedy includes targeted removal of sediment from the MFLBC to achieve a surface area-weighted average concentration (SWAC) goal within an approximately 6.5 mile stretch of the stream from the discharge point of Feeder Creek at River Mile (RM) 37.6 to RM 31.0. On June 30, 2009, an "Administrative Settlement Agreement and Order on Consent for Remedial Design" (Settlement Agreement) was signed by ROC and the USEPA, under which ROC is conducting the Remedial Design (RD) for OU-3.

As required by the Settlement Agreement, a Pre-Design Investigation (PDI) is to be conducted to further delineate the distribution of mirex in sediment and floodplain soil and to establish pre-construction baseline conditions. As specified in the Statement of Work (SOW) portion of the Settlement Agreement, Site reconnaissance is necessary prior to submittal of the OU-3 PDI Work Plan. The reconnaissance is to include a Site visit and discussions with potentially affected property owners to identify areas that may need to be assessed as part of the OU-3 PDI. This initial Site reconnaissance is the focus of this work plan memorandum.

Objectives:

The primary objective of the PDI Reconnaissance is to provide additional information about stream morphology and habitat to determine target areas for collection of PDI sediment samples for mirex analysis between RM 37.6 and RM 31.0. Mirex is associated primarily with fine-grained, organic-rich sediment, so the reconnaissance will focus on identifying the locations of fine-grained sediment bodies in the target reach. In addition, physical access and habitat information gathered during the reconnaissance will be used during the design of the targeted removal remedy. In order to facilitate dynamic decision making and efficient data collection in the PDI, the Site Reconnaissance described in this memorandum also includes some portions of the PDI scope as outlined in the SOW, in addition to the basic requirements specified for the Site Reconnaissance.

The reconnaissance will include the following tasks, as further described in the subsequent sections:

- Detailed mapping of fine-grained sediment bodies between RM 37.6 through 31 using a handheld GPS device.
- Detailed description of fine-grained sediment through collection of cores of sediment within the mapped sediment bodies for visual assessment and photographic documentation.
- Description of general sediment type in areas adjacent to fine-grained sediment bodies.
- Laboratory grain size analyses of each significant sediment type encountered.
- Photographic documentation of the stream channel and banks at each 0.1 river mile.
- Mapping of obstructions within the stream that might interfere with investigation or remediation activities (including photographic documentation).
- Identification of areas of high quality habitat.
- Discussions with potentially affected property owners.

Methods:

- **Detailed mapping of fine-grained sediment bodies**
    - The outlines of fine-grained sediment bodies will be mapped with a portable GPS device with accuracy to 1 foot or better by walking along the perimeter of the sediment body and collecting a vertex point every 5 seconds, or at an appropriate frequency to achieve a reliable polygon representing the sediment body. The frequency will be adjusted based on field conditions, such as the size and shape of the sediment body (e.g., the more curved the boundary of a sediment body, the more frequently vertices need to be collected). The GPS device will be running a Mobile Geographic Information System (Mobile GIS) application (e.g. ArcPad by ESRI) for entering attributes about the sediment bodies such as the River Mile and a general description.

- Sediment body thickness will be determined by collecting one or more sediment cores from within the sediment body. The final number of sediment cores will be determined based on field conditions, such as the size and shape of the sediment body, but a minimum of one core will be collected within each 0.1 river mile segment.

- **Detailed description of sediment type**

  - Sediment cores will be collected using a hand-driven coring device such as a split core sampler or a simple hand corer, or using a step probe. Sediment cores will be collected up to 2 ft below the sediment surface. If there is a clear interface between the stream sediment and the underlying geologic formation material, or if the grain size transitions to a medium or coarse-grain size within the sediment core, the depth of that transition will be recorded and will be used to indicate the thickness of the sediment body in that location.
  - Each sediment core will be logged using the Unified Soil Classification System (USCS) (ASTM D 2487). Color and other relevant physical characteristics will be described in addition to the USCS sediment type. The sediment details will be entered into the Mobile GIS application on the handheld GPS device for later correlation with the mapped outlines of sediment bodies. Physical observations that may help distinguish the biologically active zone will also be recorded (e.g., presence of benthic organisms, color changes, etc.).
  - A photograph will be taken of each sediment core.
  - After logging the core, sediment will be returned to approximately the same location from which it was collected and the coring device will be rinsed with stream water.
  - In addition to collection of sediment cores within the fine-grained sediment bodies, the stream bottom material in other areas will also be examined and described using the USCS method.

- **Confirmation Grain size analyses**

  - A confirmation sample will be collected for laboratory grain size analysis from each distinct type of fine-grained sediment material mapped within "sediment bodies." Up to five (5) of these samples will be selected to assess the range of grain size distribution encountered.

- **Mapping of obstructions within the stream**

  - Whenever an obstruction is encountered, such as a downed tree across the stream, a point will be entered into the mobile GIS application using the handheld GPS device and a general description will be recorded.

- **Identification of areas of high quality habitat**

Areas of high quality habitat will be identified based upon the following parameters:

  - General habitat type (riffle, run, pool, or glide).
  - Predominant substrate type(s) (e.g., boulders, cobbles, sand, etc.), including identification of whether there is extensive siltation/embeddedness.
  - Type(s) and extensiveness of instream cover (e.g., woody debris, overhanging vegetation, root mats, etc.).
  - Channel morphology, including sinuosity, presence of channelization, and stability.
  - Riparian zone quality (e.g., forest, swamp, urban, agricultural, etc.) and presence/absence of bank erosion.
  - Depth of pools, pool/glide width compared to riffle/run width, and current velocity.

In general, areas of highest-quality habitat will have a mixture of riffle/run and pool/glide areas with deep pool areas that are generally wider than riffle/run areas. In addition, they have predominantly larger substrate materials (boulders, cobbles, gravel, sand), little siltation, significant and diverse instream cover, high stream sinuosity, and a wide riparian zone of high quality (such as forest or swamp) with little bank erosion.

- **Discussions with potentially affected property owners**

  - Discussions with potentially affected property owners will be coordinated with USEPA and/or Ohio EPA and will focus on informing property owners regarding upcoming PDI work and associated access requirements.

- **Photographic Documentation:**

  - Photographic documentation will include a minimum of four photographs (one facing downstream, one facing upstream, and one facing each bank) for each 0.1 RM segment.
  - In addition to the photographs taken at each 0.1 RM segment, photographs will be taken of the following:
    - Sediment cores
    - Physical obstructions
    - High quality habitat areas
  - The location of each photograph will be entered into the Mobile GIS application using the handheld GPS device. Relevant details, such as the date and time, direction the photo is facing, and general description or comments about the photo will be recorded in the GIS application.

- **GPS Post-Processing**
  - GPS data collected using the handheld device will be post-processed using Trimble's GPS Analyst extension for ESRI's ArcGIS Desktop software. The post-processing uses known details about the position of satellites at the time data were collected to improve the spatial accuracy of the data. It is anticipated that the accuracy of all field data will be 1 ft or less after post-processing; however, interference from manmade and environmental conditions (e.g., clouds, buildings, or the tree canopy) could cause a reduction in the accuracy.

- **Reporting**
  - The results of the Site Reconnaissance will be used in determining the PDI sampling locations and they will be presented alongside the PDI sampling results in the Baseline Conditions Technical Memorandum.

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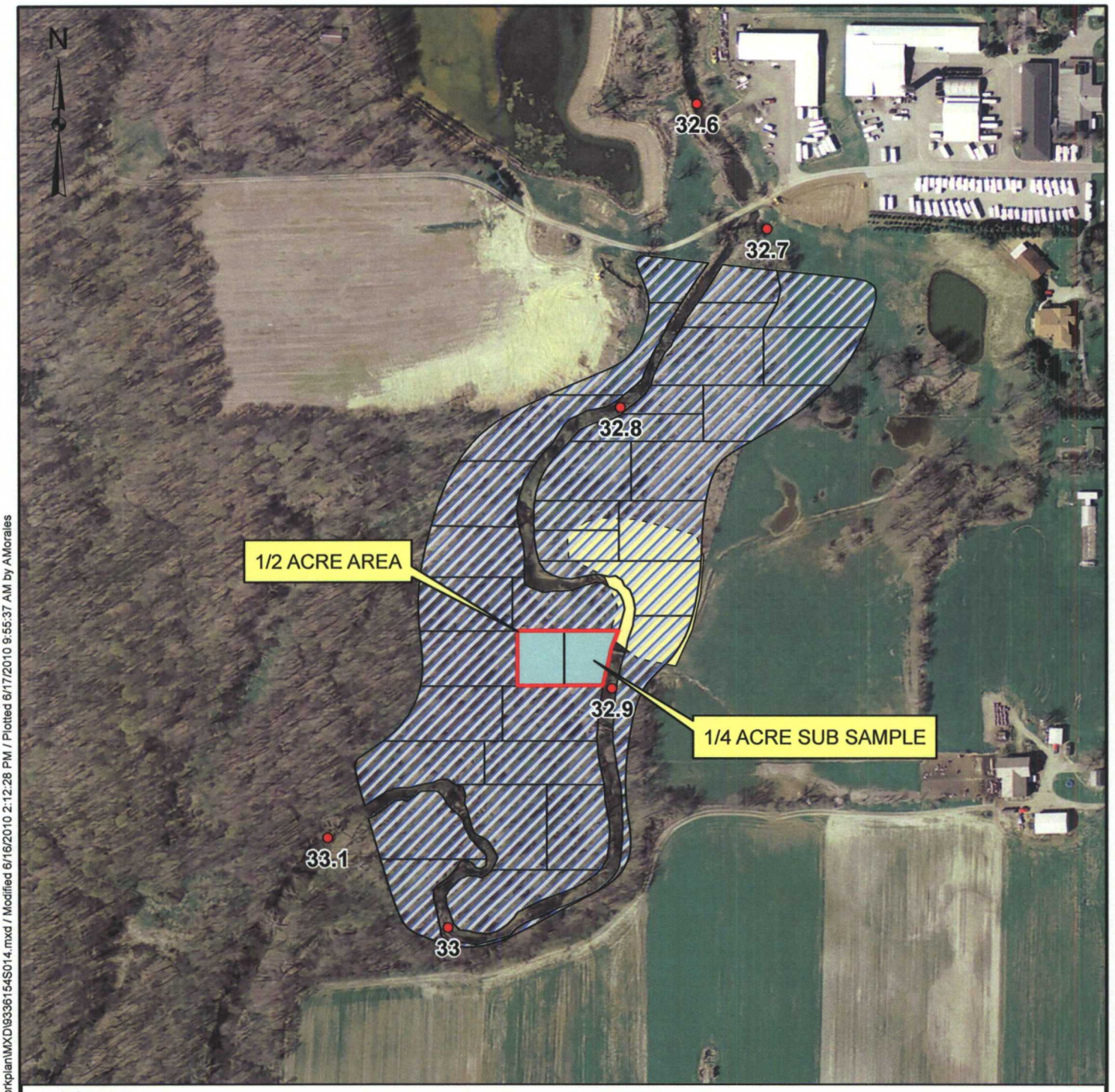
APPENDIX B

FLOODPLAIN SOIL SAMPLING APPROACH EXAMPLE FIGURE



LEGEND

- RIVER MILE
- 1/4 ACRE SUB SAMPLE AREA
- PROPOSED FLOODPLAIN SAMPLING AREA
- 2006 COMPOSITE FLOODPLAIN SOIL SAMPLE LOCATION

NOTES

1.) FLOODPLAIN SAMPLE LOCATIONS ARE APPROXIMATE AND ARE SUBJECT TO CHANGE BASED ON ACCESS AND FIELD CONDITIONS.

REFERENCES

1.) AERIAL FROM SPRING 2006 PROVIDED BY OHIO EPA.

300 150 0 300 Feet

Golder Associates
Philadelphia USA

SCALE	AS SHOWN
DATE	6/16/2010
DESIGN	APJ
GIS	AM
CHECK	APJ
REVIEW	PSF
FILE No.	9336154S014
PROJECT No.	933-6154
REV.	0

FLOODPLAIN SOIL SAMPLING APPROACH EXAMPLE

RÜTGERS ORGANICS CORPORATION

FIGURE B-1

Map Document: V:\Projects\1993\933-6154\REPORT_FILES\S_OU3_PDI_Workplan\MXD\9336154S014.mxd / Modified 6/16/2010 2:12:28 PM / Plotted 6/17/2010 9:55:37 AM by AMorales

APPENDIX C

OHIO EPA "INDICATOR" FISH SPECIES RECOMMENDATIONS MEMORANDUM

State of Ohio Environmental Protection Agency

Interoffice Memorandum

To: Mary Logan, U.S. EPA, Region 5

From: Sheila Abraham, DERR, NEDO

Date: January 22, 2008

Subject: NEASE SITE / OPERABLE UNIT 3 (MIDDLE FORK LITTLE BEAVER CREEK): "INDICATOR" FISH SPECIES RECOMMENDATIONS

At the Nease Site operable unit (OU) 3 meeting on September 27, 2006, Dr. John Estenik [Division of Surface Water (DSW)] suggested that rather than analyze all species collected in Middle Fork Little Beaver Creek (MFLBC) to track the long-term efficacy of sediment remediation, we focus on a few species as "indicator" species.

Ohio EPA recognizes that operation and maintenance (O&M) decisions normally occur later in the remedial process. However, given the nexus between the remedial process and other Ohio EPA programs, such as the fish consumption advisories, we believe it may be helpful to provide information to support long-term O&M earlier in the process.

Below is Ohio EPA's recommendation on the timing of the initial monitoring sampling, fish indicator species, and type of analysis, based on the existing biological sampling conducted to date on MFLBC and the Division of Emergency and Remedial Response (DERR) discussions with DSW (including Dave Altfater with the DSW Environmental Assessment Unit) and the Northeast District Office (NEDO) DSW.

This memo updates previous memos on this issue provided to U.S. EPA in October 2006 and October 2007.

1. Timing of the monitoring: The Agency recommends that the long-term monitoring to assess the efficacy of stream remediation activities commence at least 5 years *after* the completion of any MFLBC remediation. Based on past DSW remediation experience, 5 to 8 years is the optimum period for the impacts of remediation to translate into the fish (indicator) species. Also, we recommend that in keeping with Ohio EPA's standard surface water protocol, fish are sampled within the normal sampling window, from June 15 to October 15, unless site-specific circumstances warrant sampling outside this time window.

   The timing of future sampling(s) beyond the initial sampling should be flexible, and determined by the results of the initial sampling.

   The Agency also requests that DERR and DSW be informed of any MFLBC sampling events planned under U.S. EPA's oversight, such that sampling and analysis can be coordinated to support our Agency's programs to assess stream health.

2. Fish species recommended: We recommend analyzing at least 2 species from the 3 species listed below, in the following (hierarchical) order:

   1) common carp; 2) yellow bullhead; and 3) white sucker

   Based on the 2005 sampling, common carp and white sucker are fairly well distributed throughout the stream; yellow bullhead have been found at fewer sampling stations. All species and sizes caught at the different sampling stations during a single sampling effort should be retained until decisions have been made regarding analysis.

   Although literature exists that species' home range(s) may vary, per fishery biologists with DSW (Dave Altfater), these species do not move very much—generally less than 0.4 miles in their lifetime—in a stream habitat such as MFLBC. The data from the different sampling stations should, thus, help make conclusions regarding the pattern of mirex contamination in fish.

3. Fish size to be analyzed:

   3.1. Initial monitoring:
   Ohio EPA recommends that to assess post-remediation sediment mirex concentrations, fish tissue samples are collected no earlier than 5 years after construction completion, using fish that are 3 to 5 years old. To support this effort, below is information on age/length data provided by Dave Altfater (DSW) for the different species.

AGE	SPECIES (Length in mm)		
(Years)	COMMON CARP	YELLOW BULLHEAD	WHITE SUCKER
3	240	185	320
4	350	240	385
5	440	320	430

   3.2. Longer-term monitoring:

   Age/size fish classes analyzed for the long-term monitoring sampling effort will depend, in part, on the results of the initial sampling and remedial goals. Beyond the initial monitoring, Ohio EPA recommends that as a size cut-off, no fish less than the 25th percentile of the length should be analyzed. The Agency recognizes that any determinations on fish sizes to be analyzed will, however, be a function of sizes caught (i.e., if fish below the 25th percentile are the only ones captured, they should be analyzed).

   Also, to support any long-term effort, below are fish size data for the recommended species extracted by Dave Altfater from Ohio EPA's database, focusing on fish collected for tissue analysis in MFLBC, for the stretch of river from the former Nease facility downstream to the Lisbon Dam. Data are also provided on mean and median-sized fish; note that for MFLBC, the mean and median are very close based on the data.

Common Carp: 53 fish used for tissue analysis
25th %tile length = 300 mm (11.8 inches)
50th %tile length = 375 mm (14.8 inches)
Mean length = 373 cm (14.7 inches)

Yellow Bullhead: 13 fish used for tissue analysis
25th %tile length = 200 mm (7.9 inches)
50th %tile length = 209 mm (8.2 inches)
Mean length = 212 mm (8.3 inches)

White Sucker: 168 fish used for tissue analysis
25th %tile length = 260 mm (10.2 inches)
50th %tile length = 287 mm (11.3 inches)
Mean length = 285 mm (11.2 inches)

4. Type of fish tissue analysis: This depends on the remedial objectives and the information necessary to support them.

- If the intent of the long-term fish monitoring is solely to assess (human) fish consumption, then fish fillet data are adequate.

- However, if the remedial objective is to reduce mirex concentrations in whole body fish to acceptable levels, so as not to adversely impact populations of aquatic-prey-dependent higher trophic level species, then whole body information may be necessary.

Note that the remedial objectives should also specify the assessment area or ecological reach for the receptors of concern (i.e., the assessment area within which the remedial goals will have to be met).

Finally, the chemical analysis should focus on the Nease Site-related chemical of concern, mirex (and photomirex and kepone, as appropriate).

APPENDIX D
FIELD SAMPLING PROCEDURES

REPORT

FIELD SAMPLING PROCEDURES

PDI WORK PLAN

OPERABLE UNIT 3 NEASE SITE, OHIO

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March 2010

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1.0 DECONTAMINATION

Decontamination procedures listed below are intended for use by field personnel for cleaning all sampling equipment in the field. Deviations from these procedures should be documented in the field records and investigative reports. Specifications for standard decontamination materials follow. These materials will be used, as appropriate, for non-dedicated equipment used during sample collection (e.g., stainless steel bowls, spoon, knife).

- Detergent shall be a laboratory detergent such as Liquinox® or Alconox®. Use of other detergent must be documented in the field logbooks and investigative reports.
- Tap water may be used from any municipal water supply system.
- Distilled water is tap water that has been distilled. It is commercially available.
- Acetone shall be commercially available acetone.

The following steps will be taken to perform decontamination:

1. Initial rub/rinse with tap water.
2. Clean with tap water and detergent, using a brush to remove obvious particulate matter and surface films;
3. Rinse thoroughly with tap water;
4. Rinse thoroughly four times with distilled water;
5. Rinse thoroughly with acetone;
6. Allow equipment to completely air dry;
7. Wrap equipment in aluminum foil and/or placed in plastic bags.

All spent wash and rinse waters will be collected and treated on-Site or disposed off-site along with leachate that is routinely collected on-Site.

2.0 FIELD DOCUMENTATION

2.1 Field Notebooks

All information pertinent to the field investigation will be recorded in bound and numbered field notebooks and/or field information forms. All field notes must be legible. Any errors should be crossed out with a single line and initialed. Each sampling team will be assigned a notebook. Field records should at a minimum contain the following information:

- Date;
- Project or site name;
- Time of each data entry;
- Description of work being performed that day;
- Names and affiliations of personnel at location;
- Weather conditions on site;
- Location and type of activity;
- Visual observations;
- Pertinent field data (and any other measurements);
- Serial numbers, if any, on seals, and transportation cases, and equipment;
- Name of field custodian; and,
- Photographs taken, including date, time, direction faced, description of subject or activity, sequential number of the photo and film roll number will be recorded in the field notebook.

Specific sample information will be compiled into the field notebook and/or chain of custody form (COC). All field notebooks will be standard engineering hardbound books. All field notebooks will be photocopied so that copies of field notes can be kept in appropriate project files.

In some cases, a mobile GIS device, such as a Trimble GeoXH handheld unit, will be used to collect relevant field information.

2.2 Photographic Documentation

When photographs are taken, field personnel will record time, date, site location, general direction faced, sequential number of photograph and roll number (or image file name if digital camera is used), and brief description of the subject in a field notebook. This information will be transcribed onto the back of the photographs after they have been developed. When digital photographs are taken in place of traditional film photographs, the date and time of each photograph will be recorded in the field notebook with a description as above. The digital camera will be checked to ensure the correct date and time is recorded for each photo. The list of photos taken will be transcribed into an electronic file such as a Microsoft Word or Excel File that correlates the photograph file name with the description recorded in the field notebook. Digital photographs will also be printed and labeled similarly to film photographs and stored in the project files.

2.1 Changes in Procedure

Approval from the USEPA Remedial Project Manager will be obtained as needed prior to implementation of any major changes in sampling procedures as outlined in this work plan. Minor procedural changes may be made by Golder personnel, and if present, with the concurrence of the on-site USEPA and/or Ohio EPA representative. All changes will be documented in the field notebooks.

3.0 SAMPLE HANDLING, CUSTODY, AND SHIPMENT

3.1 Sample Handling

The laboratory will supply appropriately prepared sample containers. Preservation procedures and analytical holding times will be in accordance with the published analytical methods and USEPA Region V guidelines as further detailed below, except for archived samples for mirex analysis which must be analyzed within 200 days of collection.

3.2 Sample Preservation

Sample containers will be kept closed until the time they are to be filled. After filling, the containers will be securely closed, residue wiped from the sides of the containers, and immediately placed in a cooler. Samples shall be cooled to approximately 4°C immediately after collection. This temperature should be maintained for samples during storage and shipment to the laboratory. Samples will generally be shipped on the day of sample collection to the laboratory via overnight courier. Samples requiring chemical preservatives will be collected into pre-preserved containers obtained from the laboratory.

3.3 Sample Identification

All samples shall be adequately marked for identification from the time of collection and packaging through shipping and storage. Marking shall be on a sample label attached to the sample container. Sample identification shall include, as a minimum:

- Project name and/or code;
- Sample identification number;
- Analysis requested;
- Chemical preservatives;
- Sample date and time; and,
- Initials of the individual performing the sampling (samples for chemical analysis).

Each sample will be assigned a unique sample identification number to be recorded on the sample label. Each sample identification number will be recorded in the field notebook, a Sample Field Information Form and, as applicable, on chain of custody documentation. Designations for sample identification numbers for this project are described below.

Equipment

- Water proof marking pen;
- Sample bottle labels;
- Transparent tape; and,
- Site base map with designated sampling locations.

Procedure

Sample labels should be marked with the sample ID, the analytes to be tested, the initials of the sampler, and the date of sample collection. The labels should be affixed to the sample contaners and, if necessary, secured with tape. The corresponding sample ID should be marked in the project field notebook or in the mobile GIS device for correlation during report preparation. For soil samples, the location of the sample should be marked with the sample ID on a copy of the PDI workplan Figure 2 or 4 or in the mobile GIS device.

During drilling activities and sample collection, a system for assignment of sample identification (ID) numbers will be used for new sampling points to distinguish between different media and different quality control samples.

Sediment Discrete and Composite Area Naming Convention

Sediment sampling locations will be numbered according to the following convention:

SDAA-BB-CC

Where AA = the 2 digit year of installation (e.g. 2009 = 09, 2010 = 10)

BB = sequential number starting with 01 representing the area being sampled, either a given sediment body or a 0.1 RM segment (note that River Mile designations will not be used as some samples may cross more than one RM segment)

CC = sequential number starting with 01 representing the discrete grab sample location within the area being sampled (i.e., CC resets to 01 for each unique value of BB).

Floodplain Soil Sample and Composite Area Naming Convention

Floodplain soil samples will be named according to the following convention

SBAA-BB-DDD

Where AA = the 2 digit year of installation (e.g. 2009 = 09, 2010 = 10, etc.)

BB = Indicates the sequential number of the ½ acre area being sampled.

DDD = The sub-area label and the sequential number of the borehole. For each ½ acre area being sampled, there will be two ¼ acre sub areas labeled A and B.

Sediment and Soil Sample Naming Convention

Samples collected from sediment and soil cores will be identified according to the following protocol:

- For soil samples, the soil area ID (as assigned according to the above convention) will be appended with an underscore ("_"), followed by the sampled depth interval (in feet). The depth range will be reported as: EE-FF where EE is the depth (in feet) to the top of the interval and FF is the depth (in feet) to the bottom of the interval. If the sample is collected from a discrete depth, then EE = FF = the sampled depth. Depth measurements are reported in feet. Note that although EE and FF are used here, the values do not need to be 2 digits. For example, when a sample is collected from 0 to 6 inches, it will be labeled with 0 and 0.5.
- Field duplicate samples will be "blind" and will simply have a sequential ID beginning with SSAA-DBB (where "AA" is the 2-digit year and "BB" is the sequential number of the duplicate) for soil samples and SDAA-DBB for sediment samples.
- Samples that are chosen for MS/MSD will be collected with triple volume, but the ID will remain the same as the primary sample. The sampler will note on the COC that triple volume was collected for MS/MSD analysis.

Fish Tissue Sample Naming Convention

Fish tissue samples will be named according to the following convention

FTAA-BB.B-CC.C-DD-E

Where AA = the 2 digit year of collection (e.g. 2009 = 09, 2010 = 10)

BB.B = The downstream river mile where the fish collection began for that sample

CC.C = The upstream river mile where the fish collection ended for that sample.

DD = Fish species designation: Common Carp = CC; White Sucker = WS, and Yellow Bullhead = YB

E = The fish tissue sample type, F = fillet, O = offal

Below are examples of how the naming convention should be followed for new locations.

Description of location	ID assigned to location
First sediment area sampled in 2009	SD09-01
The first ½-acre horizontal soil composite area to be sampled in 2009	SS09-01
First sediment core location in the 3rd sediment area sampled in 2009.	SD09-03-01
The first surface soil sampling location within the first ¼ acre portion of soil sampling area SS09-01	SS09-01-A01

Below are examples of how samples collected from the above locations should be identified.

Description of sample	ID assigned to sample
A sediment sample collected from within sediment sample area SD09-01 from the 0 to 6 inch depth horizon	SD09-01-01_0-0.5
Soil sample collected from the 0 to 6 inch depth horizon from the first core location within the first ¼ acre portion of soil sampling area SS09-01	SS09-01-A01_0-0.5
Common carp fillet sample collected between RM 33.3 and RM 34.3	FT09-33.3-34.3-CC-F
The first sediment/soil field duplicate collected in 2009.	SD09-D01/SS09-D01

3.4 Sample Custody

Chain of Custody (COC) procedures have been established to ensure sample traceability from the time of collection through completion of analysis.

The National Enforcement Investigations Center (NEIC) of USEPA considers a sample to be in custody under the following conditions:

1. It is in your possession; or
2. It is in your view after being in your possession; or
3. It was in your possession and you locked it up; or
4. It is in a designated secure area.

Field Sample Custody

All environmental samples will be handled under strict chain of custody (COC) procedures beginning in the field. The following procedure should be used to maintain COC of environmental samples.

Equipment

The following equipment and materials are needed to perform COC procedures:

- Chain-of-custody forms;
- Chain-of-custody seals; and,
- A secure (locked) vehicle or building.

Procedures

Samples and sample containers must be kept under proper COC during field sampling. If custody of the samples (and sample bottles) is exchanged during field sampling, such transfer must be documented on the COC form. The departing field staff should sign indicating the custody has been relinquished, and the

arriving field staff should sign indicating responsibility for the custody of the samples. The COC form and Field notebook (and/or field information form) should include:

- Sample identification number and matrix;
- Project or site name or number;
- Sampler's name or initials;
- Sample collection date and time (military time);
- Designation as a grab or composite sample;
- Requested analysis;
- Any special comments; and,
- Any preservatives added to the sample.

When shipping samples to the laboratory, all sample bottles and requested analyses should be noted on the COC form. Where multiple analytical methods are available for a particular analysis, the specific method number should be listed on the COC form.

One member of the sampling team should sign the COC form relinquishing custody to the laboratory. If using an overnight courier service, record the tracking number on the COC. The COC form should be sealed inside the shipping container with the samples. The paperwork should be sealed inside a plastic bag to prevent damage from water condensation. The courier does not need to sign the COC form if it is sealed within the shipping container using custody seals. If the samples are hand delivered to the laboratory by field staff, the COC form should be signed at the laboratory when the samples are delivered, and the shipping container does not need to be sealed as long as it is kept under proper COC until delivered to the laboratory. If possible, COC seals should be signed and dated, and the serial numbers listed on the COC form. At least two seals should be used on each shipping container.

Laboratory Custody

Once samples are transported to the analytical laboratory, custodial responsibility is transferred to the Laboratory Sample Manager to assure that the procedures presented in the laboratory's Quality Assurance Plan (QAP) and the appropriate analytical methods are followed. The laboratory QAP will contain a detailed description of the laboratory chain-of-custody procedures, including receipt of samples, designation of a sample custodian, custody within the laboratory and laboratory storage, and disposal procedures. The laboratory chosen for this project will be furnished a copy of this work plan, which has been prepared in accordance with USEPA guidelines and specifications

3.5 Sample Packaging and Shipment

The following procedure is to be used to enhance successful shipping of samples to the laboratory.

Equipment

The following equipment and materials are required to perform this procedure:

- Overnight courier airbills and courier phone number (if applicable);
- Packing tape;
- Cushion material such as bubble wrap or vermiculite;
- Address labels; and,
- Laboratory address and phone number.

Procedure

Samples should be packed into a cooler in a manner that will minimize potential breakage of sample bottles. This may include use of laboratory-supplied bubble wrap designed to fit the particular bottle, polystyrene chips, or vermiculite. If wet ice is used for preservation, make sure that it is securely placed in zip locked bags and cannot leak. If the bottles sit in standing water, the sample integrity may be compromised. The sample containers must contain enough frozen blue ice packs or wet ice to maintain a temperature of 4°C during transport to the laboratory (fish samples will be stored and shipped on dry ice). A temperature blank should be placed in each cooler being returned to the laboratory. In general, samples should be shipped to the laboratory within 24 hours of collection.

4.0 QA/QC SAMPLING REQUIREMENTS

Quality Assurance/Quality Control (QA/QC) samples consisting of field duplicates and matrix spike/matrix spike duplicates (MS/MSD). These QA/QC samples are described briefly in the following sections. Note that Field duplicates and MS/MSD samples cannot be collected for fish tissue since these are not composited/homogenized in the field. The laboratory will prepare duplicate and spike samples as described in Section 5 of the main Work Plan text.

Field duplicates: one per 20 primary samples

MS/MSD: one per 20 primary samples

4.1 Field Duplicates

Field duplicates will be collected for all required analyses at a frequency of one per 20 primary samples per matrix. Field duplicates are collected by sampling the same location twice, but the field duplicate is assigned a unique sample identification number. Field duplicates should be collected from sampling locations with known or suspected contamination (i.e., source or near source area wells).

4.2 MS/MSD

Matrix Spike/Matrix Spike Duplicate (MS/MSD) samples will be collected for all required analyses at a frequency of one per 20 primary samples per matrix. Additional sample volume is collected from a location and submitted to the laboratory for analysis. The laboratory will use the additional volume to prepare spiked samples to assess accuracy and precision.

APPENDIX E

HEALTH AND SAFETY/CONTINGENCY PLAN

REPORT

HEALTH AND SAFETY/CONTINGENCY PLAN FOR THE OPERABLE UNIT 3 PRE-DESIGN INVESTIGATION

NEASE CHEMICAL COMPANY SITE, OHIO

Prepared For: Rütgers Organics Corporation

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August 2010

933-6154-004

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1.0 GENERAL INFORMATION AND SCOPE OF WORK

1.1 Acronyms and Definitions

As used in this Health and Safety/Contingency Plan (HASCP), the following terms have been defined:

ACGIH - American Conference of Governmental Industrial Hygienists

Authorized Personnel - Any person, such as task-specific personnel, project personnel, oversight personnel, contractor and consultants whose presence is authorized.

Breathing Zone - The worker's breathing zone is an imaginary glove of two foot radius surrounding the head.

Contamination-Reduction Zone - The area designated for removal of contaminants from personnel and equipment. This area is adjacent to the Exclusion Zone.

Contractor/Consultant - Any person or firm, retained or hired by the contractor, to carry out and/or supervise any portion of the activities conducted at the Site.

Exclusion Zone - The area in which all personnel entering must be directly involved in the ongoing work, have designated personal protective equipment (PPE), and meet training and medical monitoring requirements. An approximate 25-foot radius around the work area, which will be suitably marked, will define the Exclusion Zone.

HASCP - Health and Safety/Contingency Plan

HSO – Remedial Action Contractor's Health and Safety Officer

IDLH - Immediate Danger to Life and Health

MSDS - Material Safety Data Sheets. They provide information on the physical, chemical, and hazardous properties of chemical compounds.

NIOSH - National Institute of Occupational Safety and Health

On-site - Area designated as the property boundaries.

Off-site - Areas not designated within the property boundaries.

OSHA - Occupational Safety and Health Administration

Oversight Personnel - Any person, designated by the state, federal government, who is assigned to carry out oversight work.

PDI - Pre-Design Investigation

PEL - Permissible Exposure Limit (as established by OSHA)

PPE - Personal Protective Equipment

PPM - Parts per million; expressed as PPM(v) for gases and vapors.

Potential Source Area - The areas designated by the USEPA as areas of potential contamination and, if necessary, posted by signs with "Authorized Trained Personnel Only."

REL - Recommended Exposure Limit

SAMP - Sampling Analysis and Monitoring Plan (Part of Appendix S to this FDR)

SHSC - Site Health and Safety Coordinator (RI Contractor)

Site – The Nease Site, Salem, Ohio

Support Zone - The area outside the Exclusion Zone that is considered clean for the purpose of the HASCP. It is used for transfer of equipment and materials into the work site (i.e., support) and providing communications between the various zones.

TLV - Threshold Limit Value

1.2 Project Background

This Health and Safety/Contingency Plan (HASCP) has been prepared by Golder Associates Inc. (Golder Associates) for Rutgers Organics Corporation (ROC) in support of the Pre-Design Investigation for Operable Unit 3 of the Nease Chemical Site (Site) located in Columbiana and Mahoning Counties, Ohio. The HASCP is consistent with the requirements of the Statement of Work (SOW) portion of the Administrative Settlement Agreement and Order on Consent for Remedial Design (Settlement Agreement). This plan was prepared in accordance with the requirements established by the Occupational Safety and Health Administration (OSHA) standards 29 CFR §1910.120 and 29 CFR §1926. Other applicable guidance documents include "Guidance for Conducting Remedial Investigations and Feasibility Studies (RI/FS) under CERCLA," the NIOSH/OSHA/USCG/EPA "Occupational Safety and Health Guidance for Hazardous Waste Site Activities," and applicable Federal and state regulations and guidelines. This HASCP will be reviewed as necessary and amended, if appropriate, to ensure that the proper level(s) of protection and controls are used to manage the risk to on-site workers.

1.3 Site Description

The Site is located in northeastern Ohio in a rural area northwest of the City of Salem in northern Columbiana County. It is situated on the north side of State Route 14, and is west of Allen Road; Conrail railroad tracks traverse the property. The property is approximately 44 acres, is surrounded mostly by residences and farmland, and has an industrial plant to the east-northeast (Crane-Deming Pump Company). There is an inactive municipal landfill located upstream, approximately 1,200 feet east of the property along the west bank of the Middle Fork Little Beaver Creek (MFLBC). The property geology consist mostly of till and lake clays, with the primary bedrock units of the Middle Kittaning Sandstone (MKS) and Vanport Limestone/Putnam Hill Shale Zone, with Washingtonville Shale separating the two. The Site is a former chemical manufacturing facility that currently contains a shallow groundwater extraction and treatment system.

Certain areas adjacent to the Site will not be subjected to the requirements of 29 CFR §1910.120. These areas include the public rights-of-ways that surround the Site, and areas where access to private property is gained to collect residential well samples.

1.4 PDI Scope of Work

Various activities are planned as part of the PDI work for this Site. Fieldwork includes a Site reconnaissance consisting of detailed fine-grained sediment body mapping. This involves wading in the stream and collecting sediment cores with a stainless steel sediment coring device. Following the reconnaissance, sampling of environmental media in the MFLBC will be conducted, including collection of sediment cores, backpack electroshocking for fish collection, fish sample preparation (including filleting), floodplain soil sampling using stainless steel augers, corers, or trowels, and wetland delineation.

1.5 Project Safety Requirements

The level of protection and the procedures specified in this HASCP are based on previous information available and represent the minimum health and safety requirements to be observed by Golder personnel engaged in executing the PDI work. Should any situation arise which is obviously beyond the scope of the personal protection and decontamination procedures specified herein, work activities shall be immediately halted pending discussion with the PDI Health and Safety Officer (HSO) and PDI Project Manager (PM), and revision of the specified health and safety procedures. Any revision of the health and safety procedures will be recorded in the Field Procedure Change Authorization Form, shown in Attachment 1, and will require authorization from the HSO and the Project Manager.

All Golder personnel engaged in on-site activities will be required to read the Health and Safety Plan and sign the Safety Briefing Form (Attachment 2) before conducting any work on-site. Personnel who have any questions or concerns regarding implementation of this plan are encouraged to request clarification from the PDI HSO or Site Health and Safety Coordinator (SHSC). All personnel must follow the designated health and safety procedures, be alert to the hazards associated with working close to vehicles and equipment, and above all else, use common sense and exercise reasonable caution at all times.

1.5.1 Designated Safety Personnel and Chain of Command

Personnel responsible for the health and safety of Site employees on this project are the PDI SHSC, HSO, and Project Manager along with the Subcontractor SHSC, as designated below in order of authority:

1.	Andrew Joslyn	OU-3 PDI Site Health and Safety Coordinator (SHSC)
2.	James Valenti	Golder Associates Health and Safety Officer (HSO)
3.	Charles Lawrence	Golder Associates Project Manager (PM)

The SHSC has overall responsibility to implement health and safety procedures established herein including, if necessary, the authority to temporarily shut the project down for health and safety reasons.

The Project Manager has the overall responsibility for the project and will have the authority to take whatever actions may be necessary to provide a safe working environment for all project personnel.

The ultimate responsibility for the health and safety of the individual worker, however, rests with that worker, and his or her colleagues. Each worker is responsible for exercising the utmost care and good judgment in protecting his or her own health and safety and that of fellow workers. Should any worker observe a potentially unsafe condition or situation, it is the responsibility of that worker to immediately bring the observed condition to the attention of the appropriate health and safety personnel as designated above. The worker must then follow-up the verbal notification by completing the "Unsafe Conditions and Practices" report form provided in Attachment 3.

Should a worker find himself or herself in a potentially hazardous situation, the worker shall immediately discontinue the hazardous procedure(s) and either personally perform appropriate preventative or corrective measures, or immediately notify the SHSC or Project Manager of the nature of the hazard. In the event of an immediately dangerous or life-threatening situation, the worker shall stop work and immediately notify the appropriate safety and supervisory personnel.

"Extenuating circumstances" such as budget or time constraints, equipment breakdown, changing or unexpected conditions, etc., never justify unsafe work practices or procedures. In fact, the opposite is true. Under stressful circumstances all project personnel must be mindful of the potential to consciously or unconsciously compromise health and safety standards, and be especially safety conscious. All Site personnel are expected to consider "safety first" at all times.

1.5.2 Medical Surveillance and Training

All personnel engaged in on-site activities for this project must have baseline physical examinations and be participants in their employer's medical surveillance program. This program must meet, at a minimum, the requirements of 29 CFR §1910.120(f). Procedures beyond baseline physical and routine medical surveillance are not planned for the tasks listed in this document. The HSO will document that each worker has had a baseline physical prior to starting the project.

All on-site personnel, who have potential to contact contaminated soil, water, and/or air, must be trained in hazardous waste site investigation health and safety in accordance with 29 CFR §1910.120(e). This includes respiratory protection, personal protective clothing, field experience training, decontamination, and hazard recognition. Personnel must have OSHA 10-hr construction safety training (29 CFR §1926.21(b)(2)) and appropriate refresher courses as detailed in 29 CFR §1910.120(e) .

OSHA 29 CFR §1910.120 training requirements apply to all workers conducting on-site activities unless the employer can demonstrate that the operation does not involve employee exposure or the reasonable possibility for employee exposure to safety and health hazards. Some non-intrusive activities (e.g., supply delivery, limited surveying activities) may meet this exemption. The training requirements for non-intrusive activities will be reviewed on a case-by-case basis as Site conditions are subject to change. The Project Manager will make the determination on a case by case basis and will consult the HSO as necessary.

1.5.3 First Aid

A basic first aid kit shall be available in all field vehicles during all site activities. This kit shall be of an appropriate size in relation to the number of personnel on site and shall include at minimum two pairs of latex gloves, CPR barrier and eye wash solution in addition to typical first aid supplies.

1.5.4 Communications

The Site will have a mobile phone and will confirm coverage at the start of each work day. Note that mobile phones operating outside of their original territory may not contact the proper (i.e., local) emergency response authorities. Mobile phone users would be better served by dialing the full emergency response numbers (detailed in Attachment 4).

1.6 General Hygiene and Conduct Guidelines

The following general personal hygiene and work practice guidelines are intended to prevent injuries and adverse health effects. These guidelines represent the minimum standard procedures for reducing potential risks associated with various aspects of this project and are to be followed by all Site personnel at all times.

- A multi-purpose dry chemical fire extinguisher, a complete field first aid kit, and a bottle of emergency eye wash solution shall be maintained in every field vehicle. Additionally, Site trailers (if available on-site) will also be equipped with these emergency items, as well as an ANSI-approved eyewash capable of flushing the eyes for 15 minutes.
- Do not handle waste samples or any other potentially contaminated items unless wearing NBR (nitrile butadiene rubber) or neoprene rubber gloves, or equivalent, as a minimum. Employees should treat all excavated soil or wastes or groundwater as if it were contaminated. Always make an effort to approach any potentially contaminated feature/facility from upwind.
- Thoroughly wash hands and, if necessary face before eating or putting anything in your mouth, i.e., avoid hand to mouth contamination.
- Eating, drinking, chewing gum or tobacco and smoking are permitted only in areas designated by the SHSC. Under no circumstances will these activities be permitted in the immediate vicinity of any intrusive activities.
- Be alert to potentially changing exposure conditions, for example, as evidenced by perceptible odors or oily sheen on water.
- Do not, under any circumstances, enter or ride in or on any backhoe bucket, materials hoist, or any other similar device not specifically designed for carrying passengers.

- Be alert to the symptoms of fatigue and heat/cold stress and their affects on the normal caution and judgment of personnel (see Section 2.2).
- Noise may pose a health and safety hazard, particularly during drilling and construction activities. No drilling or construction activities are planned for this investigation, but work will be conducted near public areas where other unrelated work may produce excess noise. A good rule of thumb is if it is necessary to shout to communicate at a distance of three feet in continuous noise, hearing protection should be worn. Likewise, any impact noises from activities which is loud enough to cause discomfort, would also indicate the need for hearing protection. All employees with noise exposures in excess of 85 dB(A) on an 8-hour basis must wear hearing protection. Hearing protection is available and should be included in the standard field kit along with hard hat and safety glasses.
- Always use an appropriate level of personal protection. Reduced levels of protection can result in preventable exposure; excessive levels of safety equipment can impair efficiency and increase the potential for accidents to occur.
- Be aware of the effect of inclement weather (rain, snow, ice, lightning) on Site safety. Be prepared to suspend activities as conditions warrant.

1.7 Site Safety Meetings

The SHSC shall conduct a Site safety briefing for all personnel on their initial arrival at the Site. All personnel will be required to read the HASCP and sign the declaration (Attachment 2) before conducting any work on-site. The SHSC shall also conduct and document regular safety meetings at the start of each new task and in any event, no less frequently than weekly during field activities. The topics to be covered are determined by the task activities, and should include:

- Weather related safety issues.
- Hazards specific to the task(s) and protective equipment.
- Unusual site conditions/areas.
- Safety problems and issues.
- Changes in the HASCP.

2.0 HAZARD EVALUATION

Potential Site hazards include chemical hazards, physical hazards, and biological hazards. Each of these groups of potential hazards is addressed below.

2.1 Potential Chemical Hazards

Results of past sampling activities at the Site indicate that there has been chemical contamination of sediment and floodplain soil. Potential hazards include:

- Inadvertent ingestion of potentially toxic substances via hand to mouth contact or deliberate ingestion of materials inadvertently contaminated with potentially toxic materials. Included in this list is mirex, a chlorinated organic pesticide.
- Dermal exposure to mirex, a chlorinated organic pesticide.

Prohibition of smoking, eating, drinking and chewing in contaminated areas and good personal hygiene habits can effectively control exposure via the ingestion route. Similarly, good personal hygiene and appropriate clothing can eliminate dermal exposure. Inhalation hazards are addressed in Section 3.2 below.

2.2 Potential Physical Hazards

2.2.1 Heat Stress

Working in protective clothing can greatly increase the likelihood of developing heat stress. This can result in health effects ranging from transient heat fatigue to serious illness or death. The signs and symptoms of heat stress are presented in Section 2.4. Workers shall monitor themselves and others for signs of heat stress when ambient temperatures exceed 80 degrees Fahrenheit (70 degrees when wearing Tyvek coveralls). Currently, the HASCP indicates the use of Level D PPE for all activities. No changes to the specified levels of protection shall be made without the knowledge and approval of the HSO and PM. Golder Standard Work Procedures for preventing heat stress are included in Attachment 5.

2.2.2 Cold Stress

Personnel exposed to cold temperatures (especially during windy conditions) may be subjected to cold stress in the form of frost nip, frost bite or hypothermia. The signs and symptoms of cold stress are presented in Section 2.4. Workers shall monitor themselves and others for signs of frost nip when cold weather occurs. Extra caution will be exercised when working in windy conditions and/or when clothing becomes wet (see Section 2.4). Golder Standard Work Procedures for preventing cold stress are included in Attachment 5.

2.2.3 Excavation/Trenching

Excavation or trenching could result in physical/chemical hazards. Site personnel must be alert to hazards associated with excavations/trenches (e.g. unstable slopes and weather conditions which can increase the chances of cave-in). Excavation/trenching will not be required during the PDI activities proposed for the Site and at no times shall any person enter an excavation.

2.2.4 Confined Space Hazards

Entrance into confined spaces can result in chemical exposures. Site personnel must be alert to hazards associated with confined spaces (e.g. explosive atmosphere, low oxygen levels, and physical hazards). Personnel entry into confined spaces is not currently anticipated. Should site conditions require such an entry, entry will be performed in accordance with OSHA regulations and confined space entry procedures. The HSO and PM must be notified prior to any changes on the Statement of Work that would require confined space entry. Additional safety equipment that may be necessary for entry will be identified by the HSO and PM.

2.2.5 Electric Hazards

Since electrical equipment will be used during the PDI, there is a potential for electrical hazard. The electrical hazard control measures will include, but not be limited to, the following:

- When digging, make the one-call to locate utility lines;
- Make sure all electrical equipment is properly grounded;
- Use qualified electricians to perform all electrical work;
- Inspect all extension cords daily for damage and ground continuity;
- Cover or elevate electric wire in work areas to protect from damage;
- Keep all plugs and receptacles away from water; and,
- Use water and weatherproof supplies when available.

There are power lines located within the work area that could potentially interfere with the PDI activities. All equipment should be operated a minimum distance of 20 feet from power lines. Always assume all power lines are "live".

Additional electric hazards are posed by the use of a backpack electroshocking unit. Procedures for working with this equipment are included in Attachment 6.

2.2.6 Railway Safety

All personnel shall be aware of the dangers of working near railways. Trains may approach at high speeds without notice and pose a significant safety concern. Personnel shall always assume the rail line is electrified and stay a minimum of 50 feet off the track. No work activities shall occur within 50 feet of the rail lines. Additionally, be alert to stationary rail cars and equipment as these may move without notice. Personnel shall not stand or walk on the railroad tracks. Personnel shall not cross tracks except

at designated crossing areas. If work is scheduled for areas within 50 feet of the rail line, qualified flagpersons must be used. No work is anticipated to occur within 50 feet of the rail line.

2.2.7 Other Physical Hazards

All Site workers must take note of physical hazards that are identified during site safety briefings. These hazards may include, but are not limited to steep slopes, soft sediments, and muck (trips, falls); sharp debris (puncture wound), public traffic, and slippery and/or congested walking surfaces (falls). If work near a public right-of-way is required, consult with the local police department and county highway departments and provide necessary traffic cones or barriers to insure a safe work area. High-visibility vests will be required when working near public rights-of-way.

Field personnel must be alert to the hazards associated with Site vehicles, sharp tools (e.g., fillet knives for fish sampling and spring-retractable utility knives for sediment and floodplain soil sampling) heavy equipment, and powered hand-held equipment operations (such as the backpack electroshocker). These hazards include cuts, noise, crushing injuries, overhead hazards, electrical shock, and pinch points. Personnel must be alert to weather-related hazards (e.g., lightning) or the possibility of increased hazard due to weather (e.g., slipping on mud or ice).

Some field work will be conducted in and/or in close proximity to the Middle Fork Little Beaver Creek. This field work will be performed by field personnel by wading to collect relatively undisturbed sediment samples and perform sediment thickness measurements and to collect fish samples. For all field work performed on or near water, where the danger of drowning exists, OSHA standard 29 CFR 1926.106, entitled "Working Over or Near Water", applies. All field personnel and subcontractors are required to comply with the requirements of this regulation.

The regulation states:

- Employees working over or near water, where the danger of drowning exists, shall be provided with U.S. Coast Guard-approved life jacket or buoyant work vests.
- Prior to and after each use, the buoyant work vests or life preservers shall be inspected for defects which would alter their strength or buoyancy. Defective units shall not be used.
- Ring buoys with at least 90 feet of line shall be provided and readily available for emergency rescue operations. Distance between ring buoys shall not exceed 200 feet.
- At least one lifesaving skiff shall be immediately available at locations where employees are working over or adjacent to water.

Since the field work will be conducted in shallow water (3-4 feet), a lifesaving skiff will not be necessary. It will be the responsibility of the field team leader to ensure that this regulation is followed. Golder Standard Work Procedures for working on or over water are included in Attachment 5.

2.3 Potential Biological Hazards

Contact with contaminated soil or debris can lead to infected cuts. Personnel shall follow the guidelines in Section 1.5 and follow first aid procedures for disinfecting cuts and abrasions.

The Site area may contain ticks that can transmit Rocky Mountain Spotted Fever and Lyme Disease. During tick season (typically March to November), Site employees will check for ticks. Light colored clothing should be worn and any openings (shirt and pant cuffs) should be secured to inhibit tick movement from clothing to skin. The use of insect repellents will be permitted if its use will not interfere with sampling and/or other PDI activities. Field personnel will acquaint themselves with the symptoms of tick-borne diseases detailed in Section 2.4 and will contact a physician as well as the HSO if a disease is suspected.

The Site may also harbor potentially harmful mammals such as rodents. Personnel must be alert to these animals, particularly around holes in the ground.

The Site contains poison ivy, which can be recognized by an oily sheen on the leaf and/or three leaflets together or similar vegetation, or as vines, especially in wooded areas. It can be found at the ground surface and as vines climbing large trees. The active substances can be transmitted by direct skin contact and via contact with contaminated clothing.

Golder Standard Work Procedures for addressing biological exposure hazards are included in Attachment 5.

2.4 Signs and Symptoms of Exposure

2.4.1 Chemical Exposure

Health effects associated with the chemical contaminants at the Site are varied. Personnel who experience any of the following symptoms should report the occurrence to the SHSC promptly:

- skin, eye, or respiratory system irritations;
- skin rashes/burns;
- headaches, dizziness;
- nausea/GI tract problems;
- muscle weakness;
- convulsions; and/or
- fatigue.

Note that the above symptoms are not necessarily caused by chemical exposure. Any serious medical problem should be referred to professional medical care. If personnel experience any of the above symptoms, the SHSC will evacuate the area (upwind, if possible), if necessary, and evaluate affected

personnel for signs and symptoms of exposure. Appropriate first aid measures shall be taken. The activity will not resume until the atmospheric conditions are evaluated using monitoring instruments by personnel wearing Level C Personal Protective Equipment. Atmospheric conditions will be evaluated by monitoring for concentrations of combustible gases, VOCs, and dust as described in Section 3.0. Under no circumstances will workers upgrade or downgrade to Level C or B protection without approval from the HSO. At most, only Level C protection is anticipated. Level B (and A) protection will not be required at the Site. Table 1 summarizes the site-specific exposure hazards and the exposure/first aid information in the event of exposure.

2.4.2 Physical Exposure

The signs of heat stress are as follows:

- Heat rash may result from continuous exposure to heat or humid air.
- Heat cramps caused by heavy sweating with inadequate electrolyte replacement. Signs and symptoms include:
  - muscle spasms; and
  - pain in hands, feet, and abdomen.

Heat exhaustion is caused by increased stress on various body organs including inadequate blood circulation due to cardiovascular insufficiency or dehydration. Signs and symptoms include:

- pale, cool, moist skin;
- heavy sweating;
- dizziness;
- nausea; and
- fainting.

Heat stroke is the most serious form of heat stress. Temperature regulation fails and the body temperature rises to critical levels. Immediate action must be taken to cool the body before serious injury and death occurs. Competent medical help must be obtained. Signs and symptoms are:

- red, hot, usually dry skin;
- lack of or reduced perspiration;
- nausea;
- dizziness and confusion;
- strong, rapid pulse; and
- coma.

Working in protective clothing can greatly increase the likelihood of heat fatigue, heat exhaustion, and heat stroke, the latter being a life threatening condition. When working in ambient temperatures greater than 80°F (70°F when in Level D-2 or Level D-3 protective clothing, as described in Section 4.3), workers shall use the 'buddy system' to monitor each other's pulse rate at the start of each rest period. A rest period shall consist of a continuous time period of at least 5 minutes preferably in a shaded area. The personnel will not be assigned to other work during this rest period. If the pulse rate exceeds 110 beats per minute, the worker shall take his or her oral temperature with a clean disposable calorimetric oral thermometer. If the oral temperature exceeds 99.6°F, the next work period shall be shortened by one third. The pulse rate and oral temperature shall be monitored again at the beginning of the next rest period; and if the oral temperature exceeds 99.6°F, the work period shall again be shortened by one third, etc., until the oral temperature is below 99.6°F.

All personnel are to be alert to the possibility and symptoms of heat stress. If heat stress is suspected the worker is to leave the work area, rest, cool off, and drink plenty of cool water/Gatorade/Squelcher or equivalent. Sufficient cool potable water and clean disposable cups shall be maintained at all times in the Support Area. If the symptoms do not subside after a reasonable rest period, the worker shall notify the SHSC and seek medical assistance.

Signs of cold stress include yellow or white patches of skin on the fingertips, nose and ears. These areas will be numb. The affected parts will be warmed gently and the worker will not return to work until additional protection (e.g., gloves, hard hat liner) is obtained. It is essential to prevent frostbite, as the person may become susceptible to future cold-related medical problems. Personnel are encouraged to change into dry socks after the lunch break as perspiration held by socks prompts cooling of the feet. Should clothing become wet, it is imperative that the person changes into dry clothes before resuming work. Wet clothing can lead to hypothermia. Symptoms of hypothermia include uncontrollable shivering, decreased physical and mental capabilities, and lowered body temperature. Personnel exhibiting symptoms of cold stress or hypothermia will not return to work without the approval of the SHSC.

2.4.3 Biological Exposure

Symptoms of Rocky Mountain Spotted Fever include fever chills, headache, abdominal, muscle pain, and nausea. A red rash develops at the wrist and ankles two to five days after exposure. Symptoms develop two to fourteen days after exposure.

Symptoms of Lyme Disease include fatigue, stiffness (particularly in the neck). There may be a red circular rash. Fever may be present. Symptoms develop a few days to two years after exposure. Personnel exhibiting symptoms of Rocky Mountain Spotted Fever or Lyme Disease should consult a medical professional immediately.

Skin-sensitizing (poisonous) vegetation produces a bumpy, swollen rash at the point of contact. This rash is easily spread if the oil gets on the fingers. Wash affected area(s) including tools as soon as possible. Use over-the-counter medications to reduce the irritation. Avoid scratching the rash. Cover the affected area(s) with clean dressings. Severe exposure may necessitate evaluation by a medical professional.

Personnel scratched and/or bitten by mammals will immediately clean the wound(s) and proceed to the hospital for medical evaluation.

2.5 Task Risk Analysis

The comparative risk analysis has been prepared (Table 2) based on anticipated field activities and hazards. All personnel, however, should be aware that specific hazards and the associated potential severity may be influenced by weather, season, and fatigue.

3.0 SITE MONITORING AND ACTION LEVELS

Routine air monitoring is not required during non-intrusive activities (e.g., sampling). Field team personnel, however, should be aware of any conditions that would indicate potential airborne chemical exposure (e.g., visible plume or smoke, etc.). If such indicators are encountered, discontinue work, evacuate the area and contact the relevant emergency service providers and HSO.

3.1 Nuisance Dust and Mirex

Nuisance dust (which may contain mirex) has the potential for becoming a problem during disruptive or intrusive activities such as drilling. The scope of work for this investigation does not include intrusive activities, so dust is not expected to occur. However, the work area will be periodically monitored by the SHSC using visual means to determine if visible dust is occurring. If Site activities generate dust, engineering controls (e.g., water misting of the air and surrounding soil) will be applied before and during the activities. If dusty conditions persist, work will be temporarily stopped to monitor the dust using a Mini-RAM dust/aerosol monitor. The level of protection will be reassessed based on the dust monitoring criteria presented in Table 4. This reassessment will determine if additional personal protection and/or dust suppression is required.

Mirex does not have published exposure limits for inhalation. A skin rash or burning feeling, irritation to eyes, nose and throat, nausea, vomiting, headache, dizziness, muscle weakness, fatigue, or convulsions may indicate an exposure to Mirex. If a Site worker is suspected of being exposed to Mirex, remove the worker from the exposure area and transfer the worker to one of the appropriate medical facilities listed in Attachment 7.

4.0 ON-SITE CONTROL

4.1 Site Communication System

Personnel will perform work activities using the "buddy system." Each individual shall maintain visual and audible contact with another individual or group at all times.

4.2 Site Safety Zone and Access Control

On-Site safety zones are not required for non-intrusive activities. During electofishing, bystanders must maintain a safe distance from electrofishing activities. This is a minimum of 30 feet if they are in the water and at least 10 feet if they are on land. Electrofishing should stop if bystanders are too close or will not move.

4.3 Personal Protective Clothing and Respiratory Protection

The following scheme will be used to designate the required level(s) of personal protective equipment and respiratory protection: the alphabetical designations "B," "C," and "D" shall refer specifically to levels of respiratory protection, namely pressure-demand air supplying respirators with escape provisions, air purifying respirators, and no respiratory protection, respectively. Since potential dermal exposure hazards may require a wide variety of personal protective clothing without regard to the required level of respiratory protection, the numerical designations "1," "2," and "3" will be used to specify the level of protective clothing that is to be employed in addition to the designated level of respiratory protection as described below (i.e., the level of protective equipment can be completely defined by a designation of "C-2," "B-1," etc.).

The initial level of personal protective clothing that will be required during most Site activities would be D-1, which consists of the following:

LEVEL D-1 PROTECTIVE CLOTHING

- Standard work clothes;
- Waterproof, leak-free chest waders with steel-toe inserts (when in water);
- Waterproof, leak-free neoprene or heavy duty rubber gloves (when electrofishing)
- Steel toed boots (when on land);
- Safety glasses;
- Personal Flotation Device (PFD) (used when risk of drowning is present);
- Orange safety vests (when working near public traffic and heavy equipment);
- Hard hats (when an overhead hazard is possible) and;
- Hearing protection (if excessive noise is encountered).

Upgraded protective clothing shall consist of the following:

LEVEL D-2 MODIFIED PROTECTIVE CLOTHING

- Level 1 protective clothing;
- Liner cut-resistant gloves; and
- Outer NBR gloves.

LEVEL C PROTECTIVE CLOTHING AND EQUIPMENT

- Air-purifying respirator, full-face, cartridge-equipped (NIOSH approved);
- Chemical-resistant clothing (coveralls; hooded, one-piece or two-piece chemical splash suit; chemical-resistant hood and apron; disposable chemical-resistant coveralls);
- Coveralls;
- Long cotton underwear;
- Inner latex gloves;
- Outer NBR gloves;
- Boots (outer), chemical-resistant, steel toe and shank;
- Boot covers (outer), chemical-resistant (disposable);
- Hard hat (face shield);
- Escape mask; and
- 2-way radio communications (intrinsically safe).

LEVEL B PROTECTIVE CLOTHING AND EQUIPMENT

- Supplied-air respirator (NIOSH approved). Respirators may be positive pressure-demand, self-contained breathing apparatus (SCBA), or positive pressure-demand, airline respirator (with escape bottle for IDLH or potential for IDLH atmosphere);
- Chemical-resistant clothing (overalls and long-sleeved; hooded, one or two-piece chemical-splash suit; disposable chemical-resistant, one-piece suits);
- Long cotton underwear
- Coveralls;
- Inner latex gloves;
- Outer NBR gloves;
- Boots (outer), chemical-resistant, steel toe and shank;
- Boot covers (outer), chemical-resistant (disposable);
- Hard hat (face shield); and
- 2-way radio communications (intrinsically safe).

Tyvek or polycoated Tyvek will be worn where it is probable that there will be more than casual, inadvertent contact with subsurface soils, groundwater and/or surface water. Tyvek or polycoated Tyvek can also be worn when working in muddy conditions.

OSHA standard 29 CFR §1926.106 requires personal floatation devices (PFDs) be worn where there is a possibility of personnel falling into the water. If a task requires working near the water, affected personnel will don the required PFD. If working in the proximity of water, but not near it, the area near the water will be designated "off limits" to personnel.

If conditions are found which are beyond the required Level(s) of Protection, personnel are to leave the area immediately and notify the HSO before upgrading to Level C or above. If an inhalation hazard is suspected (e.g. unusual and continuous odors, dizziness, or respiratory irritation), personnel will immediately move upwind from the area. Such areas shall be noted on a map and the HSO will be notified. The SHSC will notify Site workers. Work will not proceed in these areas until the nature of the hazard has been assessed by air monitoring and additional protective measures are employed to the satisfaction of the SHSC, HSO and/or PM. Re-entry will be from an upwind position (when possible). Monitoring will precede re-entry. Personnel who experienced symptoms will not re-enter the area until symptoms have subsided or proper medical attention and clearance has been given, and additional equipment/precautions are employed as determined by the monitoring.

4.4 Decontamination

The HSO and/or the SHSC shall determine when decontamination is necessary, except in the case of emergencies. The nature of chemical contamination on this project is <u>not</u> anticipated to present an immediate threat to human health. In an emergency, other than removal of outer protective garments and gross contamination (e.g., mud), immediate emergency treatment of injuries should generally take precedence over personal decontamination.

Decontamination will involve two phases. Most will consist of gross decontamination of personnel and equipment, comprising removal of mud by dry brushing or scraping. All soil removed in this way will be backfilled into the borehole where possible. All personnel and equipment will undergo gross decontamination prior to moving to a new investigation location on the Site. Prior to leaving the Site, personnel and equipment will undergo full decontamination at the central decontamination pad if deemed necessary.

<u>Decontamination Procedures</u>

If necessary, full decontamination procedures will be as follows for Level D:

- Protective outer PPE shall be disposed of in plastic bags immediately after leaving the exclusion zone;
- Field personnel will then proceed to the job trailer to wash and dry their hands and face (when necessary);
- The plastic bags containing waste materials will be bagged and disposed in the exclusion zone.

Monitoring equipment and hand tools shall be retrieved and decontaminated using methods appropriate for the type of equipment. Containing equipment in plastic (as applicable) prior to Site entry will expedite decontamination. The SHSC may inspect the equipment for cleanliness.

Certain sampling equipment (e.g., hand sampling tools) may require specific decontamination procedures and/or chemicals. All chemicals brought to the Site will have the appropriate Material Safety Data Sheet(s) (MSDS).

5.0 CONTINGENCY AND EMERGENCY RESPONSE PLANS

If an unanticipated, potentially hazardous situation arises as indicated by visible contamination, unusual or excessive odors, Site workers shall temporarily cease operations, move away to a safe upwind area, and contact the SHSC. The following procedures have been established to deal with emergency situations that might occur during Site activities.

Prior to starting work at the Site and at the discretion of the PM, the local emergency response services will be contacted and informed that field activities will be in progress. If requested by the local emergency services, a practice exercise will be held with them prior to the start of intrusive activities. Site personnel will familiarize themselves with the location of the nearest phones and medical facilities on arrival at the Site. In the event of a serious emergency situation (e.g., medical problems beyond routine first aid and fire beyond incipient stage), Site personnel shall contact the Salem Police and Fire Departments, inform them of the nature of the emergency, and then notify the PDI SHSC. When help arrives, Site personnel shall defer all emergency response authority to appropriate responding agency personnel. Emergency notification information is summarized in Attachment 4 of this HASCP.

In the event of an imminent or actual emergency situation, the SHSC will perform the following tasks:

1. Notify on-site personnel;
2. Identify the problem and assess the environmental and health hazards;
3. Call Emergency Response personnel (fire, police, EMS) for backup (see Attachment 4);
4. Take all reasonable measures to stabilize the situation, including evacuation of the Site, if necessary;
5. Document the situation and actions taken;
6. Continue to monitor the situation after stabilization; and,
7. Contact the PM and HSO, who will contact the Group's Project Coordinator and State and Federal environmental regulatory agencies.

During an emergency, the SHSC will take reasonable measures necessary to ensure that fire, explosion, emission, or discharge do not occur, reoccur, or spread. These measures will include terminating activities or operations, evacuating Site personnel, and ensuring that all access roads are kept free of obstructions during an emergency situation.

After an emergency, the SHSC will consult with the PM to determine what treatment or disposal of contaminated soils or waste is appropriate. He will ensure that all necessary cleanup procedures are completed. Reporting will be as discussed in Section 5.8.

In the event of an emergency, the individual who observes the event will immediately (within 15 minutes or less) report the condition to the SHSC, who will take the appropriate action as described above. If the SHSC is unavailable, his/her backup, or the senior individual on-site will act as the SHSC's designee until the SHSC becomes available. A maintenance, operations, or design failure that does not result in personal injury or imminent harm is not considered to be an emergency situation.

External communications will be accomplished via outside telephone lines or radio communications. Direct telephone communication or radio will be used to alert outside agencies, such as the fire and police departments, in the event of an emergency. The Group's Project Coordinator or OWNER's REPRESENTATIVE, not the SHSC, shall notify the USEPA, and Ohio Environmental Protection Agency (Ohio EPA) if appropriate, of an on-site emergency situation.

5.1 Emergency Services

If response to an on-site incident requires assistance from outside emergency support agencies (police, fire department, medical emergency response teams), the SHSC will contact the appropriate agency. He/she has immediate and full authority to engage the services required. The SHSC will then relinquish control of the situation to the appropriate emergency official upon the official's arrival on-site, but continue to provide advice.

A list of contacts and emergency services is provided in Attachment 4 in case an emergency situation arises. This list and a map showing the locations of the Site and hospital will be posted in conspicuous locations on-site.

The SHSC will give the following information to the emergency agency:

1. Name and telephone of person reporting the incident;
2. Name and address of the site;
3. Time and type of incident;
4. Name, nature, and quantity of material(s) involved, to the extent known;
5. The extent of injuries, if any; and
6. Identified hazards to human health and/or the environment.

An Emergency Notification Contact List and an Incident Report Form are provided as Attachments 4 and 9, respectively.

5.2 Evacuation Plan

It is not anticipated that full evacuation of the Site or adjacent properties will be necessary. In general, evacuation will be limited to the immediate area surrounding the emergency situation. If the SHSC determines that Site evacuation is necessary, the following evacuation procedures will be implemented:

1. Notify all on-site personnel;
2. Personnel will secure equipment and materials and place them in secure areas;
3. Personnel will evacuate via site access roads;
4. Personnel will reassemble at a location predetermined by the PDI SHSC, away from the incident to verify all personnel have evacuated the area of concern;
5. PDI SHSC will account for all personnel;
6. PDI SHSC will notify the appropriate emergency response agency and others as shown in Attachment 4; and
7. Personnel will not re-enter the area until it is determined to be safe by the SHSC or the appropriate emergency official in charge.

5.3 Medical Emergency Response Plan

Should any person on the Site be injured or become ill, initiate the following emergency response plan and notify the SHSC as soon as possible:

- If able, the injured person should proceed to the nearest available source of first aid. If the injured party is extremely muddy, remove outer garments and if necessary, wash the injured area with soap and water. If the "injury" involves a potential overexposure to hazardous gases or vapors, (headache, dizziness, nausea, disorientation), get the victim to fresh air and take him or her to the nearest hospital as soon as possible.
- If the injury involves foreign material in the eyes, immediately flush the eyes with emergency eye wash solution and/or rinse with copious amounts of potable water. Obtain or administer first aid as required. If further medical treatment is required, seek professional medical assistance as discussed below. Appropriate measures should be taken to protect the privacy of workers in connection with putting on and taking off of protective clothing.
- If the victim is unconscious or unable to move, or if there is any evidence of spinal injury, do not move the injured person unless absolutely necessary to save his or her life, until the nature of the injury has been determined. A CPR trained individual should administer rescue breathing if the victim is not breathing, control severe bleeding and immediately seek medical assistance as discussed below.
- If further medical treatment is required and
  - the injury is not severe, contact the nearest hospital (see Attachment 4) and take the injured party to the hospital by private automobile (see Attachment 7 for directions).
  - the injury is severe, immediately call the Salem Police Department at (330) 337-7811or 911 (by landline only). If using a cellular phone, remember to provide specific location information to the dispatcher.

In both cases, if decontamination is not undertaken, appropriate precautions should be taken to avoid transfer of contaminants to vehicles and other facilities. This can be done by using plastic sheeting or the exposure blanket (if available) contained in the first aid kit.

- An individual designated by the SHSC to ensure prompt and proper medical attention shall accompany any injured person taken to the hospital. After proper medical treatment has been obtained, the designated companion should notify the HSO and prepare a written report. Site personnel shall maintain their medical insurance identification at the Site whenever they are on Site.

  In the event that any personnel are injured at the Site during any phase of the Remedial Action, all available technical information and supporting documentation shall be provided to any treating physicians, or treating health care workers or facilities.

5.4 Fire and Explosions

Dry chemical fire extinguishers are effective for fires involving ordinary combustibles such as wood, grass, flammable liquids, and electrical equipment. They are appropriate for small, localized fires such as a drum of burning refuse, a small burning gasoline spill, a vehicle engine fire, etc. No attempt should be made to use these extinguishers for well established fires or large areas or volumes of flammable liquids. In this event, the work area should be evacuated immediately and the fire department called.

Catalytic converters on the underside of vehicles are sufficiently hot to ignite dry grass. It should be noted that the Site is currently heavily vegetated and is therefore susceptible to brush. Personnel should avoid driving over dry grass that is higher than the ground clearance of the vehicle, and be aware of the potential fire hazard posed by the catalytic converter, at all times. Never allow a running vehicle to sit in a stationary position over dry grass or other combustible materials.

In the case of fire, prevention is the best contingency plan. There will be no smoking on Site except in pre-designated areas. In the event of a fire personnel shall attempt to extinguish the fire with on-site fire extinguishers. If a fire cannot be controlled in this manner, personnel shall evacuate and notify the SHSC.

In the event of a fire or explosion:

- If the situation can be readily controlled with available resources without jeopardizing the health and safety of Site workers, take immediate action to do so. If not:
- Isolate the fire to prevent spreading, if possible.
- Clear the area of all personnel working in the immediate vicinity.
- Immediately notify site emergency personnel and the Salem Fire Department, (330) 337-6183 or 911 (by landline only).

5.5 Chemical Exposure First Aid

In an event of exposure to chemicals through inhalation:

- Move the victim to an up-wind location for fresh air.
- Signal for help.
- Initiate CPR to revive the victim, if necessary (CPR-trained personnel only).
- Contact the Salem Police Department ((330) 337-7811 or 911), if necessary.

For exposure through dermal route (including eyes):

- Wash the affected area with copious fluids for at least fifteen (15) minutes (Signal for help if necessary).
- If irritation persists, seek professional medical care.

For ingestion:

- Consult Table 1 to see which actions should be taken initially.
- Transport the victim to the hospital. Take a copy of this HASCP to the hospital.

If decontamination is not undertaken prior to transporting the victim to the hospital, appropriate precautions should be taken to avoid transfer of contaminants to vehicles and other facilities.

5.6 Unforeseen Circumstances

The health and safety procedures specified in this plan are based on the best information available at the time. Unknown conditions may exist, and known conditions may change. This plan cannot account for every unknown or anticipate every contingency. Should personnel suspect or encounter areas of substantially higher levels of contamination, or should any situation arise which is obviously beyond the scope of the safety procedures specified herein, work activities shall be modified (such as by moving to another location) or halted pending discussions with the HSO and implementation of appropriate protective measures.

5.7 Accident and Incident Reports

If an incident or accident occurs, the HSO and PM shall be notified and the Incident Report (shown in Attachment 8) shall be completed. The report shall be completed by eyewitnesses (if possible) along with assistance from the SHSC. The report will be forwarded to the HSO as soon as possible for further investigation or follow up.

5.8 Emergency Contacts

Emergency notification information is summarized in Attachment 4 of this document.

TABLE 1
EXPOSURE/FIRST AID INFORMATION
NEASE CHEMICAL COMPANY - SALEM, OHIO PDI HASCP

Substances Present	Symptoms of Acute Exposure	First Aid
Mirex CAS No. 2385-85-5	Irritation to eyes, nose, throat; nausea, vomiting, headache, dizziness, muscle weakness, fatigue, convulsions; liver damage; amnesia.	Eye: Irrigate immediately Skin: Soap wash promptly Breathing: Respiratory support Swallow: Medical attention immediately

TABLE 2
TASK HAZARD ANALYSIS
NEASE CHEMICALCOMPANY SALEM OHIO SITE PDI HASCP

TASK DESCRIPTION	POTENTIAL HAZARDS	SEVERITY/ POTENTIAL	SAFE WORK PROCEDURES
MOBILIZATION/DEMOBILIZATION	POTENTIAL ATMOSPHERIC AND CONTACT HAZARDS	LOW	1) SAFETY ORIENTATION
	STRAINS FROM MANUAL LIFTING OF EQUIPMENT AND MATERIALS	MODERATE	1) USE PROPER LIFTING TECHNIQUES
	SLIPS, TRIPS AND FALLS	MODERATE	1) KEEP WORK ARES CLEAN AND DEBRIS FREE 2) ADEQUATE ILLUMINATION
SEDIMENT BODY MAPPING	SLIPS, TRIPS AND FALLS	MODERATE-HIGH	1) MAINTAIN CLEAR VISUAL/VERBAL COMMUNICATION 2) NOTE VISIBLE HAZARDS (DEBRIS, ROCKS, WET SOILS/SLUDGES) 3) WEAR SPECIFIC PPE IDENTIFIED IN SECTION 4.3
	SOIL CONTAMINATION	LOW	1) FOLLOW PERSONAL HYGIENE PRACTICES 2) TAKE NECESSARY PRECAUTIONS TO ELIMINATE OFF-SITE TRANSPORT OF SOIL FROM BOOTS, ETC.
SEDIMENT SAMPLING	SLIPS, TRIPS AND FALLS	MODERATE-HIGH	1) MAINTAIN CLEAR VISUAL/VERBAL COMMUNICATION 2) NOTE VISIBLE HAZARDS (DEBRIS, ROCKS, WET SOILS/SLUDGES) 3) WEAR SPECIFIC PPE IDENTIFIED IN SECTION 4.3
	DROWNING	MODERATE	1) MAINTAIN CLEAR VISUAL/VERBAL COMMUNICATION 2) FOLLOW SWP 25: WORKING ON OR OVER WATER 3) WEAR PERSONAL FLOTATION DEVICE IF RISK OF DROWNING IS PRESENT
	SEDIMENT CONTAMINATION	LOW	1) FOLLOW PERSONAL HYGIENE PRACTICES 2) TAKE NECESSARY PRECAUTIONS TO ELIMINATE OFF-SITE TRANSPORT OF SEDIMENT FROM BOOTS, ETC.
FISH ELECTROSHOCKING	SLIPS, TRIPS AND FALLS	MODERATE-HIGH	1) MAINTAIN CLEAR VISUAL/VERBAL COMMUNICATION 2) NOTE VISIBLE HAZARDS (DEBRIS, ROCKS, WET SOILS/SLUDGES) 3) WEAR SPECIFIC PPE IDENTIFIED IN SECTION 4.3
	DROWNING	MODERATE	1) MAINTAIN CLEAR VISUAL/VERBAL COMMUNICATION 2) FOLLOW SWP 25: WORKING ON OR OVER WATER 3) WEAR PERSONAL FLOTATION DEVICE IF RISK OF DROWNING IS PRESENT
	ELECTROCUTION	MODERATE	1) MAINTAIN CLEAR VISUAL/VERBAL COMMUNICATION 2) FOLLOW ELECTROSHOCKING STANDARD WORK PROCEDURE (ATTACHMENT 6)

TABLE 2
TASK HAZARD ANALYSIS
NEASE CHEMICALCOMPANY SALEM OHIO SITE PDI HASCP

TASK DESCRIPTION	POTENTIAL HAZARDS	SEVERITY/ POTENTIAL	SAFE WORK PROCEDURES
FISH FILLET PREPARATION	SLIPS, TRIPS AND FALLS	MODERATE-HIGH	1) MAINTAIN CLEAR VISUAL/VERBAL COMMUNICATION 2) NOTE VISIBLE HAZARDS (DEBRIS, ROCKS, WET SOILS/SLUDGES) 3) WEAR SPECIFIC PPE IDENTIFIED IN SECTION 4.3
	CUTS	MODERATE-HIGH	1) MAINTAIN CLEAR VISUAL/VERBAL COMMUNICATION 2) WEAR SPECIFIC PPE IDENTIFIED IN SECTION 4.3
	FISH CONTAMINATION	LOW	1) FOLLOW PERSONAL HYGIENE PRACTICES
FLOODPLAIN SOIL SAMPLING	SLIPS, TRIPS AND FALLS	MODERATE-HIGH	1) MAINTAIN CLEAR VISUAL/VERBAL COMMUNICATION 2) NOTE VISIBLE HAZARDS (DEBRIS, ROCKS, WET SOILS/SLUDGES) 3) WEAR SPECIFIC PPE IDENTIFIED IN SECTION 4.3
	SOIL CONTAMINATION	LOW	1) FOLLOW PERSONAL HYGIENE PRACTICES 2) TAKE NECESSARY PRECAUTIONS TO ELIMINATE OFF-SITE TRANSPORT OF SOIL FROM BOOTS, ETC.

TABLE 3
CHEMICAL CONSTITUENT INFORMATION
NEASE CHEMICAL COMPANY - SALEM, OHIO PDI HASCP

Constituent	OSHA PEL	NIOSH REL	IDLH	Ionization Potential, eVolts
Mirex	NE	NE, Ca	NE	NA

Notes:
Ca - Carcinogen
NE - Not Established
NA - Not Applicable
* values given are for iron oxide dust and fume (as Fe)

TABLE 4
LEVELS OF PERSONAL PROTECTION AND UPGRADE CRITERIA
NEASE CHEMICAL COMPANY - SALEM, OHIO PDI HASCP

Task	Initial Level of Protection	Air Monitoring Equipment	Upgrade Criteria	Upgraded/Downgrade Level of Protection
Site walks, survey	D1	NA	Condition Dependent	Condition Dependent
Fish Electroshocking	D1	NA	NA	NA
Fish Filleting	D2	NA	NA	NA
Floodplain soil and sediment sampling, sediment mapping	D1	NA	Upgrade to D2 if using a geoprobe cutting tool to cut plastic liners.	NA
Equipment Decontamination	D1 plus Safety Glasses, Goggles or Faceshield,	NA	Upgrade to D2 when cleaning sharp blades	NA

Condition Dependent: Personnel are to use prudent judgement and select additional PPE based on current Site conditions (e.g., wet or muddy) to prevent unnecessary contamination.

ATTACHMENT 1

FIELD PROCEDURES CHANGE AUTHORIZATION FORM

Instruction Number: _____ Date:_____

Duration of Authorization Requested ___ Today only

___ Duration of Task

Description of Procedures Modification:

Justification:

Person Requesting Change: Verbal Authorization Received From:

_____	_____	_____
Name	Name	Time
_____	_____	
Title	Title	
_____	_____	
Signature	Approved By	

(Signature of person named above to be obtained within 48 hours of verbal authorization)

ATTACHMENT 2

SITE HEALTH AND SAFETY/CONTINGENCY PLAN ACKNOWLEDGEMENT

I have read understand and agree to follow the provisions detailed in the Health and Safety/Contingency Plan for the Nease Chemical Company Site in Salem, OH.

I am aware of emergency procedures, equipment locations, and emergency telephone numbers.

I understand that my failure to comply with these provisions may lead to disciplinary actions and/or my dismissal from the Site.

Printed Name	Organization	Signature	Date/Time

This form is to be kept on file on Site. Copies should be made available to personnel from all companies involved with Site work.

ATTACHMENT 3

REPORT FORM FOR UNSAFE CONDITIONS AND PRACTICES

DESCRIPTION OF UNSAFE CONDITION OR PRACTICE _____

_____

_____

_____

_____

DESCRIPTION OF CIRCUMSTANCES SURROUNDING UNSAFE CONDITION OR PRACTICE _____

_____

_____

_____

_____

IS THIS AN EXISTING CONDITION OR POTENTIAL HAZARD? _____

REPORTED TO _____

REPORTED BY _____ DATE _____

COMMENTS _____

_____

_____

_____

_____

REPORT RECEIVED BY _____

DATE _____

ATTACHMENT 4

EMERGENCY NOTIFICATION CONTACT LIST

1. Hazardous Substance Spills (SARA Title III)

Ohio EPA Emergency Response Unit
c/o Ohio Emergency Response Commission
122 South Front Street
P.O. Box 1049
Columbus, Ohio 43215-1049
ATTN: ER Records Mgmt.

(800) 282-9378 or (614) 224-0946

2. Hospital

Salem Community Hospital (non-life threatening injuries)
1995 E. State Street
Salem, OH 44460

Emergency: 911
Main Number: (330) 332-1551

St. Elizabeth Health Center (life threatening or traumatic injuries)
1044 Belmont Avenue
Youngstown, OH 44501

Emergency: 911
Main Number: (330) 746-7211

3. Police

Salem Police
231 S Broadway Avenue
Salem, Ohio 44460

Emergency: 911
Main Number: (330) 337-7811

4. Fire

Salem Fire Department
260 S Ellsworth Ave
Salem, OH 44460

Emergency: 911
Main Number: (330) 337-6183

NOTE: **THIS LIST MUST BE POSTED AT THE PROJECT SITE AND IN ALL SITE VEHICLES ALONG WITH THE ATTACHED HOSPITAL ROUTE MAPS (ATTACHMENT 8)**

ATTACHMENT 5

GOLDER STANDARD WORK PROCEDURES

BIOLOGICAL EXPOSURE RISKS – HAZARDOUS FLORA AND FAUNA INFORMATION

The scope of services for this project may create opportunities for site personnel to come in contact with potentially dangerous plants and animals. Precautionary measures should be taken to reduce possible contact with hazardous flora and fauna which may include, but not be limited to: application of bug spray, caution and care in handling collected organisms including wearing gloves, avoiding or leaving an area identified as containing a suspected hazardous plant or animal (e.g. poisonous snake) until the hazardous organism has been properly identified and/or removed.

If a field team member comes in contact with or is bitten or stung by any hazardous plant or animal, always follow the proper first aid procedures and contact the Site Safety Officer immediately.

In general, almost any terrestrial animal can be hazardous at times. Larger mammals and some birds may attack if you are in their territory, too close to their nest, between them and their offspring, etc. Also, if you are handling / attempting to handle wildlife, there may be a risk of bites or scratches leading to infection, excessive bleeding, or worse. Please always use caution when working with/around wildlife. Although they are often wary and will stay away from you, there are specific situations/times where this is not the case.

HAZARDOUS INSECTS

Poisonous Spiders:

- **Widow Spiders** (genus *Latrodectus*) Southern Black Widow, Northern Black Widow, Brown Widow, and Red Widow
- **Recluse Spiders** (genus *Loxosceles*) - Brown Recluse, Mediterranean Recluse, Chilean Recluse

While the poisonous spiders listed above are not known to be aggressive, spider bites can occur when the spiders are disturbed or feel threatened. Be careful of reaching under objects where the spider is hiding or when putting on clothing, gloves or shoes that have been sitting or stored for a long time into which the spider has crawled. The most common symptoms are intense pain, rigid abdominal muscles, muscle cramping, malaise, local sweating, nausea, vomiting, and hypertension. Blisters frequently appear at the bite site, which can become necrotic. If bitten, remain calm, and seek medical attention (contact your physician, hospital and/or poison control center). Apply an ice pack directly to the bite area to relieve swelling and pain. If possible to do so without further endangerment, collect the spider (even a mangled specimen has diagnostic value) for positive identification by a spider expert.

Bees, Wasps, Yellow jackets, Mosquitoes and Fire Ants:
The hazards associated with insect bites and stings are based on allergic reactions to the venom produced by the insect. Anyone who has a history of severe allergic reactions to bee stings should carry an anaphylactic kit with an Epinephrine syringe (EpiPen®), with them at all times when working in the field. If an EpiPen® is unavailable, remove the victim from the area (to avoid further stinging), monitor for signs of anaphylactic shock, and seek medical attention.

Ticks

Tick Identification

- Ticks are divided into two families, "soft" ticks and "hard" ticks. Their only source of nutrition is blood sucked from their hosts.
- Hard ticks live on the ground in vegetation, such as grassy meadows, woods, brush, weeds, and leaf litter.
- Ticks produce a cement-like substance that helps anchor them to the host.
- Ticks inject saliva (the saliva may also contain disease organisms, such as *Borrelia burgdorferi* which cause Lyme disease) containing a kind of anticoagulant into the blood pool to keep the blood from clotting.
- The Brown Dog Tick (*Rhipicephalus sanguineus*) has evolved to live indoors and can be found living inside your home. Brown Dog Ticks do not feed on humans.

Avoiding Ticks / Tick bites

- Avoid working in vegetated areas if possible
- Call local state park officials to determine if ticks will be abundant during planned project work
- If work is unavoidable in vegetated areas where ticks are known to be present (especially during Spring, Summer or Fall)
  - Use insect repellent
  - Wear light colored clothing to make ticks easier to spot and remove
  - Carefully check all parts of your body at least daily for ticks and follow the procedure below if a tick is attached to your skin.
- If ticks are known to be abundant (in addition to the above)
  - Wear Tyvek ® or similar pants taped to boot covers

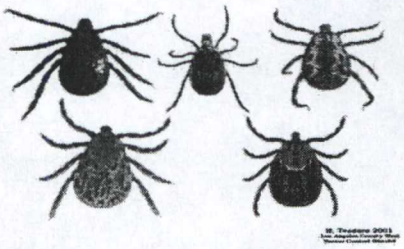
Tick Removal / Procedures

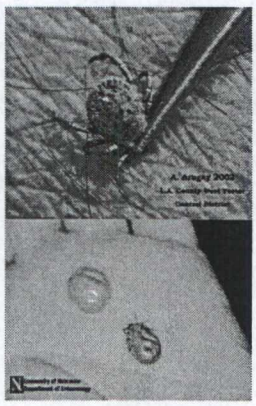
- Use a small pair of curved forceps or tweezers; wear some gloves so you don't spread bacteria from the tick to your hands.
- Using tweezers flip the tick over onto its back. Grasp tick firmly with the tweezers as close to the skin as possible. Gently pulling until the tick comes free. Do not twist or turn.
- Once removed, rinse it down a sink or flush it down a toilet. Consider keeping it in a tightly closed jar or taped to a piece of paper. You may need to show the tick to the doctor if you become ill from the tick bite.
- The area of the bite should leave a small crater or indentation where the head and mouthparts were embedded. If significant portions of the head or mouthparts remain, they may need to be removed by a doctor.
- Cleanse the bite area with soap and water or a mild disinfectant. Observe the area for several days for any reaction to the bite, such as a rash or signs of infection. Apply antibiotic cream to the area.
- Remember to wash your hands thoroughly after handling any tick or instruments that touched a tick. Clean and disinfect any instruments that were used.
- Most tick bites are probably harmless and cause no problems. The earlier a tick is removed, the less the likelihood that the tick transmitted any disease.

When to Seek Medical Attention

Medical attention, other than basic first aid as described above, is rarely necessary for tick bites. See a licensed medical professional:

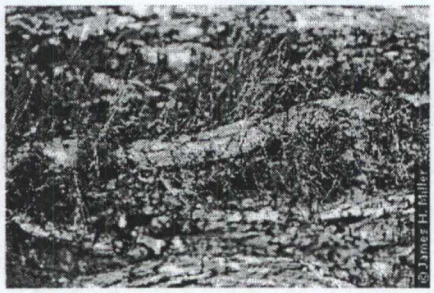
- If you cannot completely remove all parts of the tick
- If you develop any of these symptoms:
  - A rash
  - A fever
  - A stiff neck
  - Muscle aches
  - Joint pain and inflammation
  - Swollen lymph nodes
  - Flu –like symptoms





Poison Ivy

Poison Ivy (Rhus radicans) Identification



- Three forms of poison ivy, 1) Erect woody shrub, 2) Running Shrub 3) Woody Vine.
- Three leaflets, two to four inches long, dull or glossy green with pointed tips.
- The middle leaflet is generally larger than the two laterals.
- The margins of the leaflets are variable, appearing irregularly toothed, lobed, or smooth.
- The leaves are positioned *alternately* on the stems.
- Woody vines grow on trees or objects for support and have aerial roots along the stem.

Treatment for Poison Ivy

- Have a 'poison ivy action kit' ready, with rubbing alcohol, a large bottle of water, some soap and gloves
- Cleanse exposed areas with rubbing alcohol.
- Next, wash the exposed areas with water only (no soap yet, since soap can move the urushiol, which is the oil from the poison ivy that triggers the rash, around your body and actually make the reaction worse).
- Now, take a shower with soap and warm water.
- Lastly, put gloves on and wipe everything you had with you, including shoes, tools, and your clothes, with rubbing alcohol and water.
- Apply Ivy-Block to exposed areas to prevent Poison Ivy.

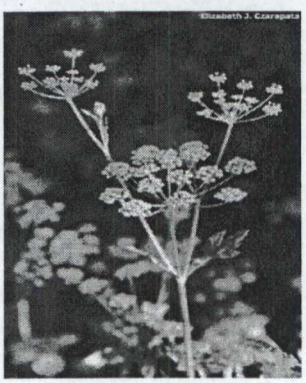
<u>Do NOT burn these poisonous plants! The smoke will irritate your skin badly, and if it enters your nasal passages, throat and lungs it will likely cause serious problems and could result in death.</u>

Wild Parsnip

Wild Parsnip Identification

- Pinnately compound leaves, with a main stem and 5 to 15 leaflets.
- Yellow flowers, in flat-topped umbrella-like clusters at the top of the plant.
- Wild parsnip rosettes are among the first plants to become green in spring, and its flowers turn a prominent yellow in midsummer. After flowering and going to seed, plants die and turn brown in fall, but first year rosettes remain green until frost.
- Grows throughout the United States along: Roadsides, abandoned fields, unmowed pastures, edges of woods, prairie restorations.

Treatment for Wild Parsnip



- Take Ibuprofen for help with swelling and pain. The burned area can be covered with a cool, wet cloth. If blisters are present, do not pop them and if the blisters rupture, leave the skin "bandage" in place. To avoid infection, keep the area clean and apply an antibiotic cream.
- Adding Domeboro® powder to cool cloth compresses can help dry any of the seeping blisters.
- Doctors will recommend a topical or systemic cortisone-steroid for extreme discomfort. See a doctor for serious cases.
- Washing with Fels-Naptha® soap <u>will not</u> help.

Snakes

- Leave snakes alone;
- Wear rubber boots, long trousers and long sleeved shirts; the fangs of a snake will not normally penetrate such clothing;
- Be noisy when walking in the bush; stamp your feet;
- Look carefully when walking through grass; and
- Do not place bare hands in hollow logs, under piles of wood or rubbish. **WEAR GLOVES.**

Snake Bite Victims

Most snake bites occur on the limbs, with 75% of reported cases being on the lower limbs. If anyone is bitten be more concerned about the casualty than about identifying the snake. Unconsciousness and breathing failure may occur. The only widely acceptable treatment for venomous snakebites involves the use of antivenin. If someone is bitten by a venomous snake, seek immediate medical attention at the nearest hospital or medical facility. Stay calm, remove any rings that could restrict circulation if tissues swell, keep the bitten limb below the level of the heart, and immediately seek medical attention.

Symptoms of Snake Bite

- Headache
- Double-Vision
- Drowsiness
- Nausea
- Pain or tightness in the chest or abdomen
- Giddiness or faintness
- Diarrhea

These symptoms do not appear immediately, but from about 15 minutes to 2 hours after being bitten. If a snake bite is suspected, act promptly and obtain medical assistance without delay - **HOWEVER, STAY CALM.**

Signs of Snake Bite

Look for:

- 2 puncture marks about 1 cm apart at the site of the bite, although sometimes they may only be a fang scratch on the skin (although this depends on the size of the snake)
- Swelling of the area around the bite
- Bruising
- Sweating
- Vomiting
- Breathing difficulties

Management of Snake Bite Victims

- Keep the victim at rest
- Reassure the victim
- Immediately apply a pressure immobilization bandage over the bitten area and around the limb
- Apply the bandage firmly enough to compress tissues, but not so firmly as to restrict the flow of blood to the limb below the bandage
- Bandage from the bite to the fingers or toes, then up to the armpit or groin
- Bandage as much of the limb as possible

- If assistance is available, get them to seek medical aid urgently. It is better to wait for the arrival of an ambulance or ambulance officer rather than to move the victim, but isolation and other circumstances should determine what is to be done
- Periodically observe and record the pulse and breathing
- Carry out rescue breathing / CPR if breathing or circulation fails

As a precaution: **Do not catch a snake and do not handle one unless you are sure it is not venomous.** In addition, for a short time after a snake is killed, its reflexes may continue to work. Those reflexes typically cause the body to writhe slowly for awhile, but they can cause a convulsive contraction and a bite, so you should not handle a freshly killed venomous snake. Though US medical professionals may not agree on every aspect of what to do for snakebite first aid, they are nearly unanimous in their views of what not to do. Among their recommendations:

WARNING - WHAT NOT TO DO

- **NEVER** wash the venom off the skin; the venom will help identify what type of snake is involved - EMERGENCY DECONTAMINATION PROCEDURES DO NOT APPLY;
- **NEVER** cut or excise the bitten area. Such measures have not been proven useful and may cause further injury.
- **NEVER** try to suck the venom out of the wound; and
- **NEVER** use a constrictive bandage or tourniquet. This cuts blood flow completely and may result in loss of the affected limb.
- **NEVER** ice or any other type of cooling on the bite. Research has shown this to be potentially harmful.

Timber Rattlers

Timber Rattlers Identification

- The Timber Rattler is an endangered species
- Adult timber rattlers average 35 to 50 inches in total length.
- The tail is short and thick, all black, and tipped with a tan rattle.
- Timber rattlers are diurnal (active during the day) and nocturnal (active after dark) in the summer, but only diurnal in the spring and fall.
- This venomous species can inflict a serious bite, and people are urged to walk away from them when encountered. Records show that there is a greater risk of being bitten if an attempt is made to kill them
- During the summer, they inhabit deciduous forests and open valleys. Rock fissures and crevices provide communal dens for over wintering.
- Timber rattlers begin emerging from hibernation in April to early May.

Treatment for Timber Rattlers

- Moderate symptoms will include mild swelling, discoloration, and pain at the wound site, and may also include general tingling, weakness, rapid pulse, and dimness of vision, nausea, vomiting, and shortness of breath.
- For moderate symptoms, apply a band 3/4 inch to 11/2 inches wide to the limb, 2 to 4 inches above the wound (but not around a joint, or on the head, neck, or trunk). Make it snug, but loose enough so you can slip a finger under it. Check the victim's pulse beyond the band periodically; to be sure blood is flowing past the band. Loosen the band if it becomes too tight.
- Severe symptoms include rapid swelling and numbness, followed by severe pain at the wound site; there may also be pinpoint-size pupils, facial twitching, slurred speech, convulsions, paralysis, and loss of consciousness. For severe symptoms, apply the band, and then make a shallow cut-just through the skin-through each fang puncture, 1/2 inch long and parallel with the

long axis of the limb. (Make no cuts on the head, neck, or trunk). Apply a suction cup for 30 minutes, or use your mouth to such out the venom. Don't apply ice packs or any other kind of cold therapy. Continue treatment until you can get medical help. Most people who die of snakebite today die because they didn't seek medical help or delayed too long in going to the hospital. Preventive medicine is the best method for dealing with snakebites.

- Begin first aid treatment by keeping the victim calm and immobile, preferably lying down. Immobilize the bitten limb, at or below heart level. If the victim will be able to get to a hospital within 4 or 5 hours-and no symptoms develop-no more first aid is necessary.

HAZARDOUS FRESHWATER AND SALTWATER LIFE

Fin spines

Many fish have sharp spines in their fins that can cause puncture wounds if not handled properly. Although most do not have toxins/venom in their spines, the wound may still get infected if not properly cleansed and bandaged. Some fish have sharp teeth that may cause moderate to severe lacerations if not handled properly. The following sections provide examples of fishes and/or organisms that may require special handling or avoidance.

Stingrays

Stingray skin can be slippery, and need to be handled with gloves. If a stingray stings you, seek immediate medical attention. Infection can develop from bacteria entering the wound via seawater or from the stinger (spine) itself. A stingray spine can also break off in the wound and cause infection if it is not removed; however, **the spine is serrated, so if the spine breaks off in the wound, do not remove it yourself**. Instead, apply first aid for a puncture wound, including flushing/rinsing the wound with fresh water or hydrogen peroxide if available, and seek immediate medical attention. Sometimes the spine will stick into the person without breaking off of the animal. In this case, cut the spine from the animal close to the base of the tail, leaving as much of it sticking out of the wound as possible. The pain of the stingray toxin can be alleviated before reaching a medical facility by applying a heat compress to the wound or soaking it in clean, hot water which breaks down the protein-based toxin.

Catfishes

Catfishes have spines very similar to stingrays. Treat as you would for a stingray wound.

Scorpionfishes, Leatherjackets, Toadfishes

Some, if not all species of fish in these groups have poisonous spines in their dorsal, pectoral, and/or anal fins. The spines are very sharp and the toxin in them can cause varying degrees of reaction. By far, the

scorpionfishes have the most potent toxin in their spines and some species can be much larger than others. In the case of being stung by any of these fish, apply first aid for a puncture wound, and watch for signs of reaction to the toxins. Seek medical attention if necessary. The pain of the toxin can be alleviated before reaching medical facilities by applying a heat compress to the wound or soaking it in clean, hot water

Sea Urchins
Sea urchins have sharp spines that project out and protect their main body, or test, from all angles. While some species have poisonous or toxic spines, the primary hazard is from puncture wounds where the spine breaks off in the wound and the resulting infection. The spines, like the rest of the urchin's body, are primarily made of calcium that can be brittle and hard to remove. If someone gets stuck with urchin spines, flush the area well with water and hydrogen peroxide (if available). If the wound is large or there are large pieces of spine embedded in the wound, apply first aid as you would for a normal puncture wound and monitor for signs of allergic reaction to possible toxins. Applying a heat pack in the immediate area of the wound may relieve some of the pain associated with the toxin if it is present. It may be necessary to go to the hospital to get the pieces of spine removed.

Jellyfish
Most species of Jellyfish have stinging cells, called nematocysts, which trigger or "fire" upon touch. If jellyfish are encountered in the net, the use of gloves in handling them can prevent the person from being stung, as the nematocysts are usually very small and will not penetrate the glove. However, be cautious after handling jellyfish even with a glove, as some of the nematocysts can rub off and remain on the glove and still fire long after the jellyfish has been removed. If someone gets stung by a jellyfish, it is best to rinse the area with vinegar (do not use fresh water, as that can cause more nematocysts to fire). Different species of jellies have various levels of toxin in their sting. There is a potential for allergic reaction, so monitor the victim and seek medical attention if necessary.

Corals and Sponges
Corals and Sponges have stinging cells similar to Jellyfish. If someone is stung by a species of coral or sponge, treat the same as above for jellyfish.

Crabs/Crayfish/Lobsters
All of the above can have very strong and very sharp claws. Use caution when handling them.

Oysters/Mussels/Clams
Oysters and Mussels have extremely sharp edges that can cut easily. Care should be taken when handling and always wear gloves.

American Alligator (may encounter on land or in the water)
The best treatment for alligator wounds is avoidance or prevention of encounters. When left alone, alligators will stay away from humans and pose little threat. The springtime is when alligator breeding season begins in Florida. During breeding season, male alligators become very territorial and will defend their area against intruders. Following the breeding, in June-July, females will lay their eggs in nests created in the preceding months. These nests are mounds of mud, leaves and other vegetation and can be ~1 meter tall and ~2 meters wide. Look out for these nests near the shoreline of swamps, rivers, and estuaries, as female alligators remain near the nest, in nearby water or other shelter, throughout the incubation period, which averages 65 days, and will defend her nest if danger threatens. If someone is attacked by an alligator, apply first aid, including pressure to any open wounds, and seek immediate medical attention.

HEAT STRESS

Employees may experience heat stress due to a combination of elevated ambient temperatures and the concurrent use of personal protection equipment; this depends in part on the location of the site, the type of work, and the time of year. The project manager (PM) should consider the need to monitor heat stress during the project planning stage. The Site Safety Officer (SSO) and/or field staff will evaluate heat stress using the techniques specified below whenever the ambient temperature exceeds 21°C or 70°F.

HEAT STRESS RELATED PROBLEMS

- **Heat Rash** - caused by continuous exposure to heat and humid air and aggravated by chafing clothes. Decreases ability to tolerate heat, as well as being a nuisance;
- **Heat Cramps** - caused by profuse perspiration with inadequate fluid intake and chemical replacement. Signs: muscle spasms and pain in the extremities and abdomen;
- **Heat Exhaustion** - caused by increased stress on various organs to meet increased demands to cool the body. Signs: shallow breathing; pale, cool, moist skin; profuse sweating; dizziness, and lassitude. If symptoms occur, the employee should leave the work area and proceed to the nearest air-conditioned location, drinks liquids such as water or Gatorade, and rest until the symptoms pass. Contact the Golder PM immediately; and
- **Heat Stroke** - the most severe form of heat stress. Body must be cooled immediately to prevent severe injury and/or death. Signs: red, hot, dry skin; no perspiration; nausea; dizziness and confusion; strong, rapid pulse; coma. Medical help must be obtained immediately. If heat stroke is suspected, implement emergency response plan. Remove excess clothing and cool the person by sponging with cool or luke warm water. Never place ice on the person or throw water on the individual. Contact the Golder Project Manager as soon as time permits.

HEAT STRESS MANAGEMENT

Heat Stress Monitoring

The American Conference of Governmental Industrial Hygienists (1992) states that workers should not be permitted to work when their deep body temperature exceeds 38°C (100.4°F).

For strenuous field activities that are part of ongoing site work activities in hot weather, the following procedures shall be used to monitor the body's physiological response to heat, and to monitor the work cycle of each site worker. There are two phases to this monitoring: initial work/rest cycle determination and physiological monitoring. The initial work/rest cycle is used to estimate how long the first work shifts of the day should be. Heart rate monitoring of each worker will establish the length of the successive work periods.

Determination of Initial work/Rest Cycles

Measure the air temperature with a standard thermometer. Estimate the fraction of sunshine by judging what percent the sun is out: 100% sunshine - no cloud cover = 1.0; 50 % sunshine and 50% cloud cover = 0.5; 0% sunshine = full cloud cover = 0.0.

Plug these variable into the following equation to determine the adjusted temperature:
T (°C, adjusted) = T (°C, actual) + (7.2 x fraction sunshine)

Use the chart below to determine the length of the first work shift. At the first break, initiate the heart rate monitoring as described in the next section.

ADJUSTED TEMPERATURE	NORMAL WORK CLOTHING	IMPERMEABLE CLOTHING
21° - 25°C (70-77°F)	150 Minutes	120 Minutes
25° - 28°C (77-82°F)	120 Minutes	90 Minutes
28° - 31°C (82-88°F)	90 Minutes	60 Minutes
31° - 32°C (88-90°F)	60 Minutes	30 Minutes
>32°C (>90°F)	45 Minutes	15 Minutes

Heart Rate Monitoring

Heart rate (HR) should be measured by radial pulse for 30 seconds as early as possible in the resting period. The HR at the beginning of the rest period should not exceed 110 beats/minute. If the HR is higher, the next work period should be shortened by 33 percent while the length of the rest period stays the same. If the pulse rate still exceeds 110 beats/minute at the beginning of the next rest period, the following work period should be further shortened by 33 percent while the length of the rest period stays the same.

Heat Stress Prevention

The best approach is preventive heat stress management. In general:

- have workers drink additional water before beginning work;
- provide disposable cups of water that is maintained at 10 to 16°C (50 to 60 °F);
- urge workers to drink one to two cups of water every 20 minutes or at each rest break for a total of four to eight litres per day;
- provide a cool, preferably air-conditioned area for rest breaks;
- discourage the drinking of alcohol at night and discourage the intake of coffee during working hours;
- monitor for signs of heat stress;
- acclimatise workers to site work conditions by slowly increasing workloads, ie., do not begin site work activities with extremely demanding activities; and
- Reschedule your work hours so that you are not working in the heat of the day between 10 am and 2 pm.

Sun Protection

- Employees are encouraged to maximize use of the shade provided by trees, buildings and other structures. Where there is limited access to natural shade, fixed or portable shade structures may be used and will be provided where practical.
- Rotate your work with others between indoor/shaded areas and outdoor/exposed locations to minimize time spent in the sun.
- The selection of appropriate protective clothing will take into account both the need to block out UV and the need to reduce the effects of heat.
- It is recommended that Golder employees tight woven clothing which has a minimum UPF of at least 30. Clothing should be lightweight, loose fitting and have a collar to assist with keeping cool.

- Hats provide shade and the bigger the brim the greater the amount of shade that is provided. Hats should be made of close-weave material and have a wide brim or be legionnaire-style. In circumstances where the wearing of a broad-brimmed hat causes difficulties due to their size sunscreen and other protective measures should be used instead.
- Safety glasses will be supplied to protect the eyes from effects of UV radiation and potential eye injuries from flying objects, dust or chemical splashes. Safety glasses complying with **ANSI Z87.1-2003** are recommended.
- Sunscreen does not offer complete protection and should always be used in conjunction with other protection such as protective clothing. Broad spectrum and water-resistant sunscreen with a sun protection factor (SPF) of 30+ should be used.
- Staff using sunscreen are encouraged to regularly check use by dates to ensure sunscreen is not out of date.
- Sunscreen will be placed in an easily accessible location and employees instructed in correct application and use. Sunscreen should be generously applied to all areas of exposed skin at least twenty minutes before going outside and should be reapplied at least every two hours.

COLD ENVIRONMENT – COLD STRESS

In a cold environment, body heat must be conserved to maintain the core temperature at normal levels and to ensure an adequate blood flow to the brain and extremities. Feelings of cold and discomfort should not be ignored, since these may be early warning signals. The effects of cold are such that problems can occur before the worker is aware of them, and furthermore, over-exposure to cold may affect judgment.

MAIN FACTORS INVOLVED IN CAUSING COLD STRESS

- Temperature
- Humidity
- Movement of air
- Radiant temperature of the surroundings
- Clothing/physical activity

COLD STRESS RELATED PROBLEMS

- **Frostbite** is a condition in which the skin and underlying tissues freeze. Usually affects fingers, hands, toes, feet, ears and nose.
- **Hypothermia** is a condition in which a person's body temperature falls below 95^0 F or 35 degrees Centigrade. Hypothermia occurs when more heat is lost from the body than the body can produce. It usually happens when a person is exposed to extremely cold temperatures but it can occur even at moderate temperatures. It does not have to be freezing outside for a person to become hypothermic. For example, falling into cold water or wearing wet clothing in cold weather can bring on hypothermia. Failing to wear a hat in cold weather can also lead to hypothermia, since a large amount of body heat escapes from the head. Extreme fatigue, hunger or lack of fluids can also lead to hypothermia. As well, excessive wind can increase the amount of heat lost and cause hypothermia.

FROSTBITE MANAGEMENT

- Move person to a warm dry area. Don't leave the person alone.
- Minimize walking on frozen feet.
- Do not apply any lotions or ointments to frozen skin.
- Remove any wet or tight clothing that may cut off blood flow to the affected area.
- **DO NOT** rub the affected area, because rubbing causes damage to the skin and tissue.
- **Gently** place the affected are in a warm (105°F) water bath and monitor the water temperature to slowly warm the tissue. Don't pour warm water directly on the affected area because it will warm the tissue too fast causing tissue damage. Warming takes about 25-40 minutes.
- After the affected area has been warmed, it may become puffy and blister. The affected area may have a burning feeling or numbness. When normal feeling, movement, and skin color have returned, the affected area should be dried and wrapped to keep it warm. NOTE: If there is a chance the affected are may get cold again, do not warm the skin. If the skin is warmed and then becomes cold again, it will cause severe tissue damage.
- Seek medical attention as soon as possible and contact the Site Safety Officer.

HYPOTHERMIA MANAGEMENT

The most obvious sign of hypothermia is a low core body temperature. The person with hypothermia may not realize that his or her prolonged exposure to cold requires emergency medical care. Other signs and symptoms include:

- apathy or loss of interest in surroundings
- lethargy or difficulty moving
- confusion
- drowsiness
- loss of coordination
- cold skin
- shock caused by decreased blood flow
- slurred speech
- uncontrollable shivering
- weakness

If a person is suspected of suffering from hypothermia, contact the Site Safety Officer, and apply first aid.

What should be done (land):

- Move the person to a warm, dry area. Don't leave the person alone. Remove any wet clothing and replace with warm, drying clothing or wrap the person in blankets.
- Have the person drink warm, sweet drinks (sugar water or sports-type drinks) if they are alert. **Avoid** drinks with caffeine (coffee, tea or hot chocolate) or alcohol.
- Have the person move their arms and legs to create muscle heat. If they are unable to do this, place warm bottles or hot packs in the arm pits, groin, neck and head areas. **DO NOT** rub the person's body or place them in a warm bath. This may stop their heart.

What should be done (water):

- **DO NOT** remove any clothing. Button, buckle, zip and tighten any collars, cuffs, shoes, and hoods because the layer of trapped water closest to the body provides a layer of insulation that slows the loss of heat. Keep the head out of the water and put on a hat or hood.
- Get out of the water as quickly as possible or climb on anything floating. **DO NOT** attempt to swim unless a floating object technical water rescue can be reached because swimming or other physical activity uses the body's heat and reduces survival time by about 50 percent.
- If getting out of the water is not possible, wait quietly and conserve body heat by folding arms across the chest, keeping thighs together, bending knees, and crossing ankles. If another person is in the water, huddle together with chests held closely.

PRECAUTIONS

- Use the buddy system.

- Recognize the environment and workplace conditions that lead to potential cold-induced illnesses and injuries.
- Learn the sign and symptoms of cold induced illnesses/injuries and what to do to help the worker.
- Dress appropriately for expected weather conditions. Dress in a minimum of three layers (a skin layer to absorb moisture and keep the skin dry, an insulating layer, and an outer protective layer), wear a hat and gloves, in addition to underwear that will keep water away from the skin.
- Take frequent short breaks in warm dry shelters to allow the body to warm up.
- Perform work during the warmest part of the day.
- Eat warm, high calorie foods like hot pasta dishes.
- Avoid vasodilators, which allow the body to lose heat faster - which can accelerate hypothermia. These include alcohol and drugs;
- Avoid vasoconstrictors, including tobacco products, which constrict blood vessels and can accelerate the onset of frostbite;
- Avoid touching cold metal with bare skin; and
- Keep active.

SLIPS, TRIPS AND FALLS

Over half of all office injuries are the result of falls. The majority of falls occur on slippery, uneven, defective, cluttered or obstructed walking surfaces. A significant number of debilitating falls are the result of a person falling out of his or her own chair, typically while in the process of sitting down, or leaning back. Falls from elevations while reaching for an overhead object are also common, and frequently cause severe injuries.

PRECAUTIONS WHEN IN THE OFFICE - HOUSEKEEPING

- Watch your step! Wipe up spilled liquids immediately. Tripping hazards such as defective floors, missing floor tiles, loose or matted carpeting, bunched-up floor mats, extension cords, phone cords, etc., should be corrected or reported and repaired immediately. Don't carry loads that are so large or bulky that the line of vision is impaired.
- Be careful when sitting down. Sitting on the edge of a seat, sitting too far back, or kicking the chair out from under one's self can result in a fall and fractured vertebrae. Occasionally check the mechanical condition of chairs commonly used.
- Be especially careful going up and down stairs. Avoid using stairs if both arms are loaded. Watch your step and if possible always have one hand free to use a railing. Maintain 3 points of contact when ascending/descending.

PRECAUTIONS WHEN OUT IN THE FIELD

In the field, falls are the second leading cause of work-related deaths.

TYPES OF FALLS

Falls are of two basic types: elevated falls and same-level falls. Same-level falls are most frequent, but elevated falls are more severe.

- Same-Level Falls: high frequency--low severity
- Elevated Falls: lower frequency--high severity

Same-level falls are generally slips or trips. Injury results when the individual hits a walking or working surface or strikes some other object during the fall. Over 60 percent of elevated falls are from less than 10 feet.

SAME-LEVEL FALLS

Examples of same-level falls are described below.

SLIP AND FALL

Slips are primarily caused by a slippery surface and compounded by wearing the wrong footwear. In normal walking, two types of slips occur. The first of these occurs as the heel of the forward foot contacts the walking surface. Then, the front foot slips forward, and the person falls backward.

The second type of fall occurs when the rear foot slips backward. The force to move forward is on the sole of the rear foot. As the rear heal is lifted and the force moves forward to the front of the sole, the foot slips back and the person falls.

The force that allows you to walk without slipping is commonly referred to as "traction." Common experience shows that dry concrete sidewalks have good traction, while icy surfaces or freshly waxed floors can have low traction. Technically, traction is measured as the "coefficient of friction." A higher coefficient of friction means more friction, and therefore more traction. The coefficient of friction depends on two things: the quality of both the walking surface and the soles of your shoes.

To prevent slips and falls, a high coefficient of friction (COF) between the shoe and walking surface is needed. On icy, wet, and oily surfaces, the COF can be as low as 0.10 with shoes that are not slip resistant. A COF of 0.40 to 0.50 or more is needed for excellent traction. To put these figures in perspective, a brushed concrete surface and a rubber heel will often show a COF greater than 1.0. Leather soles on a wet smooth surface, such as ceramic tile or ice, may have a COF as low as 0.10.

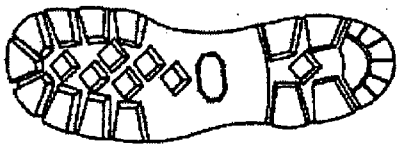


Figure 1. Shoes with soft rubber soles and heels with rubber cleats provide a high coefficient of friction (COF).

Providing dry walking and working surfaces and slip-resistant footwear are the answer to slips and their resultant falls and injuries. Obviously, high heels, with minimal heel-to-surface contact, taps on heels, and shoes with leather or other hard, smooth-surfaced soles lead to slips, falls, and injuries. Shoes with rubber-cleated, soft soles and heels provide a high COF and are recommended for most agricultural work.

In work areas where the walking and working surface is likely to be slippery, non-skid strips or floor coatings should be used. Since a COF of 0.40 to 0.50 is preferred for walking and working surfaces, we should strive for a surface which provides a minimum of 50 percent of this friction. If the working surface is very slippery, no footwear will provide a safe COF.

Trip and Fall Trips occur when the front foot strikes an object and is suddenly stopped. The upper body is then thrown forward, and a fall occurs.

As little as a 3/8" rise in a walkway can cause a person to "stub" his toe resulting in a trip and fall. The same thing can happen going up a flight of stairs: only a slight difference in the height of subsequent steps and a person can trip and fall.

CONTRIBUTING FACTORS

Proper housekeeping in work and walking areas can contribute to safety and the prevention of falls. Not only is it important to maintain a safe working environment and walking surface, these areas must also be kept free of obstacles which can cause slips and trips. One method which promotes good housekeeping in work environments is the painting of yellow lines to identify working and walking areas. These areas should never be obstructed by objects of any kind.

Adequate lighting to ensure proper vision is also important in the prevention of slips and falls. Moving from light to dark areas, or vice versa, can cause temporary vision problems that might be just enough to cause a person to slip on an oil spill or trip over a misplaced object.

Carrying an oversized object can also obstruct one's vision and result in a slip or a trip. This is a particularly serious problem on stairs.

BEHAVIORS THAT LEAD TO FALLS

In addition to wearing the wrong footwear, there are specific behaviors which can lead to slips, trips, and falls. Walking too fast or running can cause major problems. In normal walking, the most force is exerted when the heel strikes the ground, but in fast walking or running, one lands harder on the heel of the front foot and pushes harder off the sole of the rear foot; thus, a greater COF is required to prevent slips and falls. Rapid changes in direction create a similar problem.

Other problems that can lead to slips, trips and falls are: distractions; not watching where one is going; carrying materials which obstruct view; wearing sunglasses in low-light areas; and failure to use handrails. These and other behaviors, caused by lack of knowledge, impatience, or bad habits developed from past experiences, can lead to falls, injuries, or even death.

MOTOR VEHICLES AND DRIVING ON COMPANY BUSINESS

Unlike other workplaces, the roadway is not a closed environment. Preventing work-related roadway crashes requires strategies that combine traffic safety principles and sound safety management practices. Although employers cannot control roadway conditions, they can promote safe driving behavior by providing safety information to workers and by setting and enforcing driver safety policies. Vehicle crashes are not an unavoidable part of doing business.

GENERAL GUIDELINES

- Enforce mandatory seat belt use. Seat belts shall be worn by all drivers and passengers in vehicles on company business.
- Private vehicles used for work purposes must carry appropriate insurance.
- The risks presented by driving while fatigued must be considered on all projects. Do not require workers to drive irregular hours or far beyond their normal working hours.
- Develop work schedules that allow employees to obey speed limits and to follow applicable hours-of-service regulations.
- Observe all the rules and regulations pertaining to the use of public land. Always ask permission before crossing pastoral land. Leave gates as you find them. Keep to constructed vehicle tracks. Avoid areas that are easily damaged, such as swamps, alpine snow plains and vegetated sand dunes.
- Observe all fire restrictions.
- Refer to the Cell Phone Safe Work Procedure for more information.

VEHICLE MAINTENANCE AND FLEET MANAGEMENT

- Adopt and enforce a structured vehicle maintenance program for Golder-owned vehicles.
- Maintain Vehicle Condition Check-out/Check-in list for Golder-owned vehicles.
- Prior to using any vehicle (company, private or rental), the driver should test the brakes, wipers, tires, lights and turn signals, check to see that the vehicle has an inflated spare tire and jack. Address any notes or oral warnings concerning vehicle deficiencies, which must be remedied at the earliest possible opportunity. If any safety concerns are identified, the vehicle must not be used.
- Vehicle deficiencies must be reported to the Office Manager as soon as they are noticed. The Office Manager, or his/her delegate, will arrange for maintenance of the vehicle.
- Golder-owned, rented or private vehicles used for on-site work may be required to be equipped with fire extinguishers and first-aid kits.
- Rented or client-provided vehicles must be in a roadworthy condition.

SAFETY PROGRAMS

- Teach workers strategies for recognizing and managing driver fatigue and in-vehicle distractions.
- Provide appropriate training to workers operating specialized motor vehicles or equipment.
- Emphasize to workers the need to follow safe driving practices on and off the job.
- Consider fire safety when parking vehicles in areas with dried grasses. Leaves or other plant material. Hot engine fluids, catalytic converters or other vehicle equipment could ignite dry plant material, causing a fire.

DRIVER PERFORMANCE

- Each driver of a vehicle being used on company business (company owned, private or hired) must possess a valid driver's licence that is appropriate for the type of vehicle to be driven.
- Check driving records of prospective employees, and perform periodic rechecks after hiring.
- Maintain complete and accurate records of workers' driving performance.

SECURING LOADS

Unsecured and poorly secured items inside or outside of a vehicle can be extremely dangerous if they are loose or become airborne. They can harm the vehicle driver and passenger, and/or occupants in vehicles behind you. The following recommendations should be followed:

- Use tie-down straps that are in good condition and rated for the load you will carry. Ratcheting tie downs are better than bungee cords or tie downs that just pull tight
- Install mounts to secure loads that you haul frequently in the same vehicle or trailer.
- Tarps covering loads should be snug and not flap.
- Check your load after you have driven a short distance to make sure it has not shifted.
- Do not pile items higher than the side walls of the truck bed or trailer.

VEHICLE SAFETY EQUIPMENT

You may not know when a highway emergency will happen, but you can be prepared by ensuring that your vehicle has essentials for dealing with roadside emergencies. Consider carrying items such as the following, and know how to use them properly:

- Flashlight
- Reflective safety vest
- Light sticks
- Fire extinguisher
- Tire inflator or sealant
- Reflective triangles or flares

DRIVIGN TECHNIQUES FOR 4-WHEEL DRIVING

Driving In Heavy Vegetation

- If you're unsure of the ground ahead, especially if there's mud or water, get out and check road conditions before proceeding.
- Position your hands on the steering wheel so that your thumbs are on the outside the steering wheel.
- Don't change transmission gears in the middle of a hazardous area, if in doubt always choose the lower gear.
- Tire pressures play an important part in off-road driving. Lowering tire pressures helps in getting through. 140-180 kPa (20-26 psi) is a good tyre pressure for soft tracks. If you choose to use a lower tire pressure, the vehicle must be operated at a lower speed. Remember to reinflate your tires as soon as you're back on hard ground.
- Cross small ridges 'square on' and cross ditches at a slight angle.
- If you begin to lose traction going uphill, or along a rutted track, or in mud, turn the steering wheel from side to side to maintain traction and move forward.

Driving On Steep Hills

- Low second or third gear is generally best for going uphill and low first gear is best for steep downhill
- Use the footbrake sparingly and with caution
- Avoid turning the vehicle sideways on a hill. If the vehicle begins to slide sideways, very slight acceleration and steering into the slide will normally straighten your descent.
- Allow any vehicle in front of your vehicle plenty of room
- If you stall going uphill, don't touch the clutch or accelerator.

Sand Driving

- Speed and flotation are the keys to success. High transmission gear ratio is best, if possible.
- Lower the tire pressure to 20psi. If you choose to use a lower tire pressure, the vehicle must be operated at a lower speed. Remember to reinflate your tires as soon as you're back on hard ground.
- Drive in existing wheel tracks if they are present.
- Avoid sudden changes in direction or acceleration. Coast to a stop if at all possible.
- Approach dunes head on.
- Avoid braking when descending a dune. Point the front of the vehicle downhill. Don't go fast, but not so slow that the wheels stop rolling, or the vehicle begins to slide sideways. A touch on the throttle will keep the wheels moving and the vehicle pointing in the right direction.
- If the vehicle gets stuck, try to rock the vehicle backwards or forwards, building up a small stretch of hardpack sand that you can accelerate from. Don't spin the wheels!
- Be sure that recovery gear is always in the vehicle in these driving conditions.
- Wash the vehicle after use.

Snow, Rain and Ice Driving

- Carry chains and install them on the tires when required.
- Prepare your vehicle and carry safety gear.
- Travel only on roads and tracks that are open to traffic.
- Drive with low beam lights on. Don't travel when visibility is poor.
- Vehicles travelling uphill in snow and ice conditions have right of way.
- Park only where directed and as close to the bank as possible. When parking, leave the vehicle in gear. Don't use the handbrake - it could freeze in the "on" position.
- Lift the wiper blades off the wind shield when leaving the vehicle parked.
- Watch for other travellers and animals and drive slowly in areas where they may be present. In the event that an animal is encountered on a road where driving conditions are poor due to the presence of snow, ice, or rain, do not over steer to avoid hitting the animal. The act of over steering may cause the vehicle to slide or roll. Most of the time the animal will move out of the road before the vehicle reaches it.
- Consider increasing the load or weight on the rear axle of front-wheel drive vehicles to improve traction when driving in snow, ice, or rain.

Mud

- Good tires with deep tread are helpful when driving in muddy conditions.
- Low second or third are probably the best gears for vehicle operation.
- Move the steering wheel from side to side rapidly to improve traction.

- Keep a steady pace.
- Stay out of ruts if possible.
- If you do become stuck, rock the vehicle backwards or forwards by alternating between first and reverse.

Fog/Limited Visibility

- Drive with low beam lights on. Don't travel when visibility is poor.
- Drive slowly and carefully.
- Pull over to safe location if you can't see vehicles in front or behind you until weather improves.

REGULATORY CITATION

There are no Federal OSHA regulations relating to driving safety. The Department of Transportation (DOT) Title 49 (Transportation) Subtitle VI (Motor Vehicle and Driver Programs) provides information about commercial motor vehicle operations.

WORKING ON OR OVER WATER

Over or near water work is undertaken by many different groups within the Golder for various activities including: barge drilling, geophysics, piling and inspection, dredging, aquatic sampling, and transport are some of the more common uses of over-water vessels, and this work can occur over lakes, rivers, and in marine environments.
Over water work introduces an additional level of danger to other procedure(s) that may be covered by other safe work procedures (SWPs). If this is the case, the health and safety issues discussed under that procedure's heading are also relevant, and should be referred to. As a result of the additional complication associated with over water work, increased levels of alertness, caution, and diligence are required.

If working on a boat, the crew MUST file a "FLOAT PLAN" with the appropriate office prior to departure (see last page of this SWP).

KEY HAZARDS

- Falling overboard, drowning
- Falls or slips associated with wet surfaces or rough water
- Overturning or capsizing due to improperly loaded or overloaded vessels
- Collisions with other vessels or debris
- Seasickness
- Weather Changes
- Hypothermia

PRECAUTIONS

- Only trained and qualified persons should operate the controls of boats or barges. For all work carried out over water, the owner, either Golder or sub-contractors, will be responsible for the safe operation, maintenance and general sea-worthiness of the vessel.
- Workers are required to wear an approved Personal Floatation Device (PFD) that is sufficiently buoyant to keep the workers head above water. This does not apply in situations where either the depth of water is too shallow to allow the PFD to function properly, or a sufficient fall protection system has been implemented to prevent falls into the water. Employees working over or near water, where the danger of drowning exists, shall be provided with U.S. Coast Guard-approved life jacket or buoyant work vests. PFDs should provide thermal insulation if work is in cold climate.
- All buoyancy and lifesaving equipment is to be marked with reflective material, including PFD's (should have whistle and/or light beacon if used on vessel), life rafts, lifeboats, and buoys. Prior to and after each use, the buoyant work vests or life preservers shall be inspected for defects which would alter their strength or buoyancy. Defective units shall not be used. If needed, ring buoys with at least 90 feet of Floating (nylon) line shall be provided and readily available for emergency rescue operations. Distance between ring buoys shall not exceed 200 feet.
- In addition, if needed, at least one lifesaving skiff shall be immediately available at locations where employees are working over or adjacent to water.
- If a boat is to be used for the transportation of workers, it is required to be conspicuously marked

with a safe maximum load, a safe maximum horsepower (if equipped with an outboard motor in excess of 10 HP), and that it be marked with a loading water line to make overloading apparent.

- Required safety equipment includes: first aid supplies, and readily available fire extinguisher(s).
- Deck lighting or spotlights/floodlights are to be used to ensure all work areas on board are sufficiently illuminated to enable work to be carried out safely.
- A radio or other method of communication with a land based station is required. If the vessel is in excess of 5.5 m (18 ft.) in length, an Emergency Position Indication Radio Beacon (EPIRB) is required for locating the vessel in the event of an accident.
- For stationary vessels (i.e. Barges), anchor points must be placed in a safe manner to prevent long anchor lines from being snagged and dragged by other vessels. All anchor points are to be well marked with buoys, and attachment points to shore are to be similarly well marked.
- Obtain as much site-specific information as possible concerning tidal/river conditions, known navigational hazards, and water depths. This is essential if you are operating the vessel and generally useful if you are working with a contractor owner/operator. Service clearance is also required, underwater/underground cables and pipelines may be present in the area.
- Use available and up to date tidal and marine charts, check weather forecasts and conditions for the area of interest, and determine what traffic may be present (barges, log booms, freighters, ferries, etc.). Verify weather, visibility and tidal conditions prior to embarking.
- The operator of a boat is responsible for operating his/her vessel within the safe maximum load, and any posted power limits or speeds. In addition, the operator is responsible for ensuring that all passengers are provided with an approved PFD.

MINIMUM PERSONAL PROTECTIVE EQUIPMENT REQUIRED

- High Visibility Personal Floatation Device (PFD) if required *

Filing a Float Plan

Before going out on a vessel it is always a good idea to leave a float plan with a local marina, relative or friend. A float plan should:

- Describe the vessel, its number, size, make, capacity, horsepower, and type of engine.
- List where you are going, the route, and your expected return time.
- Give the number of passengers, their names and addresses, and a contact in case of an emergency.

1. Name of person reporting and telephone number
   _____
   (_____) _____
2. Description of boat
   Type_____ Color_____ Trim_____
   Registration No._____ Length_____
   Name _____ Make_____ Other _____
3. Engine type_____ H. P._____
   No. of engines _____ Fuel capacity_____
4. Survival equipment ❑ PFDs ❑ Paddles ❑ Smoke signals ❑ Anchor
5. Radio ❑ Yes ❑ No Type _____ Frequency _____
6. Mobile phone ❑ Yes ❑ No (_____) _____
7. Automobile license number _____
   Type_____ Trailer license_____
   Color_____ Make of auto_____
   Where parked_____
8. Persons on board
   Name Age Address & Telephone
   _____
   _____
   _____
   _____
9. Do any of the persons on board have a medical problem?
   ❑ Yes ❑ No If yes, what?_____
10. Trip expectations. Leave at_____ ❑ am ❑ pm
    From _____ going to_____

    Expect to return by (time) _____ ❑ am ❑ pm and
    not later than _____ ❑ am ❑ pm
11. Any other pertinent information?_____
    _____
12. If not returned by (time) call the Coast Guard or (local authority) _____ ❑ am ❑ pm
13. Telephone numbers
    (_____) _____ (_____) _____

ATTACHMENT 6

ELECTROSHOCKING PROCEDURES

Electrofishing

The following safety protocol is intended for persons using a battery-powered or a gasoline-powered backpack electrofisher unit for the purposes of capturing fish in a stream. As electrofishing involves the use of electricity in flowing water and walking over a variety of potentially unstable substrates, it is important to recognize and be aware of potential hazards associated with this activity. The Fisheries Safety Handbook (December 2008) published by the American Fisheries Society should be reviewed by sampling personnel for additional safety information.

Key hazards include:

- Electrocution;
- Fast flowing water;
- Trips, slips and falls;
- Drowning;
- Unstable footing;
- Heat stroke, dehydration, hypothermia, insects bites / stings;
- Traversing uneven ground and vegetated areas, to access a Site;
- Wildlife;
- Working in remote locations; and
- Working in the vicinity of industrial activities such as logging operations.

Precautions

- Site-specific information (e.g. ground conditions, stream conditions and access) MUST be obtained prior to going into the field to ensure that electrofishing activities can be conducted in a safe manner. Ask the Project Manager and, if possible, the Client or Client Contact for information.
- The minimum crew size consists of two people who are both certified to electrofish. Each member of the electrofishing crew must also have a valid Level 1 First Aid certificate.
- Crew members with a heart condition or any other relevant medical condition that may restrict physical activity should not engage in electrofishing activities without their doctor's approval.
- Crewmembers are to inform the crew supervisor of any allergies they have. A list of crewmember's allergies is to be included in First Aid kits. If, for example, a crew member is severely allergic to bee stings, an anaphylactic kit should be included in the First Aid kit and carried by the person with the allergy. The partner of the crew member with the allergy should familiarize themselves with the use of the anaphylactic kit.
- Before electrofishing can commence, there must be an effective means (reliable and timely) of communication between the site personnel and medical assistance, as well as an effective means of conveying an injured person to the nearest hospital.
- Before entering a stream, survey the reach where electrofishing will occur and identify potential sampling locations. Consider access requirements such as entry and exit points, obstacles such as pools, fast flowing water and logjams.
- Personal floatation devices MUST be worn by all crew members where there is a risk of drowning.
- NEVER wade into water deeper than you are comfortable with, given the stream flow and footing at a site. Know your limits.
- The MAXIMUM wading depth for the person carrying the electrofisher is reached just before the water touches the bottom of the battery holder.
- Electrofishing equipment must be operated and maintained in accordance with the manufacturer's instructions.

- The equipment electrofishing must not be used if it has been submersed in water or damaged.
- Electrode and dip net handles must be constructed of insulating material such as wood or fiberglass
- Ensure that metal items such as pipes, wires or fences are not present where electrofishing activities will occur.
- Ensure that bystanders maintain a safe distance from electrofishing activities. This is a minimum of 30 feet if they are in the water and at least 10 feet if they are on land. Electrofishing should stop if bystanders are too close or will not move.
- Make sure the all members of the crew know where you are at all times and vice versa.
- NEVER turn the power switch on until all crew members have signaled that they are ready.
- Turn the electro fisher's power off when not in use.
- Disconnect the battery from the electrofisher when not in use, or when moving between sites.
- Electrofishing crews should take regular breaks as needed.
- A crew member receiving an electrical shock MUST be transported to a hospital if: unconsciousness results; the crew member appears to be disoriented in any way; specified by the first aid attendant; the individual feels uncomfortable in any way; or the crew member so requests.

Minimum Personal Protective Equipment Required

- Waterproof, leak-free chest waders with slip resistant soles and a wading belt.
- Waterproof, leak-free elbow-length neoprene gloves with a valid 5,000-volt (minimum) rating.
- Polarized sunglasses
- Hat with a brim
- An approved personal flotation device when there is a risk of drowning

Applicable OSHA Regulation Parts

Further information can be found on electrofishing in the General Health and Safety Standards, WAC 296-24-956 Part L, Electrical.

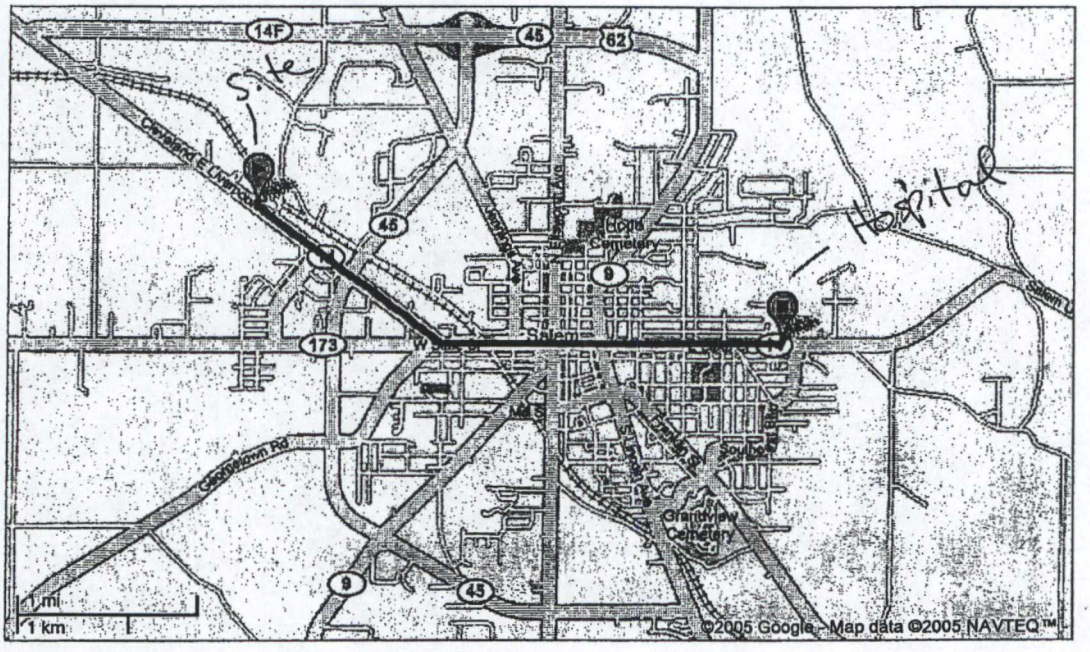
ATTACHMENT 7

LOCAL HOSPITAL ROUTE MAPS

Google Local

Directions

Start address: **1199 benton road, Salem, OH 44460**
End address:
Salem Community Hospital



Start address: 1199 benton road
Salem, OH 44460
End address: **Salem Community Hospital**
2094 E State St
Salem, OH 44460
Distance: 3.2 mi (about 6 mins)

1. Head **southeast** from **OH-14** - go **1.3 mi**
2. Bear **left** at **W State St** - go **0.6 mi**
3. Continue on **E State St** - go **1.3 mi**

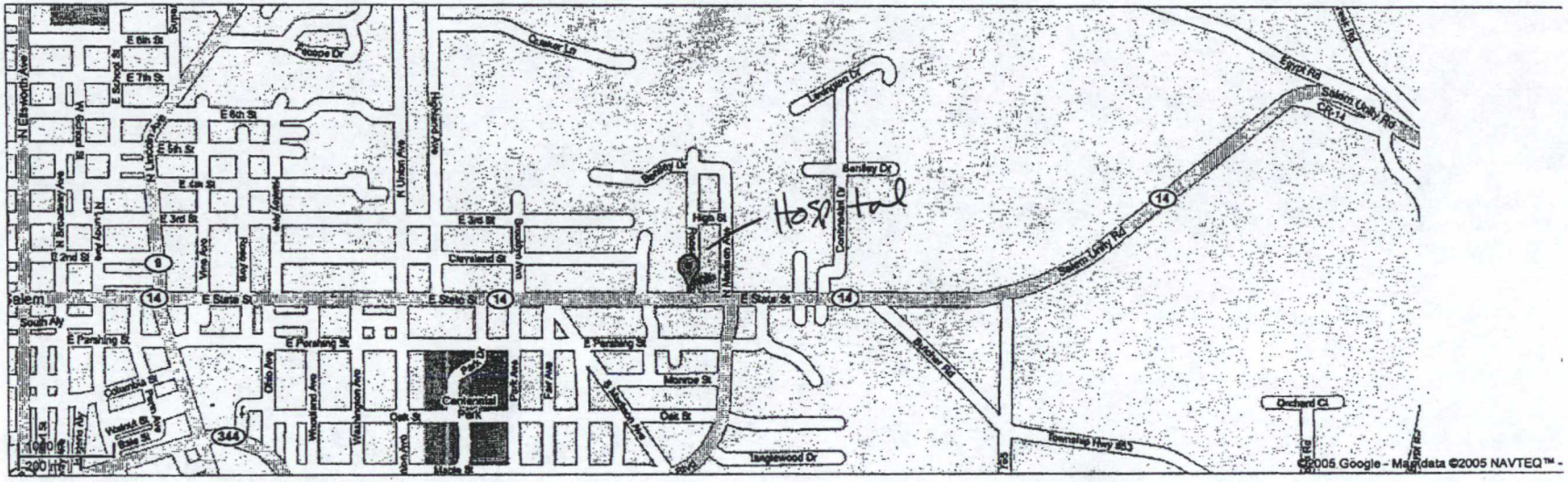
These directions are for planning purposes only. You may find that construction projects, traffic, or other events may cause road conditions to differ from the map results.

Map data ©2005 NAVTEQ™, Tele Atlas

Google Local

Directions

Start address: **1199 benton road, Salem, OH 44460**
End address:
Salem Community Hospital



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Salem, OH 44460

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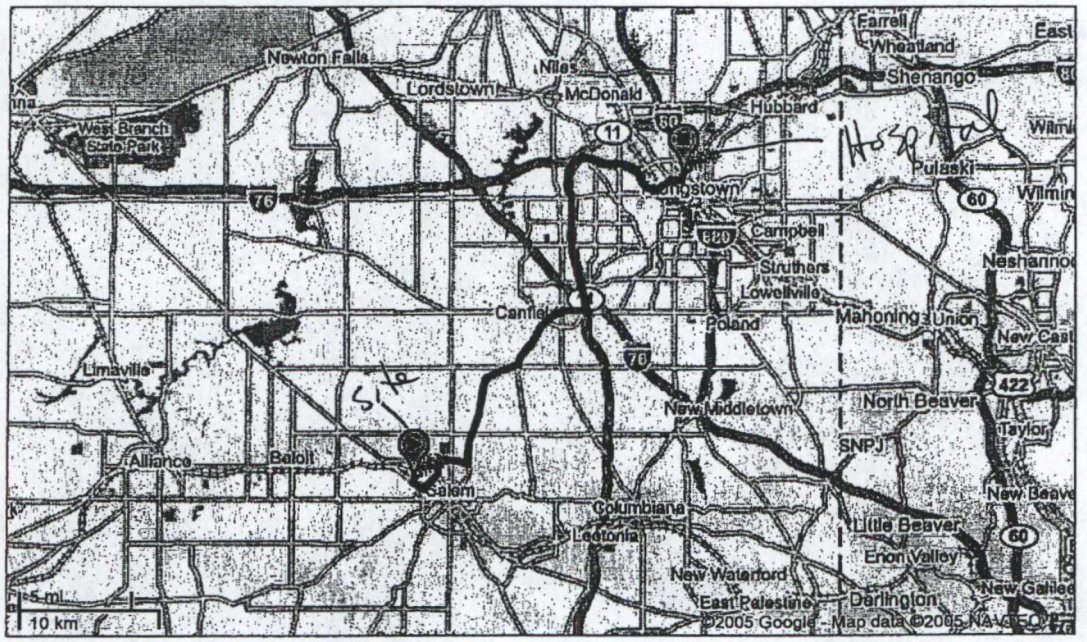
These directions are for planning purposes only. You may find that construction projects, traffic, or other events may cause road conditions to differ from the map results.

Map data ©2005 NAVTEQ™, Tele Atlas

Directions

Start address: **1199 benton road, Salem, OH 44460**
End address:
St Elizabeth Health Center



Start address: 1199 benton road
Salem, OH 44460

End address: **St Elizabeth Health Center**
1044 Belmont Ave
Youngstown, OH 44504

Distance: 27.1 mi (about 42 mins)

1. Head **southeast** from **OH-14** - go **0.6 mi**
2. Turn **left** at **US-62** - go **1.5 mi**
3. Turn **right** onto the **OH-45 N/US-62 E** ramp - go **0.3 mi**
4. Continue on **US-62 E** - go **1.1 mi**
5. Turn **left** at **US-62** - go **0.4 mi**
6. Continue on **Youngstown Salem Rd** - go **7.6 mi**
7. Continue on **Lisbon St** - go **0.8 mi**
8. Turn **left** at **S Broad St** - go **0.1 mi**
9. Turn **right** at **E Main St** - go **1.2 mi**
10. Bear **right** into the **OH-11 N** entry ramp - go **7.3 mi**
11. Take the **I-680** ramp to **Youngstown** - go **0.4 mi**
12. Merge into **I-680 S** - go **4.1 mi**
13. Take the **OH-193** exit **4A** to **New Castle (US-422)** - go **0.2 mi**
14. Continue on **OH-193 N** - go **0.6 mi**

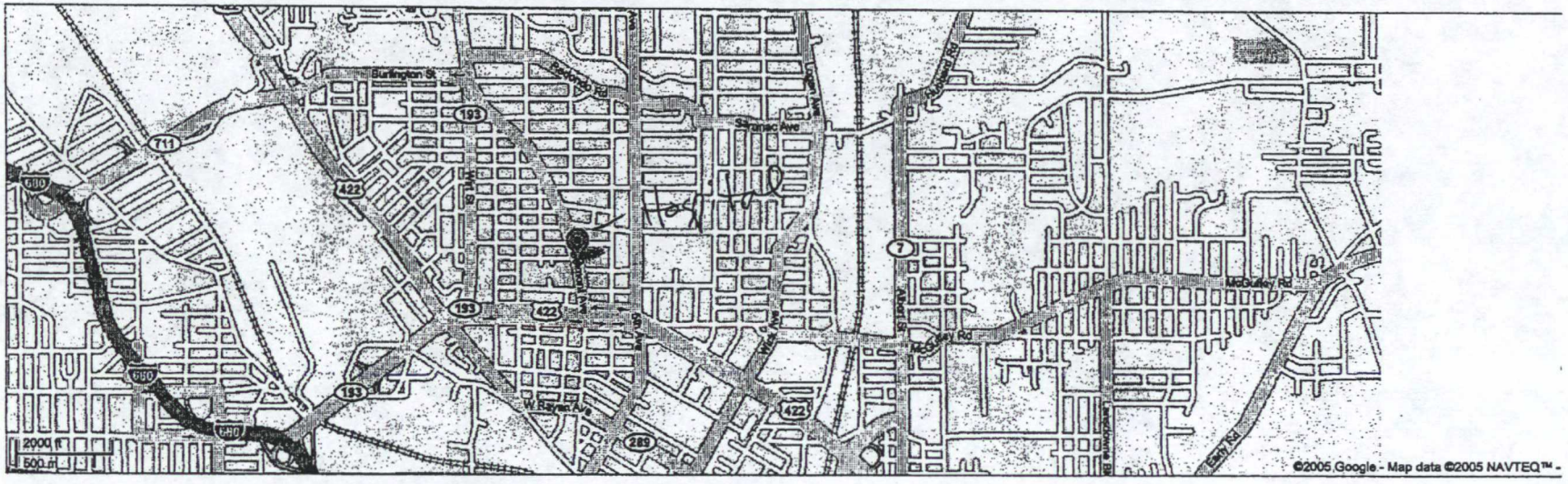
15. Take the **OH-193 N/OH-289/US-422 W** ramp - go **0.2 mi**
16. Bear **right** at **Madison Ave** and head toward **Belmont Ave** - go **0.5 mi**
17. Turn **left** at **Belmont Ave** - go **0.2 mi**

These directions are for planning purposes only. You may find that construction projects, traffic, or other events may cause road conditions to differ from the map results.

Map data ©2005 NAVTEQ™, Tele Atlas

Directions

Start address: **1199 benton road, Salem, OH 44460**
End address:
St Elizabeth Health Center



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Salem, OH 44460

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1044 Belmont Ave
Youngstown, OH 44504

Distance: 27.1 mi (about 42 mins)

1. Head **southeast** from **OH-14** - go **0.6 mi**
2. Turn **left** at **US-62** - go **1.5 mi**
3. Turn **right** onto the **OH-45 N/US-62 E** ramp - go **0.3 mi**
4. Continue on **US-62 E** - go **1.1 mi**
5. Turn **left** at **US-62** - go **0.4 mi**
6. Continue on **Youngstown Salem Rd** - go **7.6 mi**

ATTACHMENT 8

INCIDENT REPORT FORM

DATE:	
EMPLOYEE:	EMPLOYEE NUMBER:
PROJECT MANAGER:	
PROJECT NAME:	PROJECT NUMBER:
OFFICE LOCATION:	
LOCATION OF INCIDENT:	
DATE:	TIME:
DESCRIPTION:	
CAUSE OF INCIDENT:	
PORTION OF BODY INJURED/INVOLVED: ! HEAD ! NECK ! SHOULDER ! ARM ! CHEST ! BACK ! LEG ! FOOT ! HAND ! OTHER_____	
TYPE OF HAZARD: ! PHYSICAL ! CHEMICAL ! BIOLOGICAL	
EXPLAIN HAZARD EXPOSURE:	
TREATMENT: ! FIRST-AID ! CPR ! MEDICAL ATTENTION (DOCTOR, HOSPTIAL) ! OTHER_____	
LOST TIME: ! _____	! NONE
ADDITIONAL COMMENTS:	
OFFICE HEALTH AND SAFETY OFFICER:	

APPENDIX F

APPROVED RI QAPP

Volume 2: Quality Assurance Project Plan
Ruetgers-Nease Salem, Ohio Site

Revision 4: February 28, 1990
Revision 3: December 13, 1989
Revision 2: August 3, 1989
Revision 1: October 22, 1988

Submitted to

United States Environmental Protection Agency, Region 5
Hazardous Waste Enforcement Branch
230 S. Dearborn St.
Chicago, Illinois 60604

Ohio Environmental Protection Agency
Corrective Actions Section
Division of Solid and Hazardous Waste Management
1800 Watermark Dr.
Columbus, Ohio 43266

Northeast District Office
Division of Solid and Hazardous Waste Management
2110 Aurora Rd.
Twinsburg, Ohio 44087

Submitted by

Ruetgers-Nease
Chemical Company, Inc.
201 Struble Rd.
State College, Pennsylvania 16801

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LIST OF ATTACHMENTS

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Attachment #2 Enseco's SOP for the Analysis of TCL, VOA, BNA and Pesticide/PCBs with Low Detection Limits in Drinking Water

3.0 PROJECT DESCRIPTION

This Quality Assurance Project Plan (QAPP) sets forth procedures to assure the quality, quantity, and validity of data collected during the RI/FS at the Ruetgers-Nease Chemical Company (Ruetgers-Nease), Salem, Ohio Site (the Site). This section of the QAPP describes the purpose of the plan and provides background information on site conditions.

This QAPP presents the organization, objectives, functional activities, and specific sampling and analysis quality assurance (QA) and quality control (QC) procedures under which the remedial investigation at the Site will be performed. The QAPP is designed to achieve all the specific technical and data quality objectives and goals needed to identify characterize, and fully delineate the fact, nature, extent and magnitude of contaminants related to the Site in on-site and off-site locations.

3.1 Site Background and History

The purpose of providing background information is to educate RI/FS personnel regarding present site conditions and planned actions so that the QA/QC program outlined in this document can be understood.

3.1.1 Location

The Site is located approximately one mile northwest of the City of Salem (see Figure 3-1). Conrail railroad tracks separate the Site into two unequal sections that total approximately 44 acres. The Site is bounded by small light-industrial operations along Allen Road to the east, residences to the immediate southwest, State Route 14A to the south, and wooded areas and pasture lands to the north. Site

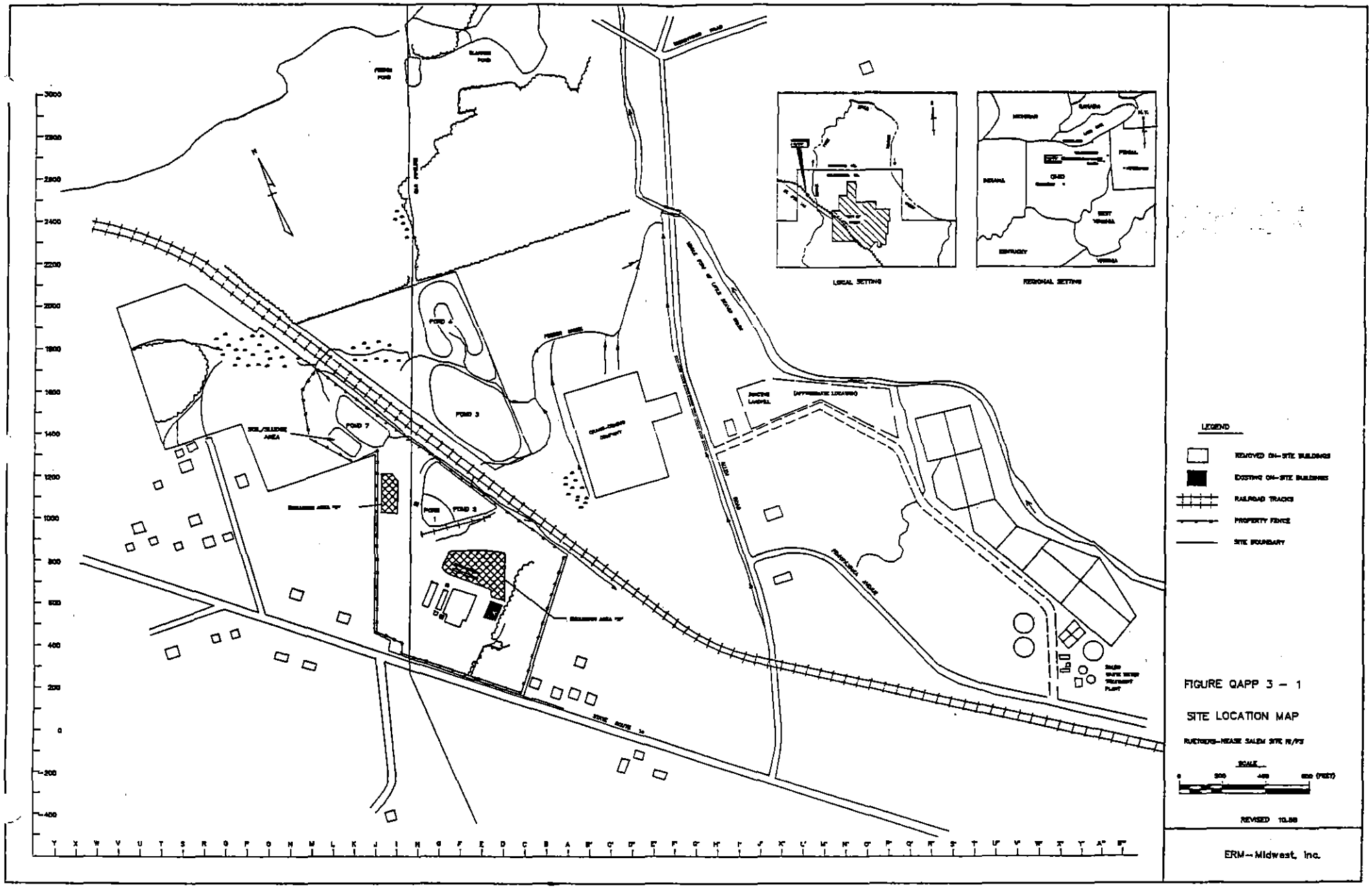

FIGURE QAPP 3 - 1

SITE LOCATION MAP

RUETGERS-NEASE SALEM SITE RI/FS

stormwater drains in a northeasterly direction to the main surface water body in the area, the Middle Fork Little Beaver Creek (MFLBC), which flows northward and then southward to Little Beaver Creek which eventually flows into the Ohio River. There is an inactive landfill located approximately 1,200 feet east of the site along the west bank of the MFLBC. This was operated as a dump and extends from the MFLBC to the east side of Allen Road. The area is presently covered with vegetation and construction rubble.

The area's potable water supply is provided by both a public water system and private wells. The small businesses along Allen Road receive drinking water from the City of Salem, although Dunlap Disposal uses well water for non-potable purposes. Residents along State Route 14A and further north on Allen Road and Goshen Road use either public water or private wells. The City of Salem has a reservoir which draws water from Cold Run Creek, approximately seven miles south of the Site.

The area is underlain by glacial deposits of the Kent Moraine, a five to fifteen mile wide belt of nonhomogeneous glacial drift. Approximately 10 to 25 feet of drift underlies the Site (Figure 3-2). This drift consists mostly of silty or sandy, gray-blue, plastic clay with some pebbles and boulders. Sand and gravel deposits within the drift have permeabilities higher than that of the silty or sandy clays. The drift is underlain by sedimentary rocks consisting mainly of interbedded sandstones, shales and coal seams.

Prior to drift deposition, glacial forces eroded a valley into the sedimentary rocks east of the Site. This eroded valley was filled as drift was deposited.

The distinct aquifers within the study area have been named the Shallow, Interface, Upper Bedrock, Lower Bedrock

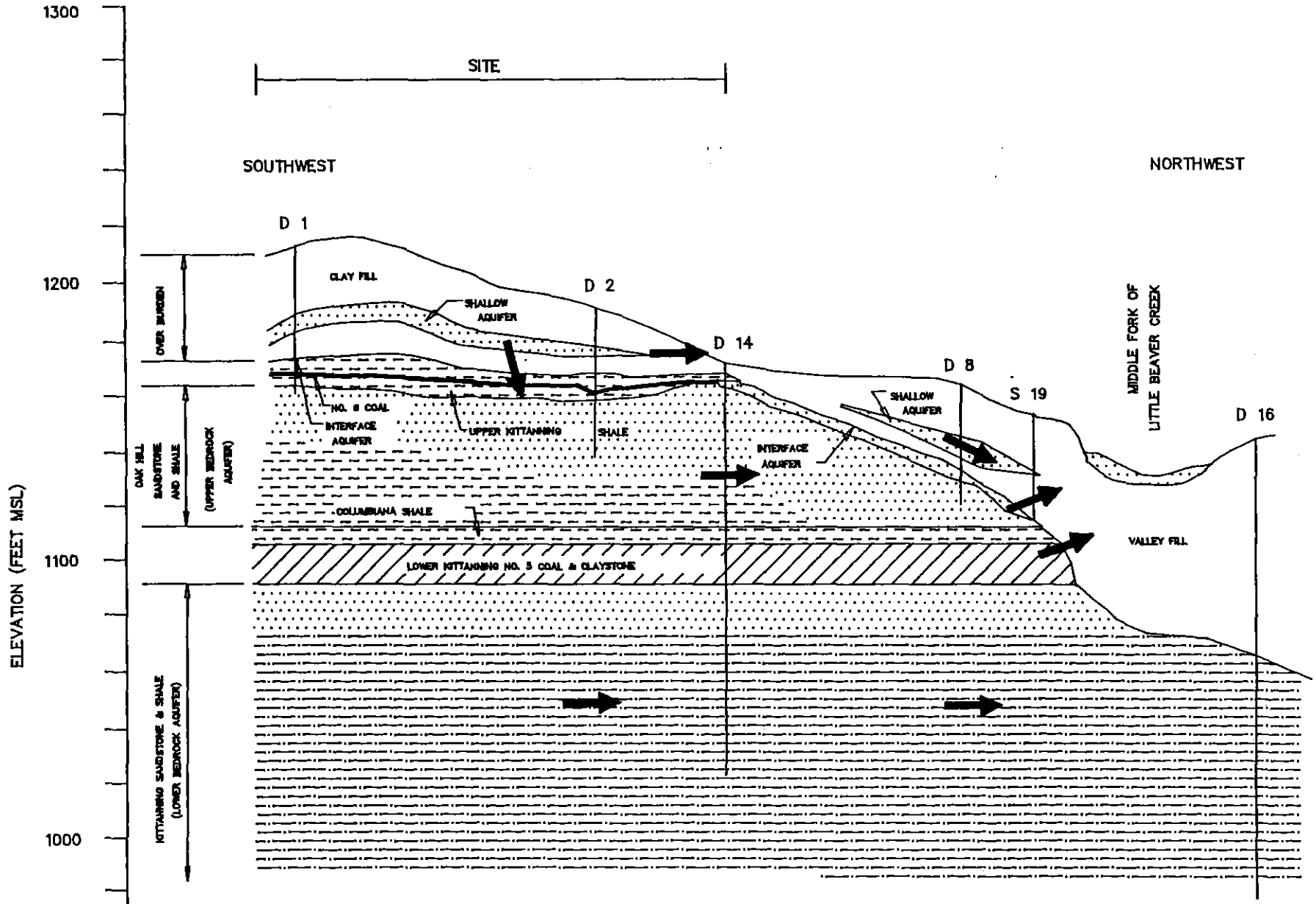

FIGURE QAAP 3 – 2

STRATIGRAPHIC AND HYDROGEOLOGIC

CROSS SECTION

RUETGERS–NEASE SALEM SITE RI/FS

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DIRECTION OF GROUND WATER FLOW

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and Valley Fill. Both confined and unconfined conditions are found in the area. Ground water beneath the Site apparently is moving in the general direction of the MFLBC. Hydrogeologic conditions near the MFLBC and within the Valley Fill have not been fully delineated.

3.1.2 History

From 1961 until 1973, Nease Chemical Company produced chemicals such as household cleaning compounds, pesticides, fire retardants, and chemical intermediates at the Site. Products and chemical intermediates were produced in batch processes.

Nease's waste handling facilities included air scrubbers and a multiple pond/settling tank system for neutralization and treatment of acidic waste. Five unlined lagoons (1, 2, 3, 4, and 7) were used for treatment and storage of either acidic waste or lime slurries from waste neutralization. In 1969, a pipeline was constructed to carry neutralized wastewater to the Salem Wastewater Treatment Plant. Some 55-gallon drums containing wastes were buried on-site in Exclusion Area A.

Following notification from OEPA of wastewater violations, Nease Chemical Company agreed in a Consent Judgment in 1973, to discontinue manufacturing operations at the Site until such time as a new wastewater permit was obtained. Instead, Nease decided to close the facility. Pond water was neutralized and removed to the Salem Wastewater Treatment Plant. Nease also filled/graded several ponds, and removed all production facilities with the exception of a warehouse and two small block buildings. On December 30, 1977, Nease merged with Ruetgers Chemicals, Inc. to form Ruetgers-Nease Chemical Company, Inc.

Since 1982, various environmental investigations and remedial actions have been conducted by Ruetgers-Nease at the Site. The objectives of these studies were to define hydrogeological conditions, identify potential migration of contaminants, and evaluate remedies. In 1983, the Site was placed on the National Priorities List (NPL). Investigation activities conducted by Ruetgers-Nease to date have included:

1. Soil borings and test pit excavations to characterize on-site contamination

2. Soil borings through ponds to characterize contaminant concentrations and quantities

3. Geophysical surveys at Exclusion Areas A and B to locate drum burial areas

4. Sediment and surface water sampling and analysis to identify contaminant migration and extent. Feeder Creek, the Crane-Deming Swamp, Slanker Pond, and MFLBC surface water and sediments have been sampled. In addition, U.S. EPA and OEPA have sampled fish from MFLBC and Slanker Pond

5. Air monitoring to evaluate on-site and off-site atmospheric releases

6. Installing and sampling monitoring wells and sampling nearby residential wells. At present, there are 38 monitoring wells at the Site, including:

   - Fourteen Shallow Aquifer wells

   - Six Interface Aquifer wells

- Fourteen Upper Bedrock Aquifer wells
- Four Lower Bedrock Aquifer wells

Three of these wells are installed into or through the Valley Fill.

7. Conducting a Risk Assessment in 1986
8. Completing a site grid system based on two sets of perpendicular lines with adjacent lines 100 feet apart

Table 3-1 lists compounds qualitatively identified at the Site. Most analyses were targeted to volatile and other organic compounds handled on-site. Several priority pollutant scans have been completed.

Remedial actions taken to date include:

1. Removing 115 buried drums from Exclusion Area A
2. Excavating and removing contaminated soils including approximately:
   - 5,400 $yd.^3$ from Exclusion Area A
   - 684 $yd.^3$ from Exclusion Area B
   - 2,790 $yd.^3$ from Pond 1
   - 630 $yd.^3$ from the ditch paralleling the south side of the railroad tracks
3. Seeding of Pond 2

QAPP TABLE 3-1

COMPOUNDS THAT HAVE BEEN QUALITATIVELY IDENTIFIED AT THE RUETGERS-NEASE SALEM SITE PRIOR TO THE RI/FS

Compounds	Range of MDLs Solid	Range of MDLs Aqueous
1,1-Dichloroethene	50-100 ug/kg[1]	0.5-1.0 ug/L[1]
1,2-Dichloroethene	50-100 ug/kg[1]	0.5-1.0 ug/L[1]
Chloroform	50-100 ug/kg[1]	0.5-1.0 ug/L[1]
1,2-Dichloroethane	50-100 ug/kg[1]	0.5-1.0 ug/L[1]
1,1,1-Trichloroethane	50-100 ug/kg[1]	0.5-1.0 ug/L[1]
1,2-Dichloropropane	50-100 ug/kg[1]	0.5-1.0 ug/L[1]
1,3-Dichloropropene	50-100 ug/kg[1]	0.5-1.0 ug/L[1]
Trichloroethene	50-100 ug/kg[1]	0.5-1.0 ug/L[1]
Benzene	50-100 ug/kg[1]	0.5-1.0 ug/L[1]
Tetrachloroethene	50-100 ug/kg[1]	0.5-1.0 ug/L[1]
1,1,2,2-Tetrachloroethane	50-100 ug/kg[1]	0.5-1.0 ug/L[1]
Toluene	50-100 ug/kg[1]	0.5-1.0 ug/L[1]
Chlorobenzene	50-100 ug/kg[1]	0.5-1.0 ug/L[1]
Ethylbenzene	50-100 ug/kg[1]	0.5-1.0 ug/L[1]
o,m,p-Xylene	50-100 ug/kg[1]	0.5-1.0 ug/L[1]
1,3+1,2-Dichlorobenzene	50-100 ug/kg[1]	0.5-1.0 ug/L[1]
Methoxychlor	50 ug/kg[2,3]	0.005 ug/L[2]-0.05 ug/L[3]
Mirex	50 ug/kg[2,3]	0.005 ug/L[2]-0.05 ug/L[3]
3,4-Dichloronitrobenzene	500 ug/kg[3]-1000 ug/kg[1]	50 ug/L[3]-200 ug/L[1]
Diphenyl Sulfone	500 ug/kg[3]-1,000 ug/kg[1]	50 ug/L[3]-200 ug/L[1]

NOTES

MDL - Method Detection Limit

[1]By GC/FID

[2]By GC/ECD

[3]By GC/MS(SIM)

4. Installing geotextile fabric barriers and rock dams across drainage swales and ditches

5. Installing hay-bale barriers around the exclusion areas

6. Completing a leachate collection system between the railroad tracks and Exclusion Area A. Leachate is collected on a regular basis and disposed at a permitted off-site wastewater treatment facility.

7. Installed a fence surrounding the western portion of the Site.

These actions were taken by Ruetgers-Nease.

3.1.3 Existing Conditions

Currently, the Site contains a single-story warehouse; two small concrete-block buildings; concrete pads, foundations, and tile floors remaining from the manufacturing facilities; concrete tank saddles; and the pond areas. The Site west of the Conrail tracks is surrounded by a fence with access from Route 14. Much of the Site has been revegetated by weeds and grasses.

The banks of Pond 1 slope steeply to the water surface, which is about eight to ten feet below grade. The surface of Pond 2 contains isolated patches of grasses and weeds surrounded by barren areas. The surfaces of Pond 3, 4 and 7 consist of weeds, grasses, small shrubs and some small trees. A soil/sludge pile west of Pond 1 is covered by weeds, grasses, shrubs and small trees, and is approximately five to eight feet above the grade. The surfaces of Ponds 3 and 7 may not support heavy equipment.

The remainder of the study area contains large fields, stands of trees, plowed fields, houses and buildings. The Crane-Deming swamp contains areas of bare soils, and wetland grasses. Trees and dense vegetation border the MFLBC.

3.2 Problem Statement

Previous studies have identified sources on-site and have indicated that contaminant migration off-site may be occurring. This RI approach has been developed, in part, to identify all sources, characterize contaminants, and determine the limits of contaminant migration. A description of known sources, affected media, and the rationale for sampling are provided in the following sections.

3.2.1 Sources

Potential sources of contamination at the Site are listed in Table 3-2. The actual contribution of sources to affected media has not been quantified. The complete set of contaminants migrating from these sources has not been confirmed by samples meeting U.S. EPA QA-QC guidelines.

3.2.2 Affected Media

Previous studies have identified contaminants in various media. Media affected or potentially affected are listed on Table 3-3.

3.3 Project Objectives

The project objectives described in this section define the purpose of the Remedial Investigation/Feasibility Study

QAPP TABLE 3-2

POTENTIAL CONTAMINANT SOURCES(1)
RUETGERS-NEASE SALEM SITE RI/FS

Source	Characterization
Exclusion Areas A and B	Some buried drums and contaminated soils (removed) contained volatile and non-volatile organics.
Pond 1, 2	Disposal of treated process waters containing volatile organic compounds in non-secure/unlined areas (some Pond 1 soil removed).
Ponds 3,4,7	Contaminants, if present in soil/sludge stockpile neutralized calcium sulfate sludge, may migrate through unlined pond bottoms or in surface runoff.
Manufacturing areas, on-site surface and subsurface soils	Possible chemical spills may have contaminated soils.

(1) Based on sampling by Ruetgers-Nease performed to date. Confirming and characterizing sources is an objective of the RI.

QAPP TABLE 3-3

AFFECTED OR POTENTIALLY AFFECTED MEDIA
RUETGERS-NEASE SALEM RI/FS

Media	Location	Potential Contaminants
Surface Water	Feeder Creek(1)	Volatile Organics Non-Volatile Organics Additional Organics
	MFLBC(1)	Volatile Organics Non-Volatile Organics Additional Organics
Sediments	Feeder Creek(1)	Volatile Organics Non-Volatile Organics Additional Organics
	MFLBC(1)	Volatile Organics Non-Volatile Organics Additional Organics
	Slanker Pond(1)	Non-Volatile Organics Additional Organics
	Crane-Deming(1) Swamp	Volatile Organics Non-Volatile Organics Additional Organics
Fish	MFLBC(2)	Non-Volatile Organics Additional Organics
Soils	On-Site(1)	Volatile Organics Non-Volatile Organics Additional Organics
Sludges	Salem Wastewater Treatment Plant Sludge Cells 4, 6 and 8 (2)	Volatile Organics Non-Volatile Organics Additional Organics

(1) Based on sampling by Ruetgers-Nease performed to date.
(2) Ruetgers-Nease has not sampled these locations.
(3) This aquifer may, or may not exist down gradient of the site.

*Non-Volatile Organics are defined here and throughout the document as TCL BNA +25, TCL pesticides/PCBs

QAPP TABLE 3-3 (Cont'd)

AFFECTED OR POTENTIALLY AFFECTED MEDIA
RUETGERS-NEASE SALEM RI/FS

Media	Location	Potential Contaminants
Ground Water	Shallow(1) Aquifer	Volatile Organics Non-Volatile Organics Additional Organics
	Interface(1) Aquifer	Volatile Organics Non-Volatile Organics Additional Organics
	Upper Bedrock(1) Aquifer	Volatile Organics Non-Volatile Organics Additional Organics
	Lower Bedrock(1) Aquifer	Volatile Organics Non-Volatile Organics Additional Organics
	Valley Fill(2)(3) Aquifer	Volatile Organics Non-Volatile Organics Additional Organics

(1) Based on sampling by Ruetgers-Nease performed to date.
(2) Ruetgers-Nease has not sampled these locations.
(3) This aquifer may, or may not exist down gradient of the site.

*Non-Volatile Organics are defined here and throughout the document as TCL BNA +25, TCL pesticides/PCBs

(RI/FS) being conducted. The objectives of the RI/FS are to gather data of adequate technical content, quality and quantity to:

- Determine fully the fact, nature, extent and magnitude of contamination on and off the Site.

- Determine if contaminants relating to the Ruetgers-Nease Site pose a threat to human health or the environment through the development of an Endangerment Assessment.

- Fully identify and characterize the source, migration pathways, routes of entry and receptors for contaminants.

- Support the identification, development and evaluation of remedial alternatives during the FS, an Endangerment Assessment, remedial technology screening, alternative development and screening, and detailed alternative evaluation.

The project data quality objectives for analytical laboratory data and field measurements are presented in Section 5 of this QAPP.

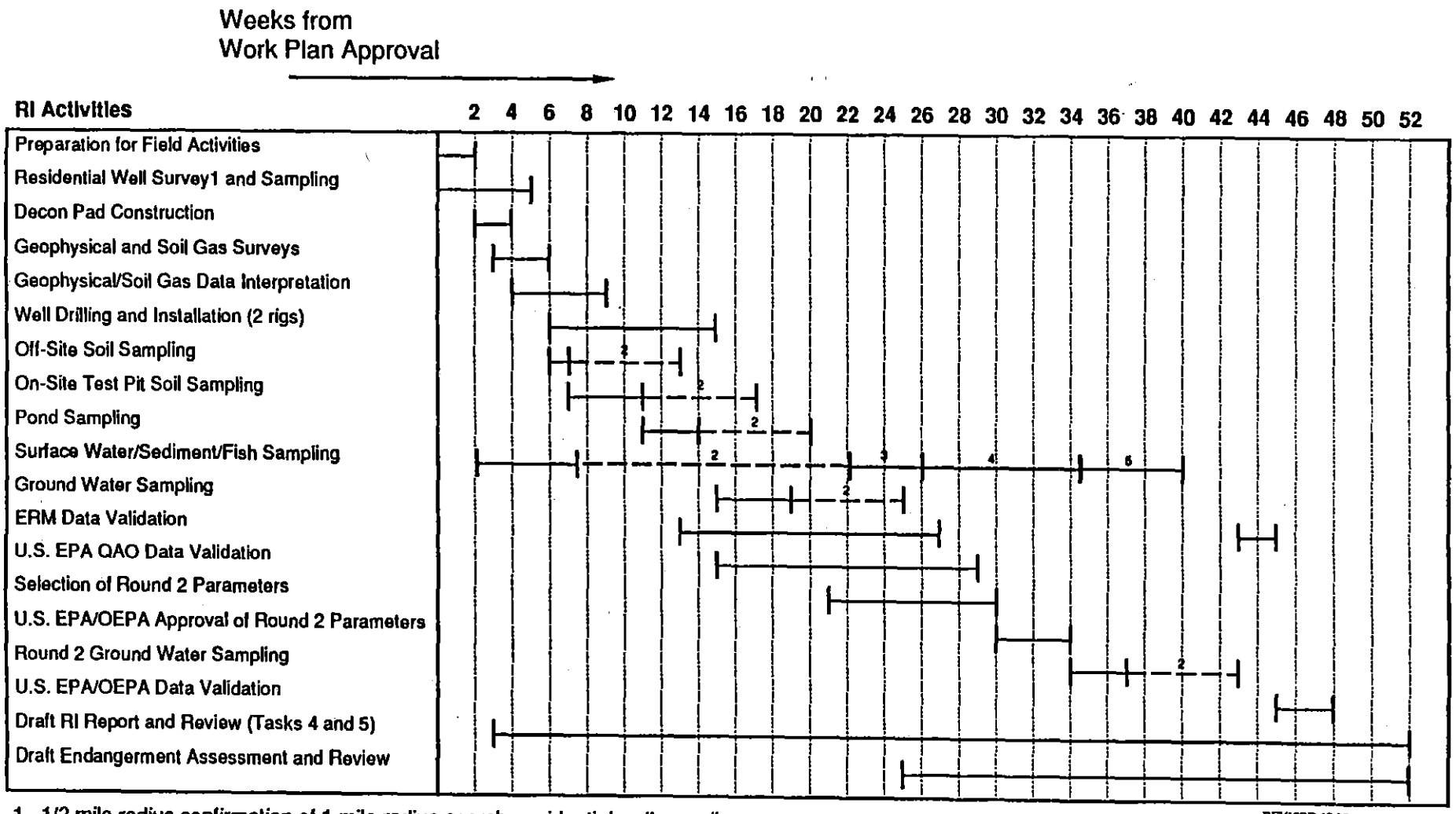
3.4 Time Schedule

3.4.1 ERM's Time Schedule

The time schedule for the implementation of RI activities is presented in Figure 3-3.

FIGURE QAPP 3-3

Schedule for Implementation of RI Activities at the Ruetgers-Nease Salem Site RI/FS



1. 1/2 mile radius confirmation of 1 mile radius search, residential well sampling
2. Laboratory analysis time
3. ERM data validation for surface water/sediment/fish sample analyses
4. U.S. EPA QAO data validation for surface water/sediment/fish sample analyses
5. Preparation of technical memorandum concerning surface water/sediment/fish sample analytical results

NOTE: Start-up and completion dates may change depending on field conditions

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3.4.2 Enseco's Time Schedule

Sample delivery groups will be organized into "Cases". One Case will consist of all samples received at Enseco Laboratory within a one week period.

Data turnaround time will be four weeks from the date on which the last sample in the Case is received.

3.5 Intended Data Use

The data collected during the RI will be used to meet the project objectives specified in Section 3.3. Soil, sediment, surface water, ground water, fish and air sample data will be used to evaluate the current public health risks related to the site in the Endangerment Assessment. The data will be carefully selected and applied to assure that data used for this purpose is of suitable quality.

At a minimum, all sampling and off-site analysis data will be consistent with EPA's CLP (Contract Laboratory Program) specifications CLP SOW-7/87. QA objectives (accuracy, precision, sensitivity), which are more stringent than CLP, are required for the interpretation of data representing ground water and surface water for those samples specified as critical. More sensitive methods will be used to analyze fish and air matrices for organic constituents in an attempt to meet levels of health-based criteria.

In addition to the off-site analysis, there will also be on-site field measurements performed utilizing HNU, OVA and pH meters. The OVA and HNU data will be used for health and safety purposes by determining the levels of volatile organic constituents in and around areas of the site. The OVA and HNU data will also be used as part of the soil gas survey to

semi-quantitatively determine the levels of volatile organic constituents present in subsurface soils. This screening will be utilized as an aid in the selectional process as to which samples will be submitted for off-site analyses. This selection process will be based upon a comparison of gross instrument responses. The pH data will be used as a field indicator of groundwater quality (as compared to background sample pH readings), and for determining the stability of groundwater with respect to a properly purged groundwater system. This determination will be based upon a comparison of pH readings during the purging process. The pH data for surface water will be used as an indication of the ambient water quality as it pertains to supporting freshwater aquatic life.

A majority of the samples collected at the Site will be analyzed to produce data of sufficient quality to evaluate the extent of contamination. For this application, CLP procedures and QA objectives are sufficient. When CLP criteria are not met due to a variety of sample matrix related reasons, the data will be carefully evaluated to determine if certain aspects of this data can still be used to meet the project objectives.

3.6 Sample Network and Rationale

Sample locations and analysis have been identified that will provide data necessary to meet the objectives of the RI/FS and to complete EA activities. The sample network rationale collection method and type of information needed are listed on Table 3-4. Samples considered critical to achieving project objectives, and a summary of activities to ensure these samples meet QA/QC criteria are listed on Table 3-5.

QAPP TABLE 3-4

SAMPLING RATIONALE
RUETGERS-NEASE SALEM SITE RI/FS

Information Needed	Rationale	Data Gathering Methods
1. Characteristics of on-site surface and subsurface soils.	1. On site soil contaminant distribution - horizontal and vertical 2. Determine contaminant concentrations, migration pathways and routes of entry in order to complete an Endangerment Assessment 3. Support the identification, development, and evaluation of remedial alternatives/ technology screening, and detailed alternative evaluation completed during the Feasibility Study.	Test pits, analysis of samples from side walls of pits and backhoe bucket
2. Characteristics of off-site surface and subsurface soils in the Crane-Deming Swamp.	1. Contaminant distribution in the Crane-Deming Swamp - horizontal and vertical 2. Determine contaminant concentrations, migration pathways and routes of entry in order to complete an Endangerment Assessment 3. Support the identification, development, and evaluation of remedial alternatives/ technology screening, and detailed alternative evaluation completed during the Feasibility Study.	Test pits, analysis of samples from side walls of pits and backhoe bucket

QAPP TABLE 3-4 (cont'd)

SAMPLING RATIONALE
RUETGERS-NEASE SALEM SITE RI/FS

Information Needed	Rationale	Data Gathering Methods
3. Characteristics of on-site Non-native Pond materials.	1. Pond contaminant distribution - vertical and horizonal 2. Non-native material physical characterization 3. Determine contaminant concentrations, migration pathways and routes of entry in order to complete an Endangerment Assessment 4. Support the identification, development, and evaluation of remedial alternatives/ technology screening, and detailed alternative evaluation completed during the Feasibility Study.	Soil borings, sampling and analysis of split spoon and shelby tube samples
4. Characteristics of Native Pond materials (soils under the pond bottom).	1. Pond contaminant distribution - vertical horizonal 2. Native material physical characterization 3. Determine contaminant concentrations, migration pathways and routes of entry in order to complete an Endangerment Assessment	Soil borings, sampling and analysis of split spoon and shelby tube samples

QAPP TABLE 3-4 (cont'd)

SAMPLING RATIONALE
RUETGERS-NEASE SALEM SITE RI/FS

Information Needed	Rationale	Data Gathering Methods
	4. Support the identification, development, and evaluation of remedial alternatives/ technology, and detailed alternative evaluation completed during the Feasibility Study.	
5. Characteristics of off-site surface and subsurface soils.	1. Off-site soil contaminant distribution 2. Determine contaminant concentrations, migration pathways and routes of entry in order to complete an Endangerment Assessment 3. Support the identification, development, and evaluation of remedial alternatives/ technology screening, and detailed alternative evaluation completed during the Feasibility Study.	Soil borings, sampling and analysis of split spoon or auger samples
6. Characteristics of on and off-site sediments.	1. Sediment contaminant distribution - horizontal - within the drainage ways, Feeder Creek, Slanker Pond and MFLBC	Collection and analysis of location specific surface sediments

QAPP TABLE 3-4 (cont'd)

SAMPLING RATIONALE
RUETGERS-NEASE SALEM SITE RI/FS

Information Needed	Rationale	Data Gathering Methods
	2. Determine contaminant concentrations, migration pathways and routes of entry in order to complete an Endangerment Assessment 3. Support the identification, development, and evaluation of remedial alternatives/ technology screening, and detailed alternative evaluation completed during the Feasibility Study.	
7. Characteristics of on and off-site surface water bodies	1. Contaminant distribution - horizontal - within the drainage ways, Feeder Creek, Slanker Pond and MFLBC 2. Determine contaminant concentrations, migration pathways and routes of entry in order to complete an Endangerment Assessment 3. Support the identification, development, and evaluation of remedial alternatives/ technology screening, and detailed alternative evaluation completed during the Feasibility Study.	Collection and analysis of location specific surface water samples

QAPP TABLE 3-4 (cont'd)

SAMPLING RATIONALE
RUETGERS-NEASE SALEM SITE RI/FS

Information Needed	Rationale	Data Gathering Methods
8. Characteristics of on and off-site ground water	1. Contaminant distribution - horizontal and vertical aquifers 2. Determine contaminant concentrations, migration pathways and routes of entry in order to complete an Endangerment Assessment 3. Support the identification, development, and evaluation of remedial alternatives/ technology screening, and detailed alternative evaluation completed during the Feasibility Study.	Sampling and analysis of monitoring and residential wells
9. Air monitoring station upwind and downwind of the site	1. Define areal extent of contaminant concentrations 2. Determine contaminant concentrations, migration pathways and routes of entry in order to complete an Endangerment Assessment 3. Support the identification, development, and evaluation of remedial alternatives/ technology screening, and detailed alternative evaluation completed during the Feasibility Study.	Sampling and analysis of 6 stations

QAPP TABLE 3-4 (cont'd)

SAMPLING RATIONALE
RUETGERS-NEASE SALEM SITE RI/FS

Information Needed	Rationale	Data Gathering Methods
10. Mapping and surveying	1. Locate existing structures and obstructions for alternatives evaluation, site features, and topography description	Site survey, site inspections, existing and updated facility maps

QAPP TABLE 3-5

CRITICAL SAMPLE SUMMARY
RUETGERS-NEASE SALEM SITE RI/FS

Sample Location	Matrix	Rationale	Activity
Well Cluster "I"	Ground Water	1) Determine existing background water quality at the site. 2) Provide basis for remediation levels.	1) Collect collocated sample from one well. 2) Complete two sample events. 3) 100%(1) completeness goal. 4) SAS low detection levels. (See Section 9)
Residential Wells	Ground Water	1) Ensure acceptable drinking water quality.	1) Collect two collocated samples during each sampling event. 2) 100%(1) completeness goal. 3) SAS low detection levels. (See Section 9)
MFLBC 1	Surface Water	1) Define background water quality conditions in MFLBC.	1) Collect one collocated sample at the upgradient location. 2) 100%(1) completeness goal 3) SAS low detection levels. (See Section 9)
Off-Site Soil (background)	Soil	1) Define natural or background soil conditions. 2) Provide remedial design Data	1) One collocated sample from one boring. 2) 100%(1) completeness goal.
MFLBC 1	Fish	1) Define background conditions	1) 100%(1) completeness goal.
Slanker Pond	Surface Water	1) Ensure acceptable water quality	1) 100%(1) completeness goal.

(1) Data with <100% completeness will be acceptable as long as the deficiency does not hinder project objectives.

Target Compound List (TCL) analysis (organic and inorganic) will be conducted using Contract Laboratory Program (CLP) procedures. The analysis of DCNB and DPS will also be analyzed using CLP procedures since these compounds are amendable to the semivolatile analytical protocol. Methods will be developed and validated for mirex, kepone and photomirex, which are not on the TCL. Methods development and validation study design and its completion will both be submitted for U.S. EPA and OEPA approval before investigation activities begin. Dioxins and furans will be analyzed by U.S. EPA PCDF/PCDD protocol, exhibit D, analytical methods, draft April 19, 1989 and revised in November 1989. If tetrachlorodioxins are found, the extract will be analyzed for 2,3,7,8-TCDD by EPA CLP SOW 8/87.

3.7 Monitoring Parameters and Frequency of Collection

The locations where investigative samples are to be collected as well as the types, frequency of collection, and analytical parameters are provided in Table 3-6. It should be noted that the number of soil samples and associated QA/QC samples collected at the on-site and off-site locations are dependent on the variables described in the Site Specific Sampling Plan. Sampling locations are shown in Figures 3-4 through 3-7. Sample locations shown on Figure 3-6 and 3- 7 are described on Table 3-7.

QAPP TABLE 3-6
ESTIMATED RI SAMPLING SUMMARY[1]
RUETGERS-NEASE SALEM SITE

Matrix	Sample Location		Number of Investigative Samples	Chemical Analysis[2] A	B	C	D	E	F	G	H	I	Number of Field QA/QC Samples[3] Field Duplicates/Replicates	Field Blanks	Matrix Total[4]
Soil	35	Test Pits[5]	105	87	18								11	6	122
	2	Pond 1 Borings[5]	8			6		2					1	1	10
	4	Pond 2 Borings[5]	15			9		3	3				2	1	18
	4	Pond 3 Borings[6]	15			9		3	3				2	1	18
	3	Pond 4 Borings[5]	12			6		3	3				1	1	14
	5	Pond 7 Borings[7]	21			12		6	3				2	2	25
	11	Off-Site Soil Borings[8]	22				22						2	2	26
	7	MFLBC Flood plain soil stations	28								28		3	2	33
Non-Native Material	2	Pond 1 Borings[5]	6	3	3								1		7
	4	Pond 2 Borings[5]	9	4	2				3				1	1	11
	4	Pond 3 Borings[7]	4	2	1				1						4
	3	Pond 4 Borings[5]	9	3	3				3				1		10
	5	Pond 7 Borings[6]	10	4	4				2				1	1	12
		Sludge/soil (Salem Treatment Plant)	3									3	1	1	5
Sediment		MFLBC, Feeder Crk, Slanker Pond, Crane Deming	61	14			22			18	7		7	4	72
Water		Ground Water Monitoring Wells[9]	66	62	4								7	4	77
		Ground Water Residential Wells[10]	6	6									1	1	8
	26	Surface Water Stations	26	12			14						3	3	32
Fish		MFLBC, Slanker Pond	56[11]	10			46						6		62
Air[12]		6 Stations	6	6									1	1	8
														Total:	574[13]

[1] Contingency analyses have not been included in this table.

[2] A=TCL volatile organics (+15), TCL nonvolatile (+25), mirex, kepone, photomirex, DPS.*
B=TCL organics (+40), TCL inorganics, mirex, kepone, photomirex, DPS,* dioxine and furans, 3,4-DCNB*.
C=TCL volatile organics (+15) mirex, kepone, photomirex, methoxychlor, DPS.*
D=TCL nonvolatile organics (+25), mirex, kepone, photomirex, DPS.*
E=TCL nonvolatile organics (+25).
F=Physical parameters: soil classification, specific gravity, permeability, effective porosity, particle size, moisture content, and Atterburg Limits.
G=Mirex, kepone, photomirex, DPS
H=Mirex, Kepone, photomirex, DPS & methocychlor
I=TCL organics (+40), TCL inorganics, mirex, kepone, photomirex, DPS,*3,4-DCNB*.
* 3,4-Dichloronitrobenzene (DCNB)* and Diphenylsulfone (DPS) will be analyzed as part of the semivolatile fraction.

[3] Frequency of field QA/QC sample collection:
Replicates/Duplicates---1 per 10 samples per matrix .
Field Blanks---1 per 10 samples for surface water samples and 1 per 20 samples for groundwater/solid/air samples. The 1 per 20 samples for field blanks apply to groundwater because dedicated sampling equipment will be utilized for the collection of samples from each monitoring well. (Note: for the submission of aliquots for laboratory MS/MSD, triple volume is required for VOAs and double volume is required for all other analyses.)

[4] Trip blanks will be submitted one per cooler containing aqueous samples for VOA analysis and one per 20 solid/air samples for VOA analysis.

[5] This number of samples assumes that test pits will be sampled to 6.5 feet below ground surface.

[6] This number of samples assumes that boring(s) will be sampled to a 12-foot depth and will have 3 feet of non-native material.

[7] This number of samples assumes that boring(s) will be sampled to a 15-foot depth and will have 6 feet of non-native material.

[8] This number of samples assumes that boring(s) will be sampled to a 3.5-foot depth.

[9] This number includes only Round 1 groundwater samples.

[10] Double the normal sampling volume will be required for residential well analyses.

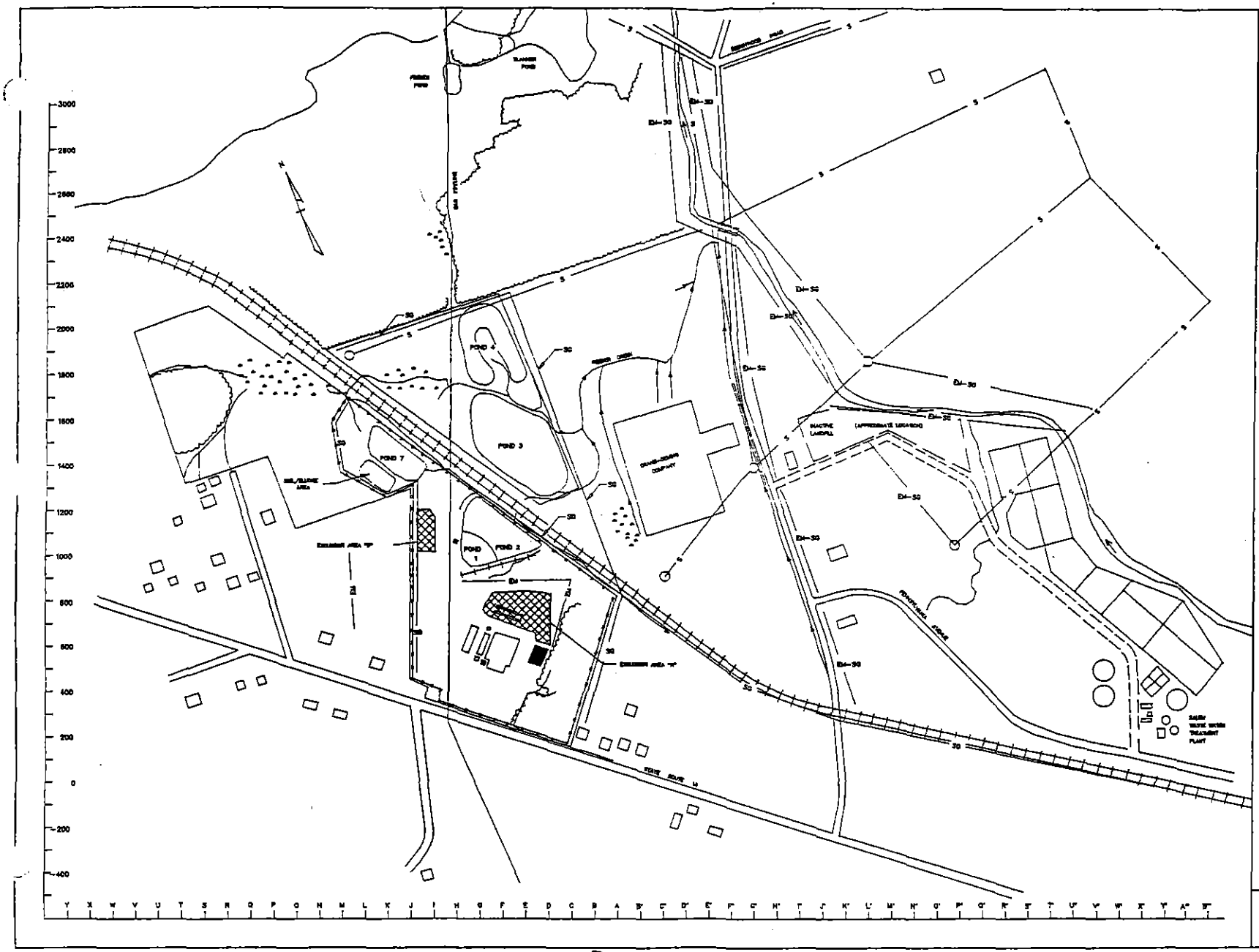
[11] 2 at Slanker, 54 in MFLBC

[12] The volatile analysis of air samples will be the TCL VOA +15 minus the 4 Kepones on the VOA TCL.

[13] This total does not include trip blanks.

NOTE: Matrix total does not include laboratory sampling.

CLP CLP



LEGEND

- REMOVED ON-SITE BUILDINGS
- EXISTING ON-SITE BUILDINGS
- RAILROAD TRACKS
- PROPERTY FENCE
- S — SEISMIC SURVEY LINE
- SG — SOIL GAS SURVEY LINE
- EM — ELECTROMAGNETIC CONDUCTIVITY SURVEY LINE
- SITE BOUNDARY

FIGURE QAAP 3 – 4

GEOPHYSICAL SURVEY LOCATIONS

RUETNERS-NEASE SALEM SITE 10/93

SCALE

0 200 400 600 (FEET)

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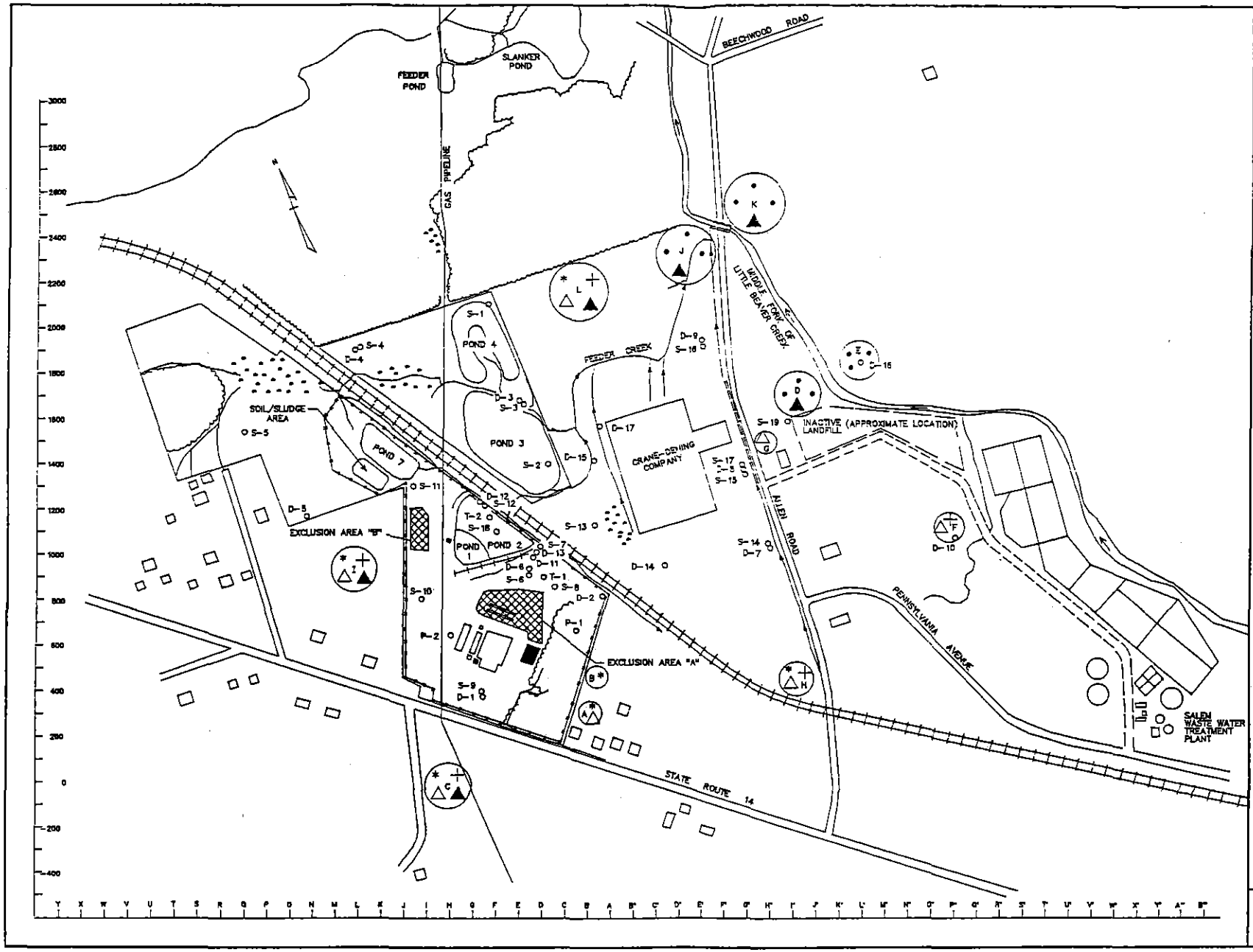

LEGEND

- VALLEY FILL WELL
- SHALLOW AQUIFER WELL
- INTERFACE AQUIFER WELL
- UPPER BEDROCK WELL
- LOWER BEDROCK WELL

NOTE: ACTUAL LOCATIONS OF WELLS WITHIN DRILLING AREAS WILL BE DETERMINED AT THE TIME OF WELL CONSTRUCTION.

- PROPOSED DRILLING AREA
- REMOVED ON-SITE BUILDINGS
- EXISTING ON-SITE BUILDINGS
- RAILROAD TRACKS
- PROPERTY FENCE
- D-16 EXISTING MONITORING WELL NO.
- EXISTING MONITORING WELL LOCATION
- SITE BOUNDARY

FIGURE QAAP 3 – 5
MONITORING WELL LOCATION AREAS

RUETGERS-NEASE SALEM SITE RI/FS

SCALE
0 200 400 600 (FEET)

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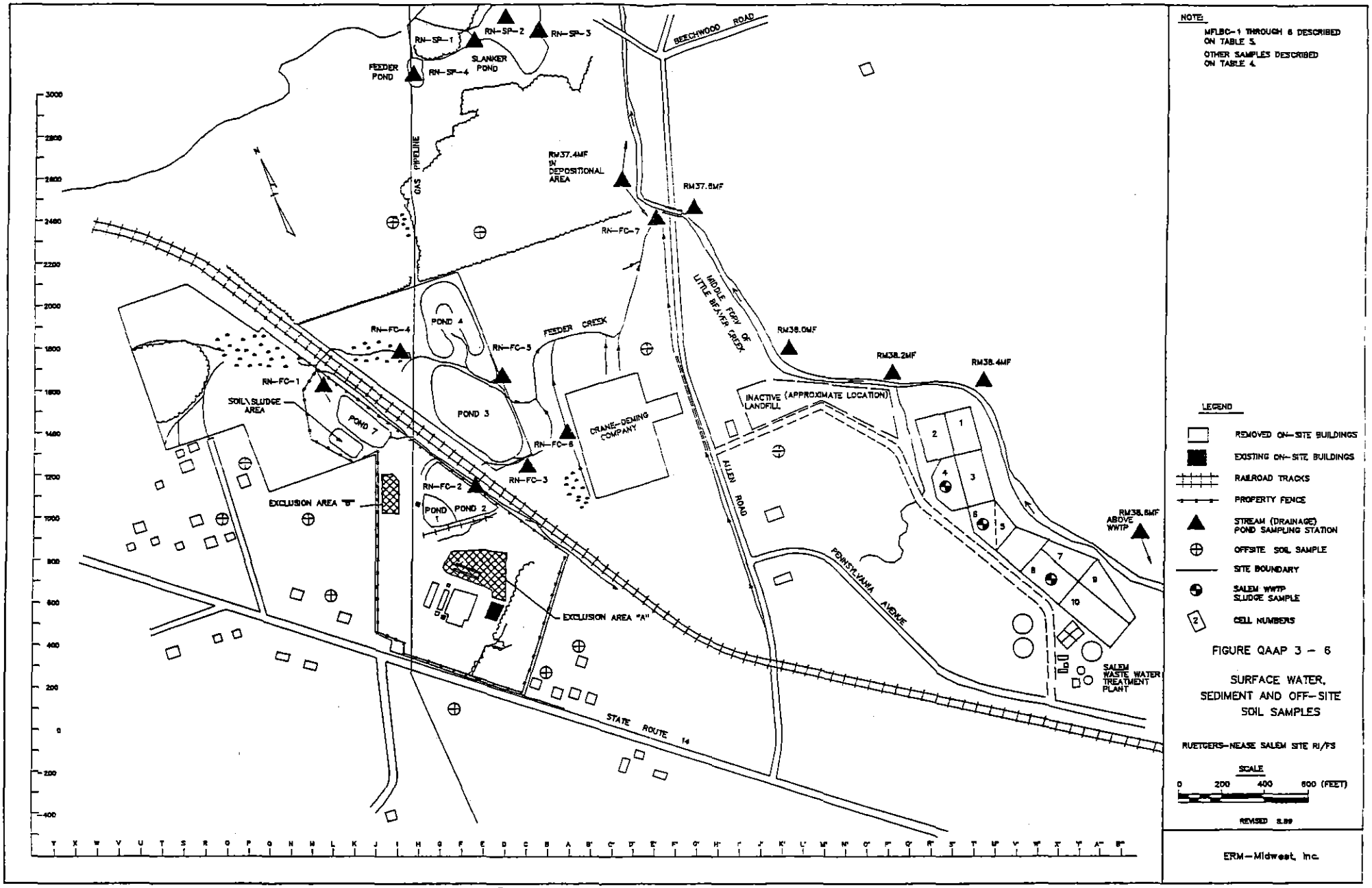

FIGURE QAAP 3 - 6

SURFACE WATER, SEDIMENT AND OFF-SITE SOIL SAMPLES

RUETGERS-NEASE SALEM SITE RI/FS

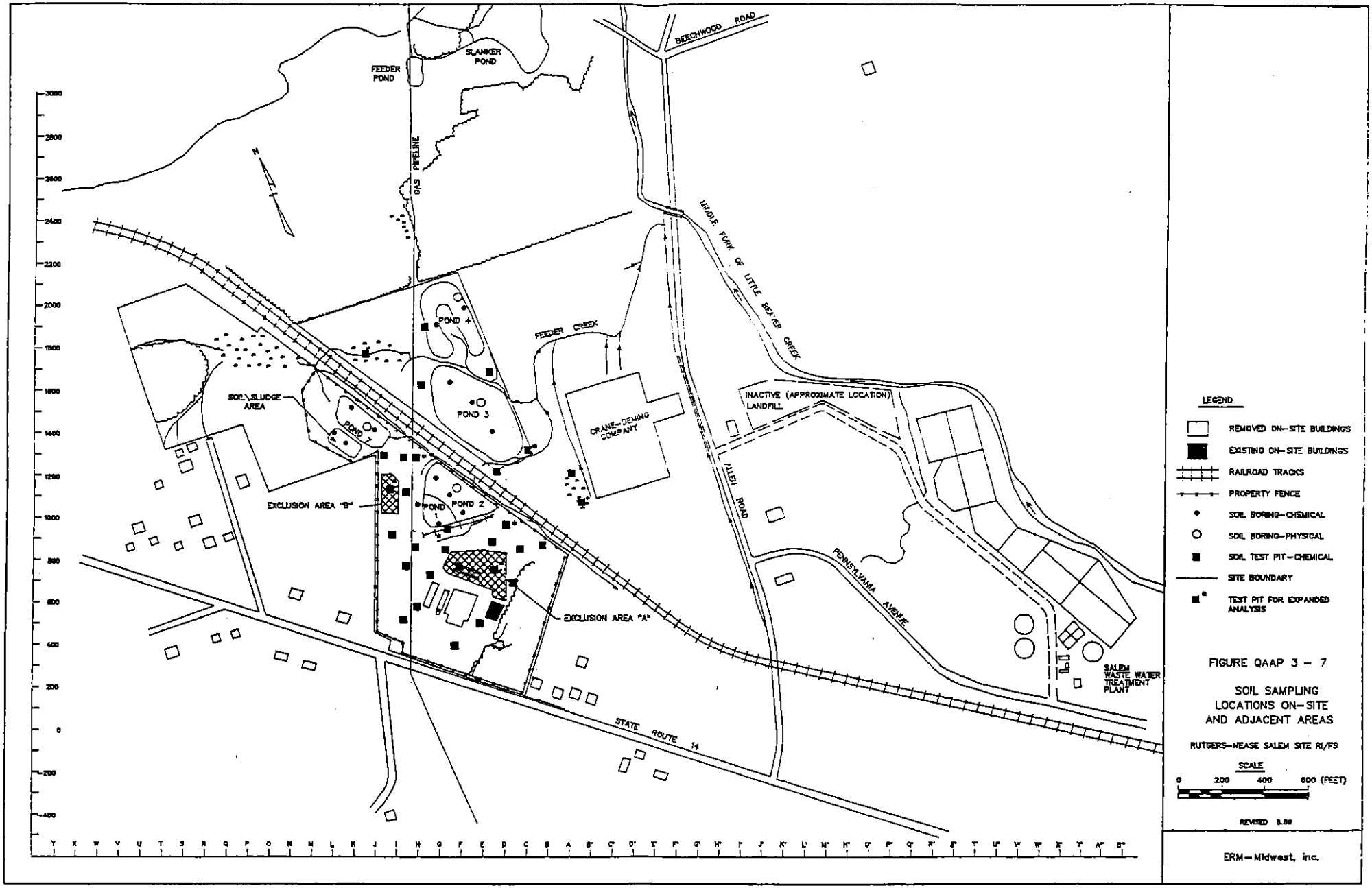

FIGURE QAAP 3 - 7

SOIL SAMPLING LOCATIONS ON-SITE AND ADJACENT AREAS

RUTGERS-NEASE SALEM SITE RI/FS

QAPP TABLE 3-7
Sampling Program for Survey of Feeder Creek, Slanker Pond, and Middle Fork of Little Beaver Creek

	Description	Station	5/16/89 Agreed Location No.	Analysis: Media F	SW	S	FP	Parameters: CLP +40	CLP Non-Vol +25	M, P, K, DPS	M, P, K, DPS, ME
A	Upstream of the WWTP as stream crosses Rte. 45	RM 38.6 MF	#1	2	1	1		4		4	0
	NE corner WWTP	RM 38.4 MF	#2		1	1		2		2	0
	Golf course stream	RM 38.2 MF	#3		1	1		2		2	0
	Discharge zone	RM 38.0 MF	#4		1	1		2		2	0
	Upstream Allen Road	RM 37.6 MF	#5	2	1	1		4		4	0
	Feeder/Slanker Pond	RN -SP- 4	#6A			1				1	0
G	Slanker Pond, inlet	RN - SP - 1	#6B			1				1	0
A	Slanker Pond, middle	RN - SP - 2	#6C	2	1	1		4		4	0
G	N. of Slanker Pond beach	RN - SP - 3	#6D			1				1	0
A	Allen Road downstream (Slanker Bridge, north)	RM 37.4 MF	#7	2	1	1		4		4	0
	Pine Lake Road bridge	RM 36.7 MF	#8	2	1			3		3	0
D	Between Goshen Road and Rte. 165	RM 35.4 MF	#9	2					2	2	0
H	Miller Farm	RM 35.0 MF	#10			1	4		0	0	5
G	Swamp area 0.3 RM south of Middletown Road		#11			1			0	1	0
H	Ruthraff Farm	RM 33.3 MF	#12			1	4		0	0	5
D	Rte. 45 (0.7 mi. N of Middletown Road)	RM 32.0 MF	#13	2	1	1			4	4	0
G	Swamp area between Rte. 45 and Rte. 62		#14			1			0	1	0
D	Rte. 62		#15	2		1			3	3	0
G	Swamp area 0.45 RM south of Rte. 62		#16			1			0	1	0
H	Sherwood Farm		#17			1	4		0	0	5
D	Rte. 165		#18	2	1	1			4	4	0
G	Beaver dam 1.85 RM south of Rte. 165		#19			1			0	1	0
H	Large swamp are west of beaver dam		#19A			1	4		0	0	5
	Large swamp are east of beaver dam (Shepherd dam)		#19B			1	4		0	0	5
D	Pine Lake Road bridge		#20	2	1	1			4	4	0
G	0.7 RM south of Pine Lake Road bridge		#21			1			0	1	0
D	Due east of intersection of E. 10th St. & Egypt Rd.		#22	2		1			3	3	0
	Private bridge 0.45 RM south of Rte. 14 bridge		#23	2	1	1			4	4	0
	N. Lisbon Rd-Rte. 14 at river bend	RM 24.5 MF	#24			1			0	1	0
G	Swamp area due west of EPA '89 station 24		#25			1			0	1	0
	Swamp area 0.53 RM south of EPA '89 station 24		#26			1			0	1	0
H	Camp Farm		#27			1	4		0	0	5

KEY:
F = Fish
SW = Surface Water
S = Sediment

B = Benthos
FP = Floodplain Sediment
M = Mirex
P = Photomirex

K = Kepone
DPS = Diphenyl sulfone
ME = Methoxychlor

ASSUMPTIONS:
- 2 fish samples per station
- 4 floodplain samples per location

NOTES:
-No Station #36
-The analysis of CLP+40 and CLP non-volatile+25 includes the analysis of methoxychlor.

QAPP TABLE 3-7

Sampling Program for Survey of Feeder Creek, Slanker Pond, and Middle Fork of Little Beaver Creek

Description	Station	5/16/89 Agreed Location No.	Analysis: Media F	SW	S	FP	Parameters: CLP +40	CLP Non-Vol +25	M, P, K, DPS	M, P, K, DPS, ME
Railroad bridge over Lisbon-Canfield Road	RM 23.5 MF	#28	2	1	1			4	4	0
Cunningham Road bridge over Stone Mill Run	RM 2.0 SMR	#29	2	1	1			4	4	0
Erie-Lackawanna bridge over E. Branch Cherry Valley Run		#30	2	1	1			4	4	0
SE bank of confluence of MFLBC & Cherry Valley Cr.		#31			1			0	1	0
0.23 RM south of old Rte. 344 bridge		#32			1			0	1	0
Swamp area due west of EPA '89 station 32		#33			1			0	1	0
Swamp area 0.68 RM north of Rte. 45		#34			1			0	1	0
Teagarden bridge on Eagleton Road	RM 17.5 MF	#35	2	1	1			4	4	0
Coleman Road bridge	RM 15.1 MF	#37	2		1			3	3	0
0.37 RM south of Furnace Road bridge		#38			1			0	1	0
Above Lisbon dam	RM 12.5 MF	#39	2		1			3	3	0
Below Lisbon spillway	RM 12.5 MF	#40	2	1	1			4	4	0
0.6 RM west of EPA '89 station 42		#41			1			0	1	0
Elkton West Point Road bridge		#42	2	1	1			4	4	0
0.2 RM east of EPA '89 station 42		#43			1	4		0	0	5
Beaver Creek State Park canoe livery 2.25 mi. east of Elkton	RM 4.6 MF	#44	2		1			3	3	0
Beaver Hollow Road Bridge		#45	2		1			3	3	0
Swamp area by Rte. 7 north of Williamsport		#46			1			0	1	0
Y Camp Road bridge	RM 14.4 WB	#47	2	1	1			4	4	0
Bell School Road bridge	RM 14.4 LBC	#48	2	1	1			4	4	0
Sprucevale Bridge-Beaver Creek State Park	RM 11.0 LBC	#49	2		1			3	3	0
Fredricktown bridge	RM 0.2 LBC	#50	2	1	1			4	4	0
1 RM south of MFLBC/NFLBC confluence		#51	2		1			3	3	0
Grimms Road bridge gauging station	RM 4.5 LBC	#52	2	1	1			4	4	0
Feeder Creek NNW of Pond 7	RN-FC-1	#53			1		1		1	
Feeder Creek East of Pond 2	RN-FC-2	#54			1		1		1	
Feeder Creek S of Pond 3	RN-FC-3	#55		1	1		2		2	
Feeder Creek (Swamp) W of Pond 4	RN-FC-4	#56			1		1		1	
Feeder Creek S of Pond 4	RN-FC-5	#57		1	1		2		2	
Feeder Creek W of Crane-Deming	RN-FC-6	#58		1	1		2		2	
Feeder Creek Prior to entering MFLBC	RN-FC-7	#59		1	1		2		2	
Subtotal			56	26	61	28	36	82	136	35
Total			171				289			

KEY:
F = Fish
SW = Surface Water
S = Sediment

B = Benthos
FP = Floodplain Sediment
M = Mirex
P = Photomirex

K = Kepone
DPS = Diphenyl sulfone
ME = Methoxychlor

ASSUMPTIONS:
- 2 fish samples per station
- 4 floodplain samples per location

NOTES:
-No Station #36
-The analysis of CLP+40 and CLP non-volatile+25 includes the analysis of methoxychlor.

4.0 PROJECT ORGANIZATION AND RESPONSIBILITY

QA/QC procedures must be strictly adhered to in order to ensure the production of the highest quality data. Several organizations will have responsibilities for implementing portions of this QAPP. Ruetgers-Nease owns the site. ERM-Midwest, Inc. has been retained to conduct most RI/FS activities. The ERCO Laboratory of Enseco will conduct chemical analyses of all samples except for dioxin and furan analysis, T01/T02 thermal desorption, and physical properties. Dioxin and furan analysis will be conducted at the California Analytical Laboratory of Enseco. The T01 and T02 thermal desorption VOA analyses will be conducted at the CRL/El Monte Air Toxics Laboratory of Enseco. GZA Laboratory of Newton, MA, will perform the analysis for the physical parameters in soils. Analytical data for this investigation will have two separate reviews for validity and usability. Enseco will validate the analytical data prior to release. ERM's Project QA/QC Manager will perform a rigorous validation to determine validity and fitness for use with appropriate qualifications. U.S. EPA and OEPA retain the option to conduct a final data review on any portion of the data within the appropriate period designated on Figure 3-3. The organization and responsibilities of individuals and organizations implementing the procedures specified in this QAPP are described in the following subsections.

4.1 Organization

The key individuals who are responsible for the overall coordination of efforts to be conducted, as well as the collection, validation, and interpretation of data generated during the RI are identified on Figure 4-1. Figure 4-2 identifies the structure of the Enseco Laboratory, where the majority of analyses will be performed. Figure 4-3 identifies

Figure QAPP 4-1
Quality Assurance Project Organization
Ruetgers-Nease Salem Site RI/FS

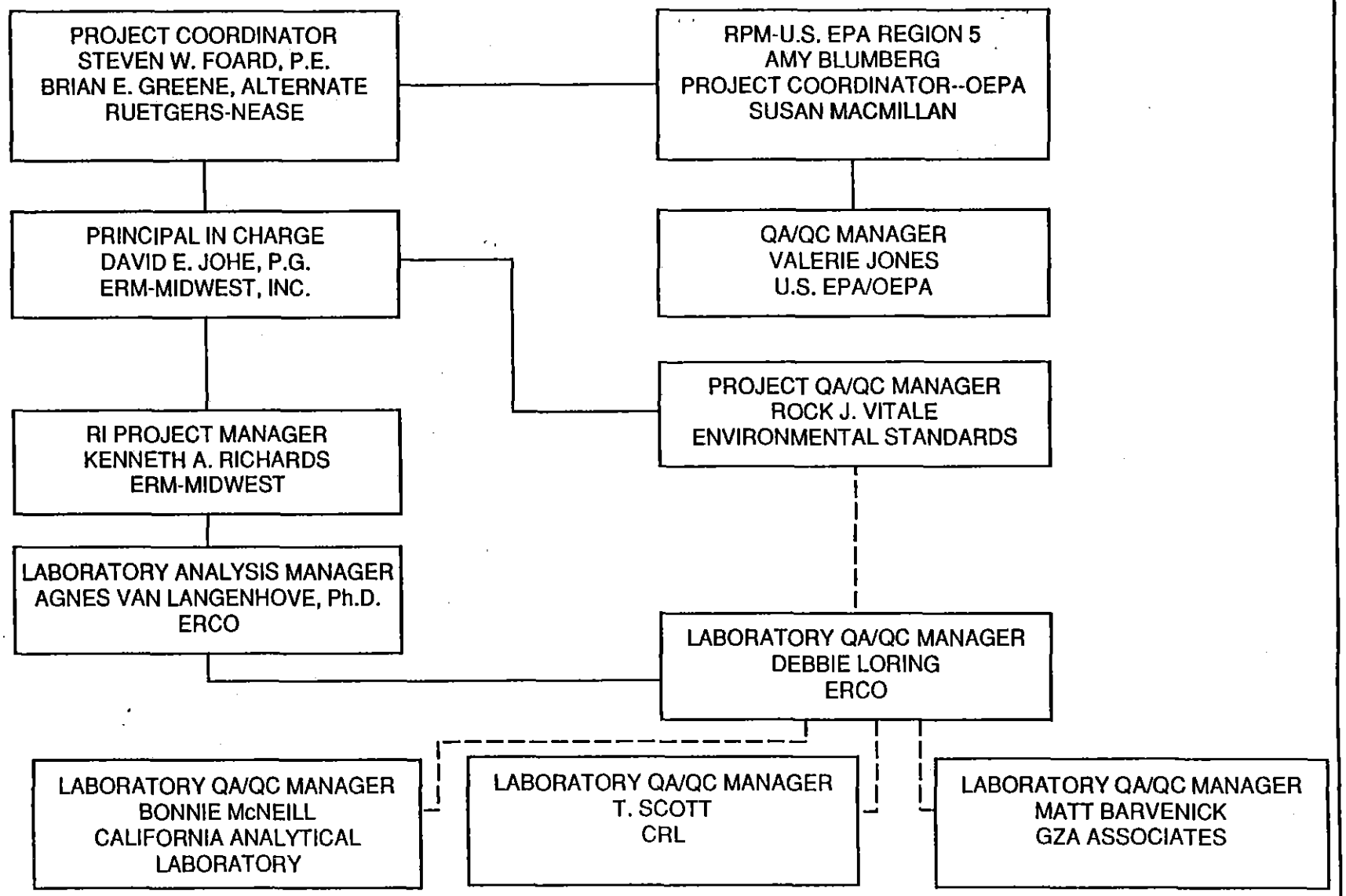

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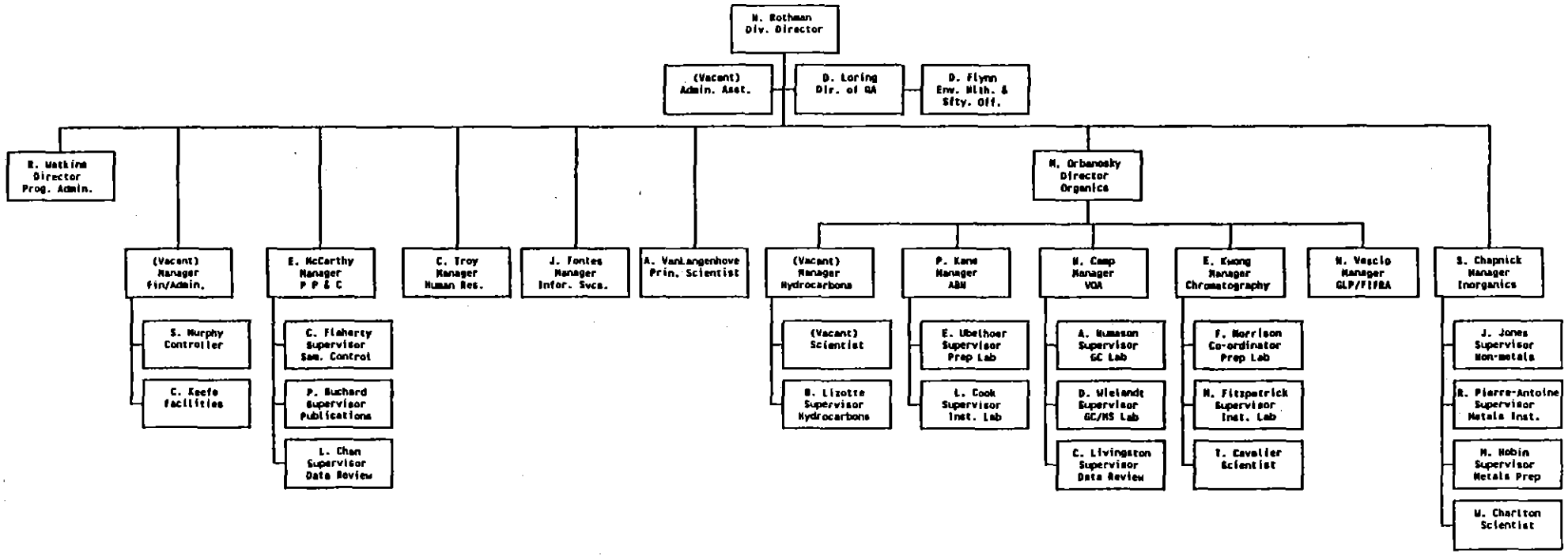
FIGURE 4-2

ERCO LABORATORY STRUCTURE



QAPP-35

SECTION: 4
Rev.4/Feb.1990

Enseco-Erco Laboratory
Cambridge, Massachusetts

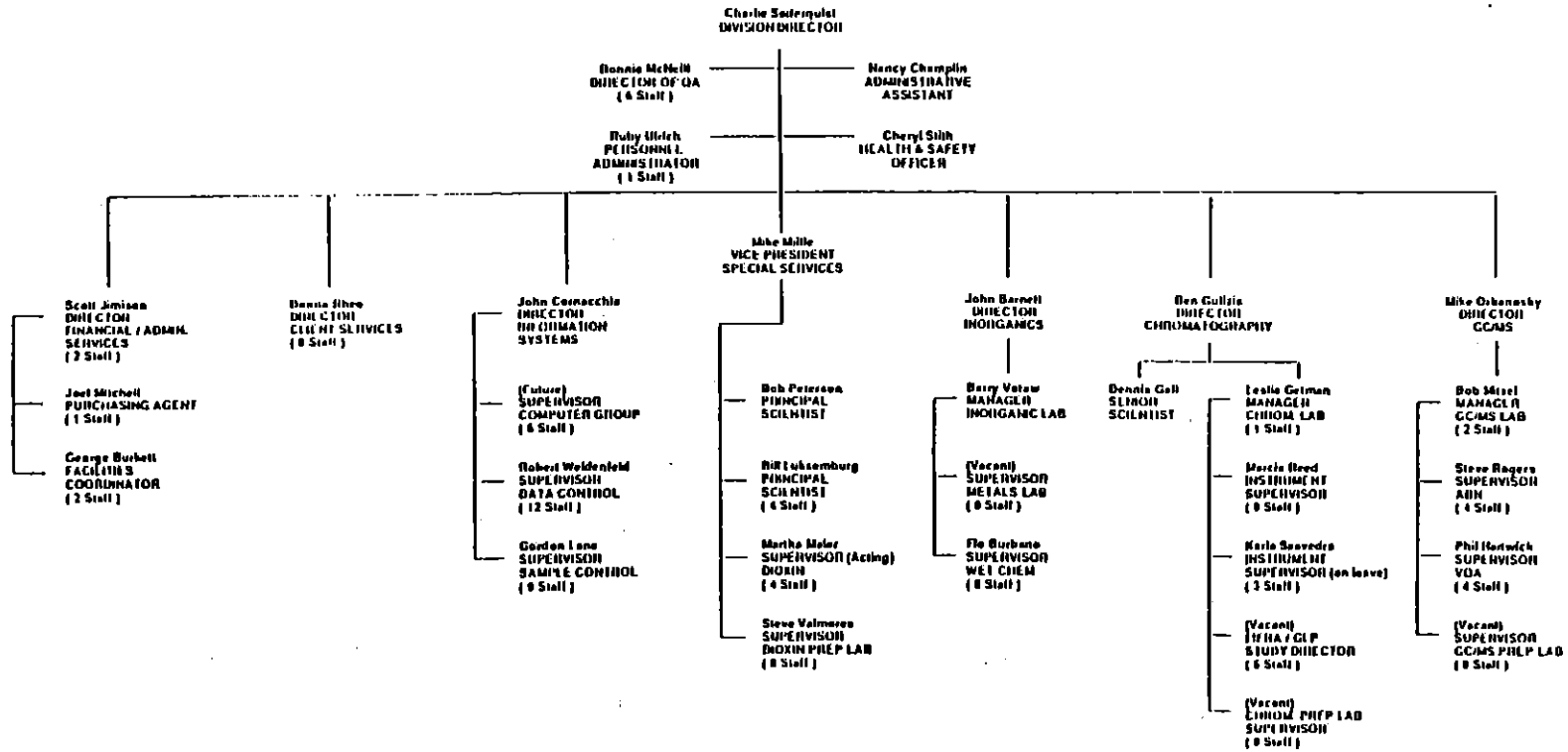
Enseco

VERSION 89-2

FIGURE QAPP 4-3

CAL ANALYTICAL LABORATORY STRUCTURE

Enseco



ENSECO-CAL LAB
Organization Chart
July 88 / Version 37

the structure of the Enseco-CAL Analytical Laboratory, where the dioxin and furans analysis will be performed. Figure 4-4 identifies the structure of the Enséco-CRL/El Monte Laboratory, where the T01 and T02 thermal desorption VOA analyses will be done.

4.2 Overall Management and QA/QC Responsibilities

General responsibilities of the individuals responsible for QA/QC are described in the following sections.

4.2.1 Project Coordinator

Steven Foard, P.E. of Ruetgers-Nease is the Project Coordinator (PC) for this investigation. The alternate PC is Brian Greene, also of Ruetgers-Nease. Responsibilities of the PC include:

1. Interface with U.S. EPA/OEPA RPM/Project Coordinator on all aspects of the RI/FS.

2. Oversight and approval of Project plans.

3. Ensure that terms of the Consent Order and Statement of Work are met.

4.2.2 Principal-in-Charge

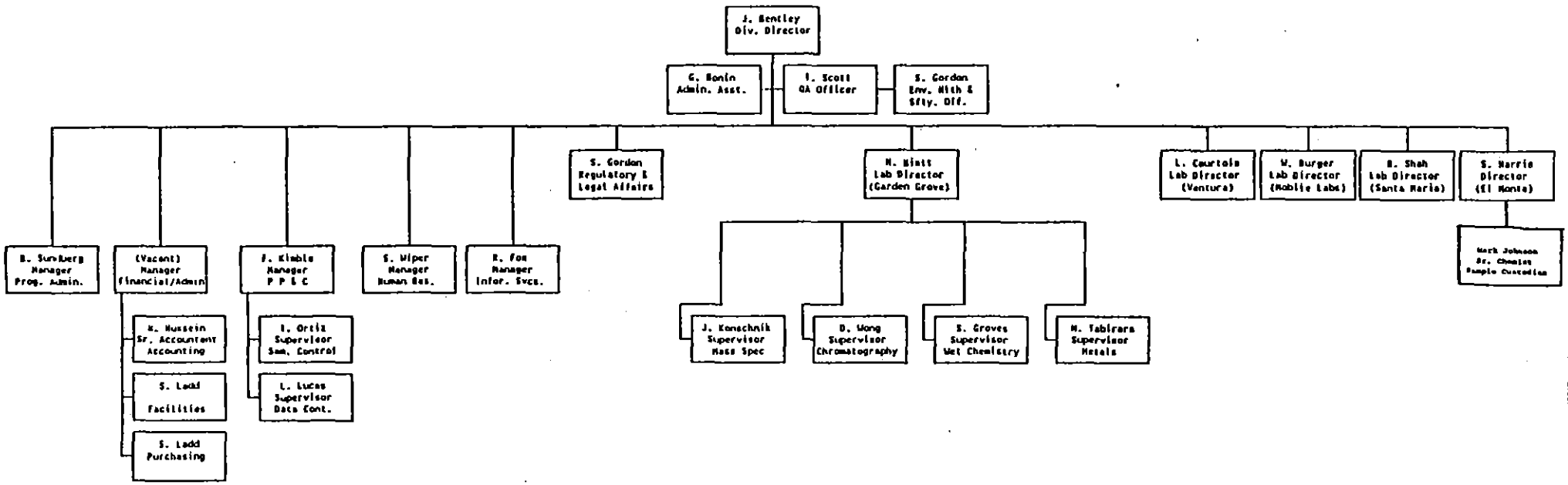
David E. Johe is the Principal-in-Charge for this investigation. Mr. Johe is a Principal with ERM-Midwest and is responsible for the following:

1. Resource availability to the project.

2. Overall technical direction of the project.

FIGURE 4-4

ENSECO-CRL LABORATORY STRUCTURE



Enseco-CRL

Garden Grove, Santa Maria, Ventura and El Monte, California

Enseco

VERSION 89-2

3. Review all technical documents for accuracy and completeness with respect to the QAPP and associated site plans.

4.2.3 Project QA/QC Manager

Rock J. Vitale, of Environmental Standards, Inc. is the Project Quality Assurance Manager for this investigation. He will be responsible for assuring that field, office, and laboratory activities and analyses are conducted in accordance with the QAPP. Specific responsibilities include the following:

1. Conduct performance and system audits.

2. Review all documents with respect to adherence to QA procedures provided in the QAPP.

3. Review SAS data and RAS data including mass spectral library searches for tentative identifications.

4. Preparation of analytical data tables and quality assurance reviews.

5. Recommend and institute corrective actions based on reviews and audits.

4.2.4 RI Project Manager

Kenneth A. Richards, of ERM-Midwest is the RI Project Manager for this investigation. He has overall project responsibility and direct responsibility for the management of staff involved in this project. Specific duties are as follows:

1. Providing an interface between the Project Coordinator and the Field Operations Manager.

2. Implementing project plans.

3. Coordinating project activities.

4. Coordinating project personnel and staffing.

5. Completing project deliverable reviews.

6. Providing input on technical direction.

4.2.5 Field Operations Manager

Robert A. Ferree, of ERM-Midwest is the Field Operations Manager for this project and will be responsible for on-site activities including well drilling, sampling, and health and safety. Specific activities include the following:

1. Ensure that field activities are conducted in accordance with the Site Specific Sampling Plan, QAPP, and Health and Safety Plan.

2. Review and evaluate field records.

3. Subcontractor coordination.

4. Oversight of field staff.

4.2.6 Analytical Laboratory QA Manager

The Analytical Laboratory QA Managers, Deborah Loring of ERCO, Bonnie McNeill of CAL Analytical, T. Scott of CRL and Matt Barvenick of GZA, will be responsible for the inspection and verification of laboratory QA/QC records and results. Any deviation or discrepancies in the data will be reported

to the Laboratory Manager or her assistant for review and, if necessary, appropriate corrective action. Additional responsibilities of the Analytical Laboratory QA Manager include:

1. Implementation of all laboratory QA/QC procedures contained in the QAPP.

2. Overseeing corrective actions as required.

3. Conducting internal system and performance audits and inspection of analytical procedures and implementing modifications as approved by the Project QA/QC Manager.

4. Developing laboratory QA programs including statistical procedures and techniques.

5. Review SAS and RAS data including mass spectral library searches for tentative identifications.

A final data package including all raw data will be submitted to the Project QA/QC Manager for a rigorous quality assurance review.

4.2.7 Laboratory Sample Custodian

Kathy Flaherty of ERCO, Gordon Lane of CAL Analytical, Ike Orvitz of CRL/El Monte and Janice Gregory of GZA are the designated sample custodians for this RI/FS. The sample custodian's responsibilities include ensuring proper sample entry, handling procedures and documentation by laboratory personnel.

4.2.8 U.S. EPA Remedial Project Manager and OEPA Project Coordinator

Amy Blumberg is the U.S. EPA Region 5 Remedial Project Manager and Susan MacMillan is the OEPA Project Coordinator for this RI/FS. Their responsibilities include:

1. Technical review and approval of all plans and data, including raw data, submitted as part of this RI/FS.

2. Coordination of RI/FS activities with the Project Coordinator.

3. Authority vested in an On-Scene Coordinator and a Remedial Project Manager by the National Contingency Plan, 40 CFR Part 300, as amended, including the authority as provided therein to halt conduct, or direct any work described in the RI/FS Work Plans, or to direct any response action undertaken by the U.S. EPA when conditions at the facility may present an imminent and substantial endangerment to the public health and welfare or the environment. The U.S. EPA Project Coordinator's actions shall, at all times be controlled and limited by provisions of the National Contingency Plan, 40 CFR Part 300.

4.2.9 U.S. EPA/OEPA QA Manager

Valerie Jones is the U.S. EPA QA Manager for this investigation. She is responsible for assuring that all laboratory activities and analytical data are of sufficient quality to meet the objectives of the investigation. Specific responsibilities include:

1. Technical review and approval of the QAPP submitted as part of this RI/FS

2. Coordinate with EPA Region V CRL on system and performance audits.

4.2.10 U.S. EPA Region V Central Regional Laboratory

It is the responsibility of the U.S. EPA Region V Central Regional Laboratory to perform the external performance and system audits for the laboratories selected for this project.

5.0 QA OBJECTIVES FOR MEASUREMENT DATA

The primary objective of the Quality Assurance (QA) program is to provide data of sufficient quality and quantity to assure that project objectives, as stated in Section 3.0 are achieved. Data quality and quantity are measured through comparison of resulting data with established acceptable limits for data precision, sensitivity, accuracy, representativeness, comparability and completeness (PSARCC) as described in U.S. EPA document EPA/540/G-87/003 titled, "Data Quality Objectives for Remedial Response Activities." Data that have certain aspects that may be outside PSARCC QA objectives will be evaluated, according to Section 3.2.3 of the DQO document, and the criteria contained in the current CLP-Statement of Work, to determine what, if any, aspects of the data can be defensibly used to meet the RI/FS objectives. Objectives for the PSARCC parameters for this RI/FS are described in this section.

5.1 PSARCC Objectives

PSARCC parameter objectives have been developed for four (4) sample categories based on sample objectives, analytical methods, historical data and published guidelines for the CLP. These sample categories are:

1. Samples scheduled for Standard TCL organic and inorganic analyses with routine analytical services (RAS) detection limits.

2. Samples scheduled for Standard TCL organic analyses with special analytical service (SAS) low detection limits.

3. Samples scheduled for Standard TCL organic and inorganic analyses with RAS detection levels but where matrix interferences due to expected high concentrations of compounds may prevent the achievement of RAS detection limits for other applicable constituents.

4. Samples scheduled for non-standard compound analysis such as mirex, kepone, photomirex, DPS, and DCNB.

Data quality objectives for the RI/FS are summarized in Table 5-1. The parameters for standard TCL-RAS and TCL-SAS analyses are based on CLP requirements and U.S. EPA guidance documents. PSARCC parameter objectives should be achieved through the use of standardized sample collection and analysis procedures.

Technically, obtaining RAS detection levels may not be feasible for samples with high matrix interference caused by high concentration analytes. For example, if a sufficiently high concentration of one compound is present in a sample, attempts to analyze the sample without a dilution may "shut-down" an instrument. Samples where this may occur include on-site soil and waste samples, and monitoring wells within the plume. All attempts will be made to analyze samples directly or with the absolute minimum dilution, or to perform all available clean-up techniques. However, the primary consideration for these high-level samples is the accurate identification and quantification of analytes since these high-level analytes will essentially drive the clean-up process. In addition, it should be noted that low-level detection limits can be obtained on these samples to determine if other contaminants are present after some remedial action has effectively removed or reduced the high-level components. Accordingly, project objectives could be

achieved even if RAS detection levels may not be met for high concentration samples.

Tables 5-1 through 5-3 contain separate PSARCC objectives for investigation samples. It should be noted that the criteria objectives not yet specified for Mirex, photomirex, kepone, DPS and DCNB on Table 5-2 will be specified once the method validation study is completed.

Analytical methods for non-standard compounds will be validated as part of the overall Work Plan approval. The mirex, photomirex, kepone, DPS, and DCNB method validation study will be approved by the agencies and incorporated into the Work Plan.

PSARCC parameter objectives and use are described in more detail below.

5.1.1 Precision

Precision measures the reproducibility of data or measurements under specific conditions. Precision is a quantitative measure of the variability of a group of data compared to their average value. Precision is usually stated in terms of relative percent difference or relative standard deviation. Measurement of precision is dependent upon sampling technique and analytical method. Both sampling and analysis will be as consistent as possible.

To ensure that precision is achieved, QC samples including field and laboratory duplicate samples, and matrix spike and matrix spike duplicate recoveries will be analyzed and used to measure precision. An additional measure of precision is the comparison of surrogate recoveries between the unspiked, matrix spike and matrix spike duplicate sample aliquots. As per Contract Laboratory Program (CLP)

QAPP TABLE 5-1
Data Quality Objectives

DQO Parameter	RAS	SAS[1]	Non-Standard Analysis[2]
Precision	Tables 5-2 and 5-3	Tables 5-2 and 5-3	Tables 5-2 and 5-3
Accuracy	Tables 5-2 and 5-3	Tables 5-2 and 5-3	Tables 5-2 and 5-3
Sensitivity	Section 9 of the QAPP	Section 9 of the QAPP	Section 9 of the QAPP
Representativeness (field replicates)	Inorganic—Aq<20%RPD@>CRDL[3] Inorganic—Sol<35%RPD@>CRDL[3] Organic—VOA<15%RPD(Aq),<25%RPD(sol)[4] Organic—BN<25%RPD(Aq)<32% RPD(sol)[4,5] Organic—Acids<45%RPD(Aq)<43%RPD(sol)[4] Organic—Pest/PCBs<25RPD(Aq)<45%RPD(sol)[4] Organic—Dioxin/furans<25RPD(Aq)<45%RPD(sol)[4]	Organics—VOA<25%RPD(air)<35%RPD (fish) Organics—BN<32%RPD(air)<40%RPD(fish) Organics—Acids<43%RPD(air)<53%RPD(fish) Organics—Pest/PCBs<45%RPD(air)<55%RPD(fish)	Mirex<25RPD(Aq)<45%RPD(sol),<45%RPD(air), <55%RPD (fish) Photomirex<25RPD(Aq)<45%RPD (sol)<45%RPD(air)<55RPD(fish) Kepone<40RPD(Aq)<50%RPD(sol), <50%RPD(air) <60%RPD(fish) Diphenyl sulfone<25%RPD(Aq),<40%RPD(sol),<40%RPD(air)<50%RPD(fish)
Completeness			
Non-Critical Samples	90%	90%	90%
Critical Samples	100%[6]	100%[6]	100%
Comparability	Based on precision & accuracy & media comparison	Based on precision & accuracy & media comparison	Based on precision & accuracy & media comparison

Notes:

[1] SAS for fish and air matrices

[2] Non-Standard Analysis—mirex, photomirex, kepone, diphyenyl sulfone, and 3,4-dichloronitrobenzene

[3] Criteria for field replicates derived from CLP Criteria

[4] Criteria for field replicates derived by calculating the mean RPD for each fraction from CLP matrix spike duplicate RPDs

[5] 3,4-dichloronitrobenzene and diphenyl sulfone will be analyzed as an additional compound under CLP BN RAS

[6] Data with <100% completeness will be acceptable as long as the deficiency does not hinder project objectives; for example, compounds that are not pertinent to the investigations or common laboratory contaminants and low RFs for ketones

QAPP Table 5-2
Accuracy & Precision Data Quality Objectives for RAS & SAS Organics

Parameter	Audit	Compounds	Fish Control Limits	Aqueous Ctrl Lmts	Air Control Limits	Solid Control Limits
VOA Components	Lab blank, field blank	All TCL Compounds	<5x CRDL methylene chloride, acetone, toluene and 2-butanone ≤ CRDL all other compounds	<5 x CRDL methylene chloride, acetone, toluene and 2-butanone ≤ CRDL all other compounds	<5 x DL methylene chloride and toluene	<5 x CRDL methylene chloride, acetone, toluene and 2-butanone ≤ CRDL all other compounds
	Matrix spike duplicate precision	See Matrix spikes	<50% RPD (95% CI)	<14% RPD (95% CI)	NA	<24% RPD (95% CI)
	Surrogate Spike Recovery	D_4—1,2-dichloroethane	(50-150%)	(76–114%)	NA	(70–121%)
		D_8—toluene	(50-150%)	(88–110%)	NA	(81–117%)
		Bromofluorobenzene	(50-150%)	(86–115%)	NA	(74–121%)
	Matrix Spike Recovery	1,1-dichloroethene	(49-172%)	(61–145%)	NA	(59–172%)
		trichloroethene	(50-150%)	(71–120%)	NA	(62–137%)
		benzene	(50-150%)	(76–127%)	NA	(66–142%)
		toluene	(49-150%)	(76–125%)	NA	(59–139%)
		chlorobenzene	(50-150%)	(75–130%)	NA	(60–133%)
BNA Components, including DCNB, DPS	Lab blank, field blank	All Parameters	<5 x CRDL phthalate esters ≤ CRDL all other compounds	<5 x CRDL phthalate esters ≤ CRDL all other compounds	<5 x CRDL phthalate esters ≤ CRDL all other compounds	<5 x CRDL phthalate esters ≤ CRDL all other compounds
	Matrix spike recovery and matrix spike duplicate precision	phenol	(9–90%) (50% RPD)	(12–89%) (42% RPD)	(12–100%) (42%RPD)	(26–90%) (35% RPD)
		2-chlorophenol	(17–123%) (50% RPD)	(27–123%) (40% RPD)	(25–123%) (50% RPD)	(25–102%) (50% RPD)
		1,4-dichlorobenzene	(18-114%) (35% RPD)	(36–97%) (28% RPD)	(28–104%) (28%RPD)	(28–104%) (27% RPD)
		N-nitroso-di-n-propylamine	(21–126%) (41% RPD)	(41–116%) (38% RPD)	(41–126%) (38% RPD)	(41–126%) (38% RPD)
		1,2,4-trichlorobenzene	(27–107%) (32% RPD)	(39–98%) (28% RPD)	(38–107%) (23% RPD)	(38–107%) (23% RPD)
		p-chloro-m-cresol	(16–102%) (47% RPD)	(23–97%) (42% RPD)	(23–103%) (42% RPD)	(26–103%) (33% RPD)
		acenaphthene	(21–142%) (34% RPD)	(46–118%) (31% RPD)	(31–137%) (31% RPD)	(31–137%) (19% RPD)
		4-nitrophenol	(6–114%) (60% RPD)	(10–80%) (50% RPD)	(10–114%) (50% RPD)	(11–114%) (50% RPD)
		2,4-dinitrotoluene	(18–99%) (51% RPD)	(24–96%) (38% RPD)	(24–98%) (47% RPD)	(28–89%) (47% RPD)
		pentachlorophenol	(4–110%) (60% RPD)	(9–103%) (50% RPD)	(9–109%) (50% RPD)	(17–109%) (47% RPD)
		pyrene	(22–142%) (41% RPD)	(26–127%) (31% RPD)	(26–142%) (36% RPD)	(35–142%) (36% RPD)
		3,4-dichloronitrobenzene	(36-76%) (30% RPD)	(54-94%) (30% RPD)	(75-115%) (30% RPD)	(33-76%) (30% RPD)
		diphenylsulfone	(0-92%) (54% RPD)	(78-118%) (30% RPD)	(78-118%) (30% RPD)	(52-126%) (37% RPD)
	Surrogate spike recovery	d_5-nitrobenzene	(13–120%)	(35–114%)	(23–120%)	(23–120%)
		2-fluorobiphenyl	(20–116%)	(43–116%)	(30–116%)	(30–115%)
		d_{14}-terphenyl	(8–141%)	(33–141%)	(18–141%)	(18–137%)
		d_6-phenol	(6–113%)	(10–94%)	(10–113%)	(24–113%)
		2-fluorophenol	(11–121%)	(21–100%)	(21–121%)	(25–121%)
		2,4,6-tribromophenol	(6–123%)	(10–123%)	(10–123%)	(19–122%)
HAS Pesticides/ PCBs	Lab blank, field blank,	All TCL Pesticides/PCBs	<CRDL for all compounds	<CRDL for all compounds	<CRDL for all compounds	<CRDL for all compounds
	Matrix spike recovery and matrix spike duplicate precision	lindane	(36–123%) (50% RPD)	(56–123%) (15% RPD)	(46–127%) (50% RPD)	(46–127%) (50% RPD)
		heptachlor	(25–130%) (31% RPD)	(40–131%) (20% RPD)	(35–130%) (31% RPD)	(35–130%) (31% RPD)
		aldrin	(24–132%) (43% RPD)	(40–120%) (22% RPD)	(34–132%) (43% RPD)	(34–132%) (43% RPD)
		dieldrin	(21–134%) (38% RPD)	(52–126%) (18% RPD)	(31–134%) (38% RPD)	(31–134%) (38% RPD)
		endrin	(32–139%) (45% RPD)	(56–121%) (21% RPD)	(42–139%) (45% RPD)	(42–139%) (45% RPD)
		4,4-DDT	(13–134%) (50% RPD)	(38–127%) (27% RPD)	(23–134%) (50% RPD)	(23–134%) (50% RPD)
	Surrogate spike recovery	dibutylchlorendate	(10–150%)	(24–154%)	(20–150%)	(20–150%)
Mirex, Photomirex, Kepone	Lab blank, field blank	All Parameters	<MDL	<MDL	<MDL	<MDL
	Matrix spike and duplicate precision	mirex	(82-122%) (30% RPD)	(74-114%) (30% RPD)	(74-117%) (30%RPD)	(57-124%) (34% RPD)
		photomirex	(65-105%) (30% RPD)	(70-110%) (30% RPD)	(74-114%) (30% RPD)	(53-122%) (35% RPD)
		kepone	(24-97%) (37% RPD)	(19-83%) (32% RPD)	(0-79%) (42% RPD)	(25-65%) (30% RPD)
Dioxins and Furans	Lab blank, field blank	All Parameters	N/A	<MDL	N/A	<MDL
	Performance check solution (supplied by USEPA)	2,3,7,8-TCDD	N/A	within 95% CI	N/A	within 95% CI
	Fortified field blank	2,3,7,8-TCDD	N/A	60–140%	N/A	60–140%
	Laboratory duplicate	2,3,7,8-TCDD	N/A	<50% RPD	N/A	<50% RPD
	Surrogate recovery	$^{37}Cl_4$-2,3,7,8-TCDD	N/A	±40%	N/A	±40%

NA – This medium is not scheduled for this analysis.

QAPP Table 5-3
Accuracy and Precision Data Quality Objectives for RAS Inorganics

Parameter	Audit	Constituent	Aqueous Control Limits	Soil Control Limits
Metals and Cyanide	Lab blank, field blank	All TCL constituents	<±CRDL	<±CRDL
	Laboratory duplicates	All TCL constituents	<20% RPD for results >5 x CRDL <±CRDL for results <5 x CRDL	<20% RPD for results >5 x CRDL <±CRDL for results <5 x CRDL
	Matrix spike recovery	All TCL constituents	75–125% unless the sample concentration exceeds the spike added concentration by a factor of 4 or more	75–125% unless the sample concentration exceeds the spike added concentration by a factor of 4 or more

Source of Criteria is EPA's SOW887 (Multimedia Inorganic Analysis)

protocols, a one-in-twenty frequency per matrix will receive a laboratory duplicate analysis (inorganics) and matrix spike/matrix spike duplicate analysis (organics). Field blanks will be collected at a frequency of one per 10 surface water samples per day, one per 20 groundwater samples and one per 20 soil/sediment samples. The one per 20 samples for field blanks for groundwater samples apply because dedicated sampling equipment will be utilized for the collection of samples from each monitoring well. Field duplicates will be collected once for every ten samples per matrix. Field duplicate results will be evaluated during ERM's data validation.

5.1.2 Sensitivity

Detection limits for analysis scheduled to be completed for this RI/FS are specified in Section 9.0 of this QAPP. Detection limits may be affected by matrix interferences, such as those caused by highly contaminated samples. In the case where low-level detection limits are not possible, sample/extract clean-ups or an alternate analytical method will be considered. If the limits are still not achievable, the applicability of the data, with respect to meeting the RI/FS objectives, will be evaluated. It should be noted that the compounds found at high levels in such contaminated samples will, in fact, be used to develop an EA, drive the clean-up process and accordingly, satisfy the project objectives.

5.1.3 Accuracy

Accuracy measures the bias in a measurement system which may result from sampling or analytical error. Sources of error that may contribute to poor accuracy are: laboratory error, sampling inconsistency, field and/or laboratory contamination, handling, matrix interference, and

preservation. Field and trip blanks, surrogate spikes, PE samples (dioxins and furans), as well as matrix spike QC samples will be used to measure accuracy for project samples.

5.1.4 Representativeness

Representativeness expresses the degree to which sample data represents the characteristics of the media or matrix from which it is collected. Samples that are considered representative are ones that are properly collected to accurately characterize the nature and extent of contamination at a general sample location. Therefore, an adequate number of sampling locations have been chosen and the sampling methods will be consistent. Representativeness will be measured by using the methods (e.g. sampling, handling, and preserving) specified in the Site Specific Sampling Plan which was prepared in accordance with the Statement of Work and state and federal guidelines for sampling of various media. Representativeness will also be measured by the collection of field replicates (VOAs). Comparison of the analytical results from field replicates will provide a direct measure of individual sample representativeness. Field replicates for volatile organics will be collected once for every ten samples per soil matrix. Duplicates will be collected for the balance of analytical parameters at the same frequency as above.

5.1.5 Comparability

Comparability is a qualitative parameter expressing the confidence with which data can be compared to one another. Comparability relies mainly upon precision and accuracy to be within appropriate QC limits before the data can be used for comparison of data sets. This will be accomplished through the consistent use of the analytical and sampling methods described in this document and the SSSP.

5.1.6 Completeness

Completeness is defined as the percentage of data that is judged to be valid to achieve the objectives of the RI compared to the total amount of data. Deficiencies in the data may be due to sampling techniques, or poor accuracy, precision, and laboratory error. While their deficiencies may effect certain aspects of the data, usable data may still be extracted from applicable samples. Completeness is of the utmost concern in relation to critical samples. Completeness goals with respect to the number of sample locations were developed during the Statement of Work process.

5.2 QA Objectives for Critical Samples

Critical samples are those data sets that must be valid in order to achieve project objectives. Their precision, sensitivity, accuracy, representativeness, completeness and comparability (PSARCC) are important, since they will be used to characterize specific media of concern (e.g., residential well water and background stations). Additional activity and more stringent QA objectives are listed in Table 5-4, in order to achieve PSARCC objectives.

Detection limits are also a concern for critical samples since several potential ARARs (applicable or relevant and appropriate requirements) and action levels for various matrices may be at or below standard detection limits. For the first round of samples, SAS low detection levels will be attempted for TCL analyses for residential wells and background ground water and surface water samples. Standard and non-standard detection levels are provided in Section 9.0 of this QAPP. Should first round results indicate the need to achieve lower detection levels, experience gained during first round analysis will be used to achieve lower detection

QAPP TABLE 5-4

CRITICAL SAMPLE SUMMARY
RUETGERS-NEASE SALEM SITE RI/FS

Sample Location	Matrix	Rationale	Activity
Well Cluster "I"	Ground Water	1) Determine existing background water quality at the site. 2) Provide basis for remediation levels.	1) Collect collocated sample from one well. 2) Complete two sample events. 3) 100%(1) completeness goal. 4) SAS low detection levels. (See Section 9)
Residential Wells	Ground Water	1) Ensure acceptable drinking water quality.	1) Collect two collocated samples during each sampling event. 2) 100%(1) completeness goal. 3) SAS low detection levels. (See Section 9)
MFLBC 1	Surface Water	1) Define background water quality conditions in MFLBC.	1) Collect one collocated sample at the upgradient location. 2) 100%(1) completeness goal 3) SAS low detection levels. (See Section 9)
Off-Site Soil (background)	Soil	1) Define natural or background soil conditions. 2) Provide remedial design Data	1) One collocated sample from one boring. 2) 100%(1) completeness goal.
MFLBC 1	Fish	1) Define background conditions	1) 100%(1) completeness goal.
Slanker Pond	Surface Water	1) Ensure acceptable water quality	1) 100%(1) completeness goal.

(1) Data with <100% completeness will be acceptable as long as the deficiency does not hinder project objectives.

limits, if possible. For non-standard compounds (i.e., mirex, kepone, photomirex), a study has been performed in order to facilitate the use of risk-based detection limits or the best detection limits achievable. The detection limits achieved during the MVS will be approved by the agencies prior to any work plan implementation.

5.3 Procedures for Monitoring PSARCC Parameters

PSARCC parameters will be monitored through the use of procedures which have been referred to in Section 5.1 and 5.2. These will include appropriate field blanks, trip blanks, laboratory method blanks, field and laboratory duplicates or replicates, matrix spikes, duplicate matrix spikes, surrogate spikes, performance evaluations (dioxins and furans), laboratory control samples, and a careful examination of all calibration and check standards. Laboratory control samples (LCS) and performance evaluation samples are samples containing a known or true value which the laboratory prepares and analyzes concurrently with project samples. They are of most use in judging analytical accuracy.

5.4 Field Measurements

Measurement data will be generated in many field activities that are incidental to collecting samples for analytical testing or unrelated to sampling. These activities include, but are not limited to, the following:

- Documenting time and weather conditions
- Locating and determining the elevation of sampling stations

- Performing geophysical surveys
- Determining pH, specific conductance and temperature of water samples
- Qualitative and semi-quantitative organic vapor screening of subsurface soil samples using an FID and/or PIDs
- Determining depths in a borehole or well
- Standard penetration testing
- Calculating pumping rates
- Verifying well development and pre-sampling purge volumes
- Performing bail-down recovery tests.

The general QA objective for field measurement data is to obtain reproducible and comparable measurements to a degree of accuracy consistent with the intended use of the data through the documented use of standardized procedures. The procedures for performing these activities and the standardized formats for documenting them are presented in the Site Specific Sampling Plan (Appendix A). A summary of the overall project Data Quality Objectives (DQOs) and the levels of DQOs is presented as Table 5-5 and Table 5-6 respectively.

TABLE 5-5
DQO SUMMARY FORM

1. Site Name Ruetgers-Nease Location Salem, Ohio	EPA Region V Phase RI 1 Phase 1 RI1, RI2, RI3, EA, FS, RA

VOLUME 2: QAPP
SECTION 5
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2. Media (Circle one)	Soil	Ground Water	Surface Water	Sediment	Air	Biota	Other

3. Use (Circle all that apply)	Site Characteristics(H&S)	Risk Assessment	Evaluate Alternatives	Engineer Design	PRP Determ.	Monitor Remedial Action	Other

4. Objective Determine transport mechanisms for site characterization and assessment of risk to human health and environmental receptors

5. Site Information
Area 44 acres
Depth to Ground Water 0-31 ft.
Ground Water Use Private potable wells within 1 mile, - also non-potable uses
Soil Types Glacial till over limestone and shale bedrock
Sensitivity Receptors Residents, Biota

6. Data Types

A. Analytical Data			B. Physical Data	
pH	Pesticides	Mirex	Permeability	Moisture
Conductivity	PCBs	Photomirex	Porosity	Soil Classification
VOA w/o ketones	Metals	Kepone	Grain Size	Specific Gravity
ABN	Cyanide	DPS	Atterburg Limits	
TCLP	Particulates			

7. Sampling Method (Circle Methods to be used)

Environmental	Biased	Grab	Non-Intrusive	Phased
Source	Grid	Composite	Intrusive	

8. Analytical Levels (Indicate Level(s) and Equipment & Methods)
Level 1 Field Screening - Equipment OVA, HNU, explosimeter
Level 2 Field Analysis - Equipment
Level 3 Non-CLP Laboratory Methods
Level 4 CLP/RAS - Methods
Level 5 Non-Standard DPS, BNA, Pest/PCB, VOA (w/o ketones), Mirex, Photomirex, Kepone

9. Sampling Procedures
Background 1 Upwind
Critical (List) On-site and downwind
Procedures Low-Vol pumps w/tenax, CMS, PUF cartridges, High-Vol for particulates

10. Quality Control Samples (Confirm or Set Standard)

A. Field	B. Laboratory
Collocated-5% or none	Reagent Blank-1 per analytical batch or
Replicate-5% or 10%	Replicate - 1 per analytical batch or
Field Blank-5% or 5%	Matrix spike-1 per analytical batch or
Trip Blank-1 per day or 5%	Other No matrix spikes for air

CONSULTANT ERM-Midwest
PREPARER Rock J. Vitale
PROJECT MANAGER Kenneth Richards
DATE 7/31/89

TABLE 5-5 (cont.)
DQO SUMMARY FORM

1. Site Name Ruetgers-Nease Location Salem, Ohio	EPA Region V Phase RI 1 Phase 1 RI1, RI2, RI3, EA, FS, RA

VOLUME 2: QAPP
SECTION 5
REV. 2-8.89

2. Media (Circle one)	Soil	Ground Water	Surface Water	Sediment	Air	Biota	Other

3. Use (Circle all that apply)	Site Characteristics (H&S)	Risk Assessment	Evaluate Alternatives	Engineer Design	PRP Determ.	Monitor Remedial Action	Other

4. Objective Assess Quality of surface water, risk to human health and biota by ingestion or contact, levels of contamination

5. Site Information
Area 44 acres Depth to Ground Water 0-31 ft.
Ground Water Use Private potable wells within 1 mile, - also non-potable uses
Soil Types Glacial till over limestone and shale bedrock
Sensitivity Receptors Residents, Biota

6. Data Types

A. Analytical Data			B. Physical Data	
pH	Pesticides	Photomirex	Permeability	Moisture
Conductivity	PCBs	Kepone	Porosity	Soil Classification
VOA	Metals	DPS	Grain Size	Specific Gravity
ABN	Cyanide	Methoxychlor	Atterburg Limits	
TCLP	Mirex	Flow + DO		

7. Sampling Method (Circle Methods to be used)

Environmental	Biased	Grab	Non-Intrusive	Phased
Source	Grid	Composite	Intrusive	

8. Analytical Levels (Indicate Level(s) and Equipment & Methods)
Level 1 Field Screening - Equipment pH meter, Conduc. meter, D.O. meter, Flow, Temp.
Level 2 Field Analysis - Equipment
Level 3 Non-CLP Laboratory Methods
Level 4 CLP/RAS - Methods VOA, BNA, Pest/PCB, DPS, Methoxychlor
Level 5 Non-Standard Mirex, Kepone, Photomirex

9. Sampling Procedures
Background Upgradient
Critical (List) All surface water samples
Procedures Grab samples using glass beaker, Kemmerer sampler for Slanker pond

10. Qualty Control Samples (Confirm or Set Standard)

A. Field	B. Laboratory
Collocated-5% or 1 at upgrad	Reagent Blank-1 per analytical batch or
Replicate-5% or 10%	Replicate - 1 per analytical batch or
Field Blank-5% or 10%	Matrix spike-1 per analytical batch or
Trip Blank-1 per day or 1/VOA cooler	Other Surrogate for Mirex per sample

CONSULTANT ERM-Midwest PREPARER Rock J. Vitale
PROJECT MANAGER Kenneth Richards DATE 7/31/89

TABLE 5-5 (cont.)
DQO SUMMARY FORM

1. Site Name Ruetgers-Nease Location Salem, Ohio	EPA Region V Phase RI 1 Phase 1 RI1, RI2, RI3, EA, FS, RA	VOLUME 2: QAPP SECTION 5 REV. 2-8.89

2. Media (Circle one)	Soil	Ground Water	Surface Water	Sediment	Air	Biota	Other

3. Use (Circle all that apply)	Site Characteristics(H&S)	Risk Assessment	Evaluate Alternatives	Engineer Design	PRP Determ.	Monitor Remedial Action	Other _____ _____

4. Objective Determine chemical composition to determine potential exposure to human health

5. Site Information
Area 44 acres Depth to Ground Water 0-31 ft.
Ground Water Use Private potable wells within 1 mile, - also non-potable uses
Soil Types Glacial till over sandstone and shale bedrock
Sensitivity Receptors Biota, Recreational user - consumers

6. Data Types

A. Analytical Data			B. Physical Data	
pH	Pesticides	Methoxychlor	Permeability	Moisture
Conductivity	PCBs	Mirex	Porosity	Soil Classification
VOA	Metals	Photomirex	Grain Size	Specific Gravity
ABN	Cyanide	DPS	Atterburg Limits	
TCLP	Kepone	3,4-DCNB		

7. Sampling Method (Circle Methods to be used)

Environmental	Biased	Grab	Non-Intrusive	Phased
Source	Grid	Composite	Intrusive	

8. Analytical Levels (Indicate Level(s) and Equipment & Methods)
Level 1 Field Screening - Equipment
Level 2 Field Analysis - Equipment Scale-weight, Ruler-size
Level 3 Non-CLP Laboratory Methods
Level 4 CLP/RAS - Methods VOA, BNA, Pest/PCBs, Methoxychlor, 3,4-DCNB, DPS
Level 5 Non-Standard Mirex, Kepone, Photomirex

9. Sampling Procedures
Background Several upstream specimens
Critical (List) All fish samples
Procedures Electroshock & fyke nets,sort,weight,homogenize lower-level,fillet-upper

10. Quality Control Samples (Confirm or Set Standard)

A. Field	B. Laboratory
Collocated-5% or none	Reagent Blank-1 per analytical batch or
Replicate-5% or 10%	Replicate - 1 per analytical batch or
Field Blank-5% or none	Matrix spike-1 per analytical batch or
Trip Blank-1 per day or 5%	Other Surrogate spikes for Mirex-1/sample

CONSULTANT ERM-Midwest PREPARER Rock J. Vitale
PROJECT MANAGER Kenneth Richards DATE 7/31/89

TABLE 5-5 (cont.)
DQO SUMMARY FORM

1. Site Name Ruetgers-Nease Location Salem, Ohio	EPA Region V Phase RI 1 Phase 1 RI1, RI2, RI3, EA, FS, RA	VOLUME 2: QAPP SECTION 5 REV. 2-8.89

2. Media (Circle one)	Soil	Ground Water	Surface Water	Sediment	Air	Biota	Other

3. Use (Circle all that apply)	Site Characteristics(H&S)	Risk Assessment	Evaluate Alternatives	Engineer Design	PRP Determ.	Monitor Remedial Action	Other

4. Objective Site characterization, determine risk to receptors, evaluate or level of contaminants in the aquifers under the site, study hydraulics and aquifer characteristics

5. Site Information
Area 44 acres Depth to Ground Water 0-31 ft.
Ground Water Use Private potable wells within 1 mile, - also non-potable uses
Soil Types Glacial till over sandstone and shale bedrock
Sensitivity Receptors Residents

6. Data Types

A. Analytical Data			B. Physical Data	
pH	Pesticides	Kepone	Permeability	Moisture
Conductivity	PCBs	Photomirex	Porosity	Soil Classification
VOA	Metals	DPS	Grain Size	Specific Gravity
ABN	Cyanide	3,4-DCNB	Atterburg Limits	
TCLP	Mirex	Dioxins/Furans		

7. Sampling Method (Circle Methods to be used)

Environmental	Biased	Grab	Non-Intrusive	Phased
Source	Grid	Composite	Intrusive	

8. Analytical Levels (Indicate Level(s) and Equipment & Methods)
Level 1 Field Screening - Equipment pH, Cond., D.O.-meter, OVA, HNU, temp.
Level 2 Field Analysis - Equipment
Level 3 Non-CLP Laboratory Methods
Level 4 CLP/RAS - Methods VOA, BNA, pest/PCBs, metals, diox/Fur, DPS & 3,4-DCNB
Level 5 Non-Standard VOA, BNA, pest/PCB, VOA (w/o ketones), Mirex, Photomirex, Kepone

9. Sampling Procedures
Background none
Critical (List) Well Cluster "I", Residential Wells, Country Club Well
Procedures Bailer/Bladder Pump after installation & develop., Tap-Resid. wells

10. Quality Control Samples (Confirm or Set Standard)

A. Field	B. Laboratory
Collocated-5% or	Reagent Blank-1 per analytical batch or
Replicate-5% or 10%	Replicate - 1 per analytical batch or
Field Blank-5% or 5%	Matrix spike-1 per analytical batch or
Trip Blank-1 per day or 1/VOA cooler	Other Surrogates for Mirex - 1 per sample

CONSULTANT ERM-Midwest PREPARER Rock J. Vitale
ROJECT MANAGER Kenneth Richards DATE 7/31/89

TABLE 5-5 (cont.)
DQO SUMMARY FORM

1. Site Name Ruetgers-Nease Location Salem, Ohio	EPA Region V Phase RI 1 Phase 1 RI1, RI2, RI3, EA, FS, RA	VOLUME 2: QAPP SECTION 5 REV. 2-8.89

2. Media (Circle one)	Soil	Ground Water	Surface Water	Sediment	Air	Biota	Other

3. Use (Circle all that apply)	Site Characteristics(H&S)	Risk Assessment	Evaluate Alternatives	Engineer Design	PRP Determ.	Monitor Remedial Action	Other _____ _____

4. Objective Determine transport mechanisms for site characterization and assessment of risk to human health and environmental receptors

5. Site Information
Area 44 acres Depth to Ground Water 0-31 ft.
Ground Water Use Private potable wells within 1 mile, - also non-potable uses
Soil Types Glacial till over limestone and shale bedrock
Sensitivity Receptors Residents, Biota

6. Data Types

A. Analytical Data			B. Physical Data	
pH	Pesticides	Kepone	Permeability	Moisture
Conductivity	PCBs	Photomirex	Porosity	Soil Classification
VOA	Metals	DPS	Grain Size	Specific Gravity
ABN	Cyanide	3,4-DCNB	Atterburg Limits	Hydraulic Conductivity
TCLP	Mirex	Dioxins/Furans		

7. Sampling Method (Circle Methods to be used)

Environmental	Biased	Grab	Non-Intrusive	Phased
Source	Grid	Composite	Intrusive	

8. Analytical Levels (Indicate Level(s) and Equipment & Methods)
Level 1 Field Screening - Equipment OVA and HNU
Level 2 Field Analysis - Equipment Permeameter, Conductivity, Seiomic, OVA (soil gas)
Level 3 Non-CLP Laboratory Methods Physical testing
Level 4 CLP/RAS - Methods VOA, BNA, Pest/PCBs, Dioxins/Furans, DPS, 3,4-DCNB
Level 5 Non-Standard Mirex, Kepone, Photomirex

9. Sampling Procedures
Background Multiple off-site and floodplain soils
Critical (List) off-site, on-site ponds
Procedures Composite intervals for non-volatiles, grab highest OVA interval for VOA

10. Quality Control Samples (Confirm or Set Standard)

A. Field	B. Laboratory
Collocated-5% or 1 per interval	Reagent Blank-1 per analytical batch or _____
Replicate-5% or 10%	Replicate - 1 per analytical batch or _____
Field Blank-5% or 5%	Matrix spike-1 per analytical batch or _____
Trip Blank-1 per day or 5% for VOA	Other Surrogate Spike for Mirex

CONSULTANT ERM-Midwest PREPARER Rock J. Vitale
PROJECT MANAGER Kenneth Richards DATE 7/31/89

TABLE 5-5 (cont.)
DQO SUMMARY FORM

1. Site Name Ruetgers-Nease Location Salem, Ohio	EPA Region V Phase RI 1 Phase 1 RI1, RI2, RI3, EA, FS, RA	VOLUME 2: QAPP SECTION 5 REV. 2-8.89

2. Media (Circle one)	Soil	Ground Water	Surface Water	Sediment	Air	Biota	Other

3. Use (Circle all that apply)	Site Characteristics(H&S)	Risk Assessment	Evaluate Alternatives	Engineer Design	PRP Determ.	Monitor Remedial Action	Other

4. Objective Determine stream sediment and pond sediment composite. Determine risk to biota, recreational users and possible transport mechanisms

5. Site Information
Area 44 acres Depth to Ground Water 0-31 ft.
Ground Water Use Private potable wells within 1 mile, - also non-potable uses
Soil Types Glacial till over limestone and shale bedrock
Sensitivity Receptors Biota, Recreational Users, Residents

6. Data Types

A. Analytical Data			B. Physical Data	
pH	Pesticides	Kepone	Permeability	Moisture
Conductivity	PCBs	Mirex	Porosity	Soil Classification
VOA	Metals	Photomirex	Grain Size	Specific Gravity
ABN	Cyanide		Atterburg Limits	
TCLP	DPS			

7. Sampling Method (Circle Methods to be used)

Environmental Source	Biased	Grab	Non-Intrusive	Phased
	Grid	Composite	Intrusive	

8. Analytical Levels (Indicate Level(s) and Equipment & Methods)
Level 1 Field Screening - Equipment
Level 2 Field Analysis - Equipment
Level 3 Non-CLP Laboratory Methods
Level 4 CLP/RAS - Methods VOA, BNA, Pest/PCBs, DPS, Methoxychlor
Level 5 Non-Standard Kepone, Mirex, Photomirex

9. Sampling Procedures
Background Upstream seds
Critical (List) Slanker Pond, upstream & all downstream samples
Procedures Stream 4-6" Cores, Dredge-Pond, Composite non-volatile aliquots

10. Quality Control Samples (Confirm or Set Standard)

A. Field	B. Laboratory
Collocated-5% or	Reagent Blank-1 per analytical batch or
Replicate-5% or 10%	Replicate - 1 per analytical batch or
Field Blank-5% or 5%	Matrix spike-1 per analytical batch or
Trip Blank-1 per day or 5% for VOA	Other Surrogate spike for Mirex

CONSULTANT ERM-Midwest PREPARER Rock J. Vitale
PROJECT MANAGER Kenneth Richards DATE 7/31/89

QAPP TABLE 5-6
LEVELS OF DATA QUALITY OBJECTIVES

SAMPLE MATRIX	FIELD PARAMETERS	LABORATORY PARAMETERS	LEVEL OF DQOs
Soil	OVA HNu- (Screening)	-	I
	Hydraulic Conductivity, OVA- (Soil Gas)		II
		Soil Physical Testing	III
		CLP RAS VOA +15, CLP RAS BNA +25, CLP Pesticides & PCBs, TAL Inorganics, CLP Dioxins/Furans, DPS & 3,4-DCNB	IV
		Mirex, Kepone, Photomirex	V
Sediment	-	CLP RAS VOA +15, CLP RAS BNA +25, CLP Pesticides & PCBs, DPS & 3,4-DCNB, Methoxychlor	IV
		Mirex, Kepone, Photomirex	V
Surface Water	OVA, HNu pH, Conductivity, DO & Temperature		I
		-	
		CLP RAS VOA +15, CLP RAS BNA +25, CLP Pesticides & PCBs, DPS, Methoxychlor	IV
		Mirex, Kepone, Photomirex	V
Groundwater	pH, Conductivity, DO & Temperature		I
		-	
		CLP RAS VOA +15, CLP RAS BNA +25, CLP RAS Pesticides & PCBs, TAL Inorganics, CLP Dioxins/Furans, DPS, 3,4-DCNB	IV
		TCL VOAs +15 (Low Detection Limits), TCL BNAs +25 (Low Detection Limits), Mirex, Kepone, Photomirex	V
Biota	Weight,Size		II
		CLP RAS VOA +15, CLP RAS BNA +25, TCL RAS Pesticides & PCBs, DPS, Methoxychlor	IV
		Mirex, Kepone, Photomirex	V
Air	OVA,HNu	-	I
		TCL RAS VOA+15 (minus 4 Ketones), TCL RAS BNA +25, TCL RAS Pesticides & PCBs, DPS, Particulates	IV
		Mirex, Kepone, Photomirex	V

Sampling utensils such as trowels, scoops, hand augers, ı triers are routinely used to collect surface soil samples a depth of 6 inches. Only stainless steel utensils will used for sample collection. Once a sample has been llected using any one of these devices, the sample will be ansferred directly in appropriate laboratory-supplied ttleware for volatile organic analysis. The remainder will transferred into a clean stainless steel bowl, mixed oroughly and will then be placed into the appropriate ttleware for organic and inorganic analysis as appropriate.

ı.2 Subsurface Soils

6.2.2.1 Soil Borings

Subsurface soil samples will be collected from soil rings. Soil borings will be completed using a bucket ger, power auger or a drilling rig. Off-site samples will collected from ground level to 0.5 feet BLS and from 0.5 et BLS to 3.5 feet BLS directly from the bucket auger or om the split-spoon sampler if a drilling rig is used. A ction of the sample will be transferred in the appropriate ttleware for volatile organic analysis using a clean ainless steel spatula. The remainder will be transferred o a clean stainless steel bowl, mixed thoroughly and will en be placed into the appropriate bottleware for organic ı inorganic analysis as appropriate.

On-site soil borings in Ponds 1,2,3,4,7 and the il/sludge area beside Pond 2 will be conducted using a ill rig. Samples will be collected from these borings in 3 ot intervals. These borings will extend to bedrock or 9 et into native soil below the pond bottoms whichever is ss.

To determine which subinterval of each 3-foot interval will be submitted to the laboratory for volatile organics analysis, samples will be screened for volatile organics using an FID or PID. A portion of the sample from each 1-foot subinterval containing the highest FID or PID reading will be placed into a laboratory-supplied glass bottle for volatile organic analysis. If there is no observed difference in FID and PID readings, the second 1-foot subinterval will be collected as a grab for analysis for volatile organics and library searches for up to 15 compounds. The remainder of the entire 3-foot interval will be composited in a stainless steel bowl and placed into sample bottles for analysis for CLP non-volatile organics plus a library search for up to 25 additional compounds plus mirex, kepone, photomirex, and DPS, as well as for inorganic analysis as required.

6.2.2.2 Test Pits

Thirty test pits are planned on-site and in the swamp with additional pits to be located along the railroad tracks. The number and location of test pits along the railroad tracks will be based upon results of the soil gas survey.

A backhoe will open the test pits, and samples will be collected in undisturbed soils from the walls or from the base of the excavation. Equal volumes of each layer will be homogenized on an enamel pan with a stainless steel trowel prior to placing the appropriate volume in the sample container. The sample collected for volatile organic analysis will be collected as a grab sample from the center depth to avoid VOC loss during the homogenization process.

Sample acquisition of the test pit material will be dependent on the conditions encountered during digging the test pit. If safety conditions allow close proximity to the

test pit, and depths are not too great, a stainless steel bucket auger will be used to obtain the sample from the desired depth/location. If the above is not allowed, the sample will be obtained from material brought up by the bucket of the backhoe from the required depth. Samples will be obtained using a clean stainless steel spatula and transferred into appropriate bottleware.

6.2.3 Surface Water and Sediment Sampling

Most of the sediment samples will be collected using clean stainless steel utensils and dewatered as much as possible. Samples will be collected from the stream bottom to a depth of six to eight inches. Samples for volatile organic analysis will be placed directly in the appropriate bottleware. The remaining sample aliquot will be placed in a stainless steel bowl, thoroughly homogenized and placed into the appropriate bottleware.

Sediment samples collected from the bottom of Slanker Pond will be obtained using a clean Ponar^{R} dredge sampler from a small boat. This dredge is a clam shell scoop and is lowered into the pond on dedicated nylon rope until it reaches the bottom. The dredge closes and collects the sediment on contact with the pond bottom. The dredge is retrieved, opened and the sediment is transferred to the appropriate bottleware using a clean stainless steel spatula filling the containers for volatile organic analysis first.

At some of the sediment sampling locations, a surface water sample will be collected. Surface water sampling will proceed from the farthest downstream location and proceed to upstream locations to minimize potential cross-contamination from suspended material in the stream from sampling activities. Surface water samples will be collected before sediment samples. Dependent upon depth and stream velocity,

samples will be either collected directly into the appropriate sample containers at a point upstream of the samplers position or through the use of a long-handled polyethylene grab sampler from the bank of the stream.

Stream velocity and flow characteristics, pH, conductivity, DO and temperature will be noted at each sampling location. Stream velocity will be measured using a velocity meter (e.g. Pygmy). Flow will be determined based on the calculated cross-section area at the stream sampling station.

A water sample collected from the Slanker Pond will be obtained using a Kemmerer[R] sampler. The sampler will be slowly lowered to the mid-depth of the water column and allowed to remain at that depth for several minutes. It will them be retrieved and the sample transferred to the appropriate bottleware, filling the vials for volatile organic analysis first.

6.2.4 Groundwater

Groundwater samples will be collected from numerous monitoring wells and several residential wells.

Prior to purging and sampling, the well protective casing, lock and apron will be inspected for damage or signs of tampering. Static water depth and total depth will be measured using a decontaminated probe with permanent depth markings. The static and total depth will be used to calculate the volume of standing water. One bailer of ground water from the top of the water column and one from the bottom of the well will be collected to detect immiscible layers before purging.

Where possible, at least three volumes will be removed prior to sampling. Less than this amount will be purged only if all standing water is removed before three volumes are purged.

Bailing or pumping methods will be used during purging to ensure that all standing water is removed. Purging will take place at the top of the water column where possible. Suction hoses for centrifugal pumps will be capped with a "foot valve" to prevent purged water from flowing back into the well as the hose is removed.

Sampling whenever possible will take place on the same day after purging procedures are completed. Where the well is purged dry, purging procedures are described in SSSP Appendix A, Section 2.1.

Samples will be collected from monitoring wells using Teflon^{R} bailers dedicated to each well. The first bailer withdrawn from the well will be checked for temperature, pH and specific conductance. Subsequent bailer volumes collected from within the screened interval will be used to directly fill sample bottles. Bottles intended for volatile organic compound analysis will be filled first, followed by the extractable organic fraction and finally the inorganic analysis bottles filled last.

Samples will be collected from five residential wells and the well at the country club. If possible, sampling points will be located at a water tap closest to the pump prior to any water softeners. Prior to sample collection, the faucet aerator, if any, will be removed and any in-line water conditioning units turned off. If the well is active, an appropriate volume of water in the supply line preceding the tap will be removed. An estimated three well volumes will be removed.

The sample will be collected after stabilization of temperature, pH, and conductivity. The sample will be collected directly from the tap into the sample containers.

If artesian flow conditions exist at any of the ground water sampling locations, the well will be allowed to flow until the appropriate amount of water is removed from the well prior to sample collection.

6.2.5 Fish Sampling

Fish species representing two trophic levels, upper and lower, will be collected from 28 sampling stations using one or more of the following types of equipment; electroshocker, fyke nets, seine nets, and gill nets. The fish collected at each station will be sorted by species, counted, weighed, measured, and recorded. One representative species from each trophic level will be selected for submission to the laboratory to be analyzed. The representative species, in order of preference is as follows; upper trophic level fish: bass, bluegill, crappie, and catfish; lower trophic level fish: carp, sucker, chubs, and bullheads. The upper trophic level species will be filleted in the field with skins on, using a stainless steel knife in a stainless steel pan. The fillets will be combined and wrapped accordingly. The lower trophic level fish will be packaged as whole fish. Total fish weight will be a minimum of 150 grams per species per sampling location.

6.2.6 Air Sampling

In addition to the sampling of air (and particulates) for quantitative laboratory analysis of various organic pollutants, field monitoring of air will also be performed utilizing FID and PID (soil gas survey) and an explosimeter.

The specific sampling protocol for these field measurements are provided in the Site Specific Sampling Plan (SSSP) (Volume 3).

6.2.6.1 Century OVA Meter 128 and HNU Model P1 101 Photoionization Meter

A survey of the Site using flame ionization (FID) and photoionization (PID) detectors will be completed prior to the initiation of any field work. The survey will define specific work zone boundaries identified in the Health and Safety Plan, and identify areas with elevated levels of volatile organic compounds that may also require inclusion as an exclusion area. Measurements of VOCs at ground surface and three feet above ground surface using the PID and FID will be made at 100 foot intervals along the site boundaries at stations, around each pond at stations every 50 feet, and at several locations within the proposed clean support zone. The area between discrete sampling points will be walked with the instrument operating in order to detect any unexpected elevated levels of volatile compounds. The work and exclusion zones around each test pit excavation, boring or well drilling location will be screened during site work using a PID and/or FID to determine proper health and safety protection. All measurements at discrete monitoring stations will be recorded in the field log book along with location, time and area weather condition. If no elevated levels of volatiles are detected during the walk through between discrete monitoring stations using the organic vapor meter (FID) and/or the photoionization instrument (PID), only the concentration measured at discrete monitoring stations will be individually recorded in the field notebook. Subsurface soil borings will be screened with an OVA in order to provide information as to what soil boring interval will be selected for quantitative analysis. Weather condition (wind speed, wind direction, temperature, etc.) will also be obtained from

a local weather bureau. Wind rose data collected at the nearest weather station will be included in the RI report.

6.2.6.2 Tenax, XAD-2, and CMS Trap Sampling

There will be three phases to the volatile/semivolatile sampling program. In general, volatiles will be trapped by drawing ambient air through a cartridge containing one to two grams of Tenax (EPA Method TO1 "Method for the Determination of Volatile Organic Compounds in Ambient Air Using Tenax Adsorption and for Gas Chromatography/Mass Spectometry"). Highly volatile compounds will be trapped by drawing ambient air through a cartridge containing approximately 0.4 grams of carbon molecular sieve (CMS) adsorbent. Semivolatile compounds will be trapped by drawing ambient air through XAD-2 adsorbent traps. The sample flow rate and sample volume (and thereby sampling time) will be dependent upon the ability of the adsorbent to trap the organic compounds (retention volume) and other properties of the organic compound (polarity, boiling point, etc.). Enseco will conduct method validation studies for this project. Flow rates will be maintained by flow controllers. Appropriate sampling pumps will be utilized. At a minimum samples will be collected at the following sites:

1. Off-site upwind
2. Off-site downwind
3. Pond 1 downwind
4. Pond 2 downwind
5. Pond 7 downwind
6. Between the leachate collection system and the railroad tracks.

On-site samples will be collected at a height of 3 feet while the sampling height will be at a level of 5 to 6 feet above ground level for off-site samples (ambient breathing zone).

6.2.6.3 High Volume Particulate Sampling

Air is drawn into a covered housing and through a filter by means of a high-flow-rate blower at a flow rate (1.13 to 1.70 m^3/min.; 40 to 60 ft^3/min.) that allows suspended particles having diameters of less than 100 um. to pass to the filter surface. Particles within the size range of 100 to 0.1 um. diameter are ordinarily collected on glass fiber filters. The mass concentration of suspended particulates in the ambient air (ug/m^3) is computed by measuring the mass of collected particulates and the volume of air sampled.

When the sampler is operated at an average flow rate of 1.70m^3/min. (60 ft^3/min.) for 24 hours, an adequate sample will be obtained even in an atmosphere having concentrations of suspended particulates as low as 1 ng/m^3. If particulate levels are unusually high, a satisfactory sample may be obtained in 6 to 8 hours or less. For determination of average concentrations of suspended particulates in ambient air the sampling period will be 24 hours.

Weights are determined to the nearest 0.1 milligram, airflow rates are determined to the nearest 0.01 m^3/min. (1.0 ft^3/min.), times are determined to the nearest 2 minutes, and mass concentrations are reported to the nearest microgram per cubic meter.

Samples will be collected by opening the shelter, installing a numbered, preweighed, glass-fiber filter in position (rough side up), replacing the faceplate without disturbing the filter, and securely fastening the wing nuts.

After the 24-hour sampling period, the filter is carefully removed from the holder, touching only the outer edges. The filter is folded lengthwise so that only surfaces

with collected particulates are in contact, and the filter is placed in a manila folder. The filter number, sampler location, and any other factors, such as meteorological conditions or razing of nearby buildings, is recorded on the folder.

The objective of this sampling (particulate) is to obtain particulate data which may be used in a health risk assessment study of the site; therefore, samples will be taken from a height of 3 feet on-site, and 5 to 6 feet off-site. Representative areas of the site will be sampled; that is, the sampling will attempt not to be biased toward worst or best case scenarios. An upwind sample will be obtained, as will downwind samples near the boundary lines.

6.2.6.4 Organochlorine Pesticide and PCB Sampling

Sampling utilizing instrumentation and procedures specified in EPA Method TO4 (Method for the Determination of Organochlorine Pesticides and Polychlorinated Biphenyls in Ambient Air) will be used. Samples will be collected from the six sampling sites denoted previously. In this method, a modified high volume sampler consisting of a glass fiber filter with a polyurethane foam (PUF) backup adsorbent cartridge is used to collect ambient air at a rate of 200 to 280 liters/minute. The filter and PUF cartridge will each be analyzed for TCL organochlorine pesticides and PCBs as well as mirex, photomirex and kepone. Two sampling units will be used during sampling activities.

6.3 Sample Packing, Handling and Shipment

After a given sample has been collected, a self-adhesive label will be prepared with indelible ink and affixed to each container. At a minimum, the sample label will contain:

- The Investigation Name (Ruetgers-Nease - RI/FS)
- Field Sample Number (Section 5.2 of SSSP)
- Sample Description
- Date and Time Collected
- Sampler's Initials
- Testing Required
- Preservatives Added.

Immediately after sample collection, each labeled sample container will be sealed in an individual plastic bag. Samples will then be placed immediately into an insulated cooler with ice or ice packs for shipment to the laboratory. Fish samples will be wrapped in clean aluminum foil, sealed in a plastic bag and packed in a separate cooler with dry ice.

Sorbent traps (Tenax, XAD-2, and CMS) will be stored in individual glass culture tubes. These will then be stored in friction top cans containing a layer of charcoal on the bottom. These cans will be placed in a separate cooler for shipment to the laboratory.

The PUF plug is placed into the glass sampling cartridge using polyester gloves after initial cleaning. The module is wrapped in clean aluminum foil, placed in a labeled container and tightly sealed. The process is repeated after sampling. The filter is stored in cleaned petri dishes.

The hi-volume particulate filters are stored in manila folders prior to and after sampling.

Chain-of-custody aspects of sample handling are discussed in detail in Section 6.6. These records are sealed in ziplock bags to protect them against moisture and will be taped to the underside door of appropriate sample coolers. Each cooler will contain sufficient ice and /or ice packs to insure that proper temperature is maintained, and will be packed in an appropriate packing material (e.g. vermiculite) to prevent damage to sample containers.

The sample coolers will be shipped by an overnight courier according to current U.S. DOT regulations.

6.4 Sample Containers, Preservatives and Holding Times

The specific containers, preservatives and holding times that will be utilized for this investigation are presented on Table 6-1.

6.5 Preparation of Sampling Equipment and Containers

6.5.1 Decontamination of Sampling Equipment

This section describes procedures for decontaminating drilling and sampling equipment. Detailed personnel decontamination procedures are discussed in Section 9.0 of the Health and Safety Plan. Decontamination protocols will be strictly adhered to in order to minimize the potential for cross-contamination between sampling locations and contamination of off-site areas.

QAPP Table 6-1
Sample Containers, Preservatives, and Holding Times+

Analysis	Matrix	Container And Volume[1]	Preservative	Maximum Holding Time*[2]
Semivolatiles, Dichloronitro benzene and Diphenyl Sulfone	Aqueous	(2) Glass, amber 1/2 gal with teflon lined caps (filled to shoulder)	None, cool, 4°C	5 days extn./40 days anal.
	Solid	(1) Glass, amber 8 oz. jar with teflon lined caps	None, cool, 4°C	10 days extn./40 days anal.
	Fish	Aluminum foil	None, cool, 4°C	10 days extn./40 days anal.
	Air	XAD canister	None, cool, 4°C	10 days extn./40 days anal.
Dioxins and Furans	Aqueous	(2) Glass, amber, 1/2 gal. with teflon lined caps	pH 6-8 with either NaOH or H_2SO_4, cool, 4°C	5 days extn./40 days anal.
	Solid	(1) Glass, amber, 8 oz. with teflon lined caps	None, cool, 4°C	10 days extn./40 days anal.
Volatiles	Aqueous	(3) 40 ml VOA vials teflon lined caps (no headspace)[3]	HCl to pH2, cool, 4°C	10 days anal. (7 days anal.[4])
	Solid	(2) 120 ml glass vials with teflon lined caps (filled to shoulder)	None, cool, 4°C	10 days anal.
	Fish	Aluminum foil	None, cool, 0°C	10 days anal.
	Air	Tenax and CMS Tubes in cans with carbon	None, cool, 4°C	10 days anal.
Pesticides/PCBs	Aqueous	(2) Glass, amber, 1/2 gal. with teflon lined caps	pH adjusted to 5-9, cool, 4°C and preserve with 0.008% $Na_2S_2O_3$	5 days extn./40 days anal.
	Solid	(1) Glass, 8 oz. jar with teflon lined caps	None, cool, 4°C	10 days extn./40 days anal.
	Fish	Aluminum foil	None, cool, 0°C	10 days extn./40 days anal.
	Air	Foam canister (PUF)	None, cool, 4°C	10 days extn./40 days anal.
Mirex, Photomirex, Kepone	Aqueous	(2) Glass, amber, 1/2 gal. with teflon lined caps	None, cool, 4°C	5 days extn./40 days anal.
	Solid	(1) Glass, 8 oz. jar with teflon lined caps	None, cool, 4°C	10 days extn./40 days anal.
	Fish	Aluminum foil	None, cool, 0°C	10 days extn./40 days anal.
	Air	Foam canister	None, cool, 4°C	10 days extn./40 days anal.
Metals, except Mercury	Aqueous	(1) Polyethylene, 1 liter with teflon lined caps	HNO_3 to pH<2	6 mos.
	Solid	(1) Glass, 8 oz. jar with teflon lined caps	None, cool, 4°C	6 mos.
Cyanide	Aqueous	(1) Polyethylene, 1 liter with teflon lined caps	NaOH to pH>12	14 days
	Solid	(1) Glass, 8 oz. jar with teflon lined caps	None, cool, 4°C	14 days
Mercury	Aqueous	(1) Polyethylene, 1 liter with teflon lined caps	HNO_3 to pH<2	26 days
	Solid	(1) Glass, 8 oz. jar with teflon lined caps	None, cool, 4°C	26 days

NOTES:

* From date of validated time of sample receipt (VTSR) at the analysis laboratory

+ Reference: U.S. EPA's CLP Statement of Work for Organics, Inorganics and Dioxins/Furans (SOW 887).

[1] Triplicate the sample volume must be submitted to the laboratory for samples designated as matrix samples/matrix sample duplicates.

[2] All samples will be shipped by overnight delivery to the laboratory except on occasions of late night or Sunday sampling. On these occasions, refrigeration will occur on-site for any samples requiring this preservation and will be shipped at the next available time. However, the next available sample shipment day will not exceed more than one day after sample collection. It is understood that samples shipped out on Saturday will not be received by the laboratory until Monday.

[3] The VOA samples collected from the residential well will require four 40 ml VOA vials with teflon lined caps.

[4] The VOA holding time for residential wells will be 7 days.

extn: extraction
anal: analysis

6.5.1.1 General Considerations

The following general procedures will be adhered concerning decontamination efforts:

1. All decontamination and subsequent use of decontaminated equipment will be documented in a field book.

2. If visual signs such as discoloration indicate that decontamination was insufficient, the equipment will again be decontaminated. If the situation persists, the equipment will be taken out of service until the situation can be corrected.

3. All spent wash and rinse waters will be collected and stored in an on-site tank pending analysis and proper disposal.

4. Verification of the sampling equipment cleaning procedures will be documented by the collection of field blanks and trip blanks.

5. Drill cuttings from off-site locations will be containerized and handled according to procedures described in Section 9.4 of the Health and Safety Plan (Volume 4).

6. All properly decontaminated equipment will be stored in plastic bags when not in use.

7. All fluids and solids generated from sample location decontamination will be transported and disposed according the Hazardous Materials

Handling procedures described in the Health and Safety Plan, Section 9.4.

8. Personnel and equipment will proceed directly to the decontamination pad between sample locations and at the end of each day.

6.5.1.2 Heavy Equipment

Drill rigs, backhoe buckets, and appropriate other heavy equipment will be decontaminated prior to the commencement of field activities, between each sample location, and after the completion of field activities at the on-site decontamination pad. Decontamination procedures will be as follows:

1. Remove all loose soil from equipment with a brush.

2. Steam or high pressure wash using non-phosphate soap.

3. Potable water rinse.

6.5.1.3 Drilling Equipment

Drilling equipment (i.e., rods, auger flights, bits, casings) will be cleaned between each boring location and sample. Decontamination procedures will be as follows:

1. Remove all loose soil.

2. Steam or high pressure wash using non-phosphate soap.

3. Potable water rinse.

All wash water will be stored in an on-site tank pending proper disposal. All soil will be containerized.

All cleaned equipment will be transported and stored in plastic sheets.

6.5.1.4 Sampling Equipment

Equipment used to collect environmental samples will be cleaned prior to its initial use and between each sample location and after the final use. All equipment will be transported and stored in plastic sheets.

6.5.1.4.1 Soil and Sediment Sampling Equipment

Soil sampling equipment will be decontaminated at the sample locations. Equipment that will be cleaned will include: split spoons, Shelby Tubes, hand augers, stainless steel scoops/trowels, and stainless steel compositing containers. Specific procedures are as follows:

Inorganics	Organics
1. Non-phosphate detergent wash	1. Non-phosphate detergent wash
2. 0.1 N HCL rinse	2. Tap water rinse
3. Tap water rinse	3. Deionized/Distilled water rinse
4. Deionized water rinse	4. Methanol rinse
5. Air dry	5. Pesticide quality hexane rinse
	6. Methanol rinse

7. Four rinses with deionized/distilled water

8. Air dry

6.5.1.4.2 Ground Water and Surface Water Sampling

Equipment used for ground water and surface water sampling will be decontaminated before sampling activities begin and between each sample location if dedicated equipment is not used. This equipment will include: pumps, hoses, glass beakers and bailers. The following procedures will be used for decontaminating equipment:

Inorganics

1. Non-phosphate detergent wash
2. 0.1 N HCL rinse
3. Tap water rinse
4. Deionized water rinse
5. Air dry

Organics

1. Non-phosphate detergent wash
2. Tap water rinse
3. Deionized/Distilled water rinse
4. Methanol rinse
5. Pesticide quality hexane rinse
6. Methanol rinse

7. Four rinses with deionized/distilled water

8. Air dry

6.5.1.4.3 Fish Sampling Equipment

Sampling equipment such as the fish shocker, fyke net, seine net, and gill nets will be rinsed off with potable water after each station to minimize cross-contamination.

6.5.1.4.4 Soil Gas Probe

The soil gas probe used during the survey will be decontaminated using the procedures specified below:

1. Remove loose soil.

2. Non-phosphate soap wash.

3. Potable water rinse.

4. Deionized water rinse.

5. Field scan with FID.

6.5.1.4.5 Temperature, Conductivity, pH, and Depth to Water Probes

These probes used during ground water and surface water sampling will be decontaminated via the procedures specified below:

1. Wash with non-phosphate detergent solution.

2. Potable water rinse.

3. Deionized water rinse.

All equipment will be transported and stored in plastic sheeting.

6.5.1.5 Monitoring Well Materials

Well screens, riser pipes, and outer casings will be steam cleaned at the decontamination area, wrapped in plastic sheeting, and stored in the on-site warehouse building.

6.5.1.6 Electronic Equipment

Electronic equipment such as PIDs, FIDs, explosimeters, and portable air pumps will be decontaminated prior to their initial use and at the end of each day. The procedure for decontaminating this equipment is as follows:

1. Remove particulate contamination.

2. Wipe down with clean damp cloth (deionized water).

3. Air dry.

Equipment will be wrapped in plastic and stored in the office trailer when not in use.

6.5.2 Preparation of Sample Containers

Enseco will provide appropriately prepared sample containers and coolers for this project. All containers for volatile organics, extractable organics, and inorganics are purchased, precleaned and prebaked from I-Chem of Hayward, CA. Enseco purchases 200 Series bottles which are cleaned

for the specific analyses according to U.S. EPA specifications. Preservation using ultra-pure reagents, if necessary, will be done prior to container shipment. The following are preservation techniques applicable to this project.

Volatile organics -- water 0.2 ml 1:1 HCL (pH $\leq$2)

Metals -- water 2.5 mls 1:1 HNO_3 (pH $\leq$2)

Cyanide -- water 7-10 pellets NaOH (pH $\geq$12)

6.5.2.1 Air Tube Preparation

For initial cleanup, the PUF plug is placed in a Soxhlet extractor and extracted with acetone for 14-24 hours at approximately 4 cycles per hour. If cartridges are reused, 5% diethyl ether in n-hexane will be used as the cleanup solvent.

The extracted PUF is placed in a vacuum oven connected to a water aspirator and dried at room temperature for approximately 4 hours. The PUF is placed into the glass sampling cartridge using polyester gloves. The module is wrapped with aluminum foil, placed in a labeled container and tightly sealed.

6.5.2.1.1 Method TO1

Bulk Tenax is placed in a glass extraction thimble and held in place with a plug of clean glasswool. The resin is then placed in the soxhlet extraction apparatus and extracted sequentially with methanol and then pentane for 16-24 hours (each solvent) at approximately 6 cycles/hour. Glasswool for cartridge preparation will be cleaned in the same manner as Tenax.

The extracted Tenax is immediately placed in an open glass dish and heated under an infrared lamp for two hours in a hood. Care will be exercised to avoid overheating of the Tenax by the infrared lamp. The Tenax is then placed in a vacuum oven (evacuated using a water aspirator) without heat for one hour. An inert gas (helium or nitrogen) purge of 2-3 ml/minute is used to aid in the removal of solvent vapors. The oven temperature is then increased to 110°C, maintaining inert gas flow and held for one hour. The oven temperature control is then shut off and the oven is allowed to cool to room temperature. Prior to opening the oven, the oven is slightly pressurized with nitrogen to prevent contamination with ambient air. The Tenax is removed from the oven and sieved through a 40/60 mesh sieve (acetone rinsed and oven dried) into a clean glass vessel.

The cartridge is packed by placing a 0.5-1cm glasswool plug in the base of the cartridge and then filling the cartridge to within approximately 1 cm of the top. A 0.5-1cm glasswool plug is placed in the top of the cartridge.

The cartridges are then thermally conditioned by heating for four hours at 270°C under an inert gas (helium) purge (100-200 ml/min).

6.5.2.1.2 <u>Method TO2</u>

The carbon molecular sieve cartridges are conditioned for initial use by heating at 400°C overnight (at least 16 hours) with a 100 mL/minute purge of ultra-pure nitrogen. Reused cartridges need only to be heated for 4 hours.

For cartridge conditioning ultra-pure nitrogen gas is passed through a gas purifier to remove oxygen, moisture and organic contaminants. The nitrogen supply is connected to

the unmarked end of the cartridge and the flow adjusted to 50 mL/minute using a needle valve. The gas flow from the inlet (marked) end of the cartridge is vented to the atmosphere.

After 10-16 hours of heating (for new cartridges) the Variac is turned off and the cartridge is allowed to cool to 30°C, under continuing nitrogen flow.

The exit end of the cartridge is capped and then the entire cartridge is removed from the flow line and the other endcap immediately installed. The cartridges are then placed in a metal friction top (paint) can containing 2 inches of granulated activated charcoal (to prevent contamination of the cartridges during storage) in the bottom, beneath a retaining screen. Clean paper tissues (e.g. Kimwipes) are placed in the can to avoid damage to the cartridges during shipment.

6.5.2.1.3 XAD-2

The XAD-2 is placed in a glass extraction thimble and held in place with a plug of clean glasswool. The resin is then placed in the soxhlet extraction apparatus and extracted with methylene chloride for 16-24 hours at 6 cycles/hour. Glasswool is cleaned in the same manner. XAD-2 is stored in methanol until the tube is packed.

6.6 Documentation of Sampling Activities

In order to ensure that all pertinent information and data collected during the RI/FS are documented completely and correctly, the following procedures and protocols described in the following sections will be implemented.

6.6.1 Log In/Log Out Record

A sign-in/sign-out log will be kept at the office trailer for use by authorized personnel. Unauthorized personnel will not be granted access on site unless approved by Ruetgers-Nease, in advance. The record will contain at a minimum: date, name, organization, and entrance/exit times.

6.6.2 Field Notebooks

All information pertinent to the field investigation will be recorded in bound and numbered field notebooks. Each team member will be assigned an individual notebook. Field records should at a minimum contain the following information:

1. Date
2. Time of each data entry
3. Description of work being performed that day
4. Names and affiliations of all personnel at location
5. Weather conditions on-site
6. Location and type of activity (monitoring well, surface water sample, etc.)
7. Sample or Boring Methods in use
8. Visual observations
9. Pertinent field data (pH, specific conductance, temperature, and any other field measurements such as from a FID or Explosimeter)

10. Serial numbers, if any, on seals and transportation cases

11. Name of field custodian

12. Photographs taken, including date, time, direction faced, description of subject or activity, sequential numbers of the photos and film roll numbers.

All field notebooks will be standard engineering hardbound books. All field notebooks will be photocopied so that copies of field notes can be kept. An example of some typical field notebook entries are presented on Figure 6-1.

6.6.3 Sample Log Book

Specific sample information will be compiled into one sample log notebook. The following information will be included in the sample log notebook:

1. Unique Sample number (See Section 5.2 of SSSP)

2. Sample Date

3. Sampler's Initials

4. Sample Matrix (soil, water, etc.)

5. Number of samples

6. Analyses previously performed

7. Current analyses requested

8. Date shipped to the lab

9. Method of shipment

FIGURE QAPP 6-1

Typical Logbook Entries

Acme Waste Company

Joe Smith-EPA Region V	(315) 672-3396
Marin Astare-PADER	(717) 629-3396
Peter Shoperton -Site Owner	(215) 276-9377
Paul Detchic-Project Manager	(215) 687-9560
Joe Perryton-Geologist	(215) 687-9560
Pauline Jenlan-Sampler	(215) 687-9560

Checked out traffic reports 1600-1609 with 3 chain of custody 8/25/87 _ bottles delivered by lab courier 8/22/87. JP took bottle custody. Arrived on site 8/27/87 @ 11.00. Cool (57f), Sunny, Entire site on a slight slope to the north. Met Joe Smith, Marin Astare & Peter Shoperton for site recon.
<u>OVA Backround Levels</u> 0ppm. Stable

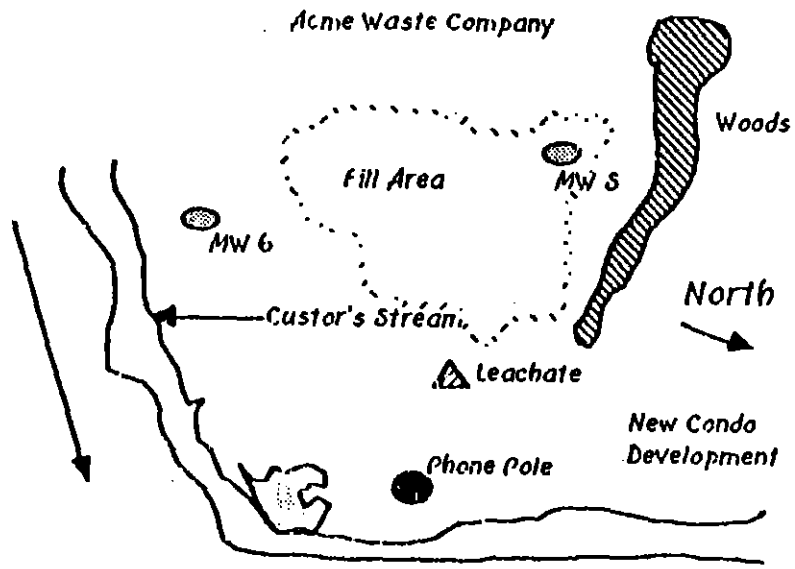
-OVA calibrated 8/25/87 see log entry page 2

12:10 Pauline sampling leachate TR 1601 photo 3, sample smells like sulfur and is rainbow in color with an oily sheen. Leachate was observed 3 feet west of the phone on site.

1250 Joe P. samples a seep at the base of Custor's Stream TR 1602 seep is bright red and has the viscosity similar to motor oil, Photo 4.

page 3

Paul Detchic



13:00 Paul is Purging Monitoring well #5 while Joe is preparing to collect a grab sample.
14:00 Joe is bailing the well #5 and @ 14:13 is filtering for metals (dissolved).
Sample acidified w/HNO_3 pH<2 after filteration was complete TR 1603 Photo 5

15:30 Sampling from well #5 completed in tripticate so an MS and MSD can be submitted for well #5. (1604-MS, 1605-MSD)

16:50 Team left site.

Paul Detchic

6.6.4 Photo-Documentation

All photographers will record time, date, site location, general direction faced, sequential number of photograph and roll number, and brief description of the subject in a field notebook.

6.6.5 Correspondence/Communications

All documents including field notes will be copied at the field office once a week and will be checked for completeness and filed. Filing cabinets will hold files in the field office. All correspondence received or sent from the field office will be dated and labeled with a project filing identification control number. All telephone conversations will be documented and filed.

6.7 Summary of Sampling and Analysis

This section provides a summary of the number of samples from each medium that will be collected, what parameters will be analyzed for these samples and the frequency and type of both field and laboratory QC that will be instituted.

6.7.1 Quality Control Samples

Duplicate or replicate samples, as well as field (prepared in the field) and trip (prepared in the laboratory) blanks will be collected and submitted to the analytical laboratories to provide an assessment of the quality of sampling activities and results. Field duplicate samples are samples that have been homogenized and divided into two or more portions at some step in the measurement process and submitted to the laboratory as unique independent samples. These samples will be analyzed to monitor analytical

precision. Since homogenization will not yield acceptable data for volatile organics, duplicates will not be performed for volatile organics. Instead, replicates will be collected for this fraction. Field replicates are separate grab samples collected from one sample location. Replicates are not homogenized. These samples will be analyzed to monitor sample representativeness. Field blank samples will be analyzed to check for procedural contamination which is a function of accuracy. Field blank sampling involves the use of deionized, laboratory supplied water which is either poured over or passed through sampling devices. Field blanks for air cartridges involve opening the cartridges on-site and allowing the materials to passively absorb any ambient contaminants. The location of the air field blank will be upwind of the site. Performance Evaluation (PE) samples will be supplied by the U.S. EPA to accompany samples for dioxin and furan analysis. Once dioxin and furan data has been received, ERM's QA Manager will request the 95% confidence interval results from the U.S. EPA for inclusion as part of ERM's quality assurance reviews. PE samples provide an excellent indication of analytical accuracy.

To confirm the level of cleanliness of the bottles, laboratory personnel will prepare the appropriate trip blanks by filling two VOA sample bottles per shipment container with organic free water. Trip blanks for air samples are sealed cartridges which are prepared by the laboratory, sent to the site, and returned with project samples for analysis. Frequencies for collecting field QA samples and the required types and numbers of laboratory QC samples are presented on Table 6-2. Although field blanks and trip blanks are not required for the collection of soil samples, they will be submitted as a qualitative indication of contamination.

QAPP Table 6-2
Sampling, Analysis: A Sample Summary

Matrix	Field Parameters	Number of Samples	Chemical Analysis A(3)	B	C	D	E	F	G	H	I	QA Samples: Replicates No.	Replicates Freq.	Replicates Total	Field Blanks(1) No.	Field Blanks Freq.	Field Blanks Total	Performance Evaluations(2) No.	Freq.	Total	Matrix Spike (4) Matrix spike duplicate No.	Freq.	Total	Total Matrix
Test Pit Soil(7)	Qualitative organic screening with OVA and/or HNu	105	87	18								11	1	11	6	1	6	1	1	1	6	2	12	135
Borings Soil(7)	Qualitative organic screening with OVA and/or HNu	93			42	22	17	12				10	1	10	8	1	8	0	0	0	5	2	10	121
Non-Native Borings Solids(7)	Qualitative organic screening with OVA and/or HNu	41	16	13				9			3	5	1	5	3	1	3	1	1	1	3	2	6	56
Sediments & Native Soils	Qualitative organic screening with OVA and/or HNu	89	14			22			18	35		10	1	10	6	1	6	0	0	0	5	2	10	115
Ground water (MW)(5)(6)(8)	pH, spec cond, temp, DO	66	62	4								7	1	7	4	1	4	1	1	1	4	2	8	86
Ground water (residential)(6)	pH, spec cond, temp, DO	6	6									1	1	1	1	1	1	0	0	0	1	2	2	10
Surface water(6)	pH, spec cond, temp, DO	26	12			14						3	1	3	3	1	3	0	0	0	3	2	6	38
Fish(7)	Weight, size	56	10			46						6	1	6	0	0	0	0	0	0	3	2	6	68
Air/particulates(7)	Qualitative organic screening with OVA and/or HNU	6	6									1	1	1	1	1	1	0	0	0	1	2	2	10

A = TCL volatile organics (+15), TCL nonvolatile (+25), mirex, kepone, photomirex, DPS*
B = TCL organics (+40), TCL inorganics, mirex, kepone, photomirex, dioxins/furans, 3,4-DCNB and DPS*
C = TCL volatile organics (+15), mirex, kepone, photomirex, methoxychlor, DPS*
D = TCL nonvolatile organics (+25), mirex, kepone, photomirex, DPS*
E = TCL nonvolatile organics (+25)
F = Physical parameters: soil classification, specific gravity, permeability, effective porosity, particle size, moisture content, and Atterburg limits
G = Mirex, Photomirex, Kepone, DPS*
H = Mirex, Photomirex, Kepone, DPS* & Methoxychlor
I = TCL organics (+40), TCL inorganics, mirex, kepone, photomirex, 3,4-DCNB and DPS*
* 3,4-Dichloronitrobenzene (DCNB) and/or diphenyl sulfone (DPS) will be analyzed as part of the semivolatile fraction

Notes:
1. Based on Table 3-6; The number of blanks may be more depending on the number of samples collected per day. Although field blanks and trip blanks are not required for soil samples, they will be submitted as a qualitative indication of contamination.
2. Performance evaluation and samples will be supplied by USEPA for Dioxin/Furan analysis (one per batch)
3. The volatile analysis of air will be TCL (+15) minus the four ketones on the VOA TCL
4. Matrix spikes will also be performed for mirex, kepone, photomirex, diphenyl sulfone, and DCNB
5. This number includes only Round 1 groundwater samples
6. Trip blanks will also be submitted one per cooler of aqueous samples for the analysis of TCL VOAs +15
7. Trip blanks will be submitted one per 20 samples.
8. Dedicated sampling equipment will be used for each monitoring well.

7.0 SAMPLE CUSTODY

7.1 Field Activities Sample Custody

The primary objective of sample custody procedures is to create an accurate written record which can be used to trace the possession and handling of all samples from the moment of their collection, through analysis, until their final disposition. Sample custody for samples collected during this investigation will be maintained by the Field Operations Manager (FOM) or the field personnel collecting the samples. The FOM or field personnel are responsible for documenting each sample transfer and maintaining custody of all samples until they are shipped to the laboratory.

All necessary sample bottles will be shipped to ERM or the field office by Enseco Laboratories and received by the FOM or field personnel. All necessary chemical preservatives will be added to the bottle by Enseco. Sample bottles needed for a specific sampling task will then be relinquished by the FOM to the sampling team after the FOM has verified the integrity of the bottles and assured that the proper bottles have been assigned for the task to be conducted.

Immediately after sample collection and bottle labeling, each sample container will be sealed in an individual plastic bag and stored in an on-site refrigerator at 4°C until packaging for shipment to the laboratory occurs. Samples will be removed from the on-site refrigerator and will then be placed immediately into an insulated cooler with "Blue Ice" and packing materials (e.g. vermiculite) for shipment to the laboratory. ERM field Chain-of-Custody records (Figure 7-1) completed at the time of sample collection will accompany the samples inside the cooler for shipment to the laboratory. The samples will be properly relinquished on the

FIGURE QAPP 7-1

ERM-Midwest, inc.
2000 West Henderson Road/Columbus, OH 43220

CHAIN OF CUSTODY RECORD

Project No./I.D. _____ Sheet _____ of _____
Sampled By _____ Bottles Supplied By _____
NOTE: When analyses are complete return this form to:
Name: _____ @ ERM-Midwest, Inc., Columbus, Ohio

Sample I.D.	Sample Description	Collection Date/Time	No. of bot-tles	Analysis Requested	Remarks

Shipping package opened by: _____ Date: _____ Time: _____
Employer: _____ Containers intact (Y/N) _____

Samples packaged & sealed by: _____ Date: _____ Time: _____
Employer: _____ Method of Shipment: _____

Received in laboratory by: _____ Date: _____ Time: _____
Employer: _____ Seals intact (Y/N) _____
Containers intact (Y/N) _____ If not, describe in Comments section

NOTE: Laboratory's Chain of Custody shall be in effect from receipt through analysis.

Relinquished By		Received By		Samples Intact	Date	Time	Initials	
Name	Employer	Name	Employer				Sender	Rec'r

COMMENTS:

Copies: White - Project file; Yellow - Client; Pink - Sampler

field Chain-of-Custody record by the sampling team. These record forms will be sealed in a ziplock plastic bag to protect them against moisture. The coolers are then sealed utilizing custody seals (Figure 7-2).

Prior to releasing the coolers, the FOM will require the courier to sign an ERM Cooler Transfer Acknowledgment (Figure 7-3). All sample coolers will be delivered to the analytical laboratory by either direct courier or 24-hour delivery courier (i.e., Federal Express).

7.2 Enseco Sample Custody

The laboratory chain-of-custody begins when containers are sent from the laboratory. While on site, sample containers are always under the personal custody of a member of the sampling team or are secured in sealed coolers. Upon completion of sampling, samples are appropriately labeled. The Enseco sample label is included as Figure 7-4. The samples are then stored in sealed containers and shipped to Enseco with a completed chain-of-custody record (Figure 7-5).

Samples are received by the Enseco sample manager, who records and files all shipping documentation. A copy of the Enseco's shipping log is presented as Figure 7-6. Shipments are then directed to the Sample Custodian. Internal chain-of-custody begins as the samples are received by this custodian. The coolers are inspected for proper seals and labels. Contents are removed and coordinated with packing lists and chain-of-custody records. The samples are logged into the computerized sample tracking system by completing a sample receiving form (Figure 7-7). This form is used to assign Enseco identification numbers to the samples and document their condition. This number consists of the booking number followed by a chronological number for each

FIGURE QAPP 7-2

ERM Custody Seal

ERM	OFFICIAL CUSTODY SEAL	Name:_____ Date :_____

FIGURE QAPP 7-3
ERM COOLER TRANSFER
ACKNOWLEDGEMENT

ERM COOLER TRANSFER ACKNOWLEDGEMENT

DATE	TIME	CLIENT / PROJECT	CLIENT NUMBER	NUMBER OF COOLERS	ERM RELEASE (SIGNATURE)	RECEIVED BY (SIGNATURE)	DATE	TIME

FIGURE QAPP 7-4

ERCO SAMPLE LABEL

ERCO *A DIVISION OF* **ENSECO** *INCORPORATED*
205 Alewife Brook Parkway, Cambridge, Massachusetts 02138 (617) 661-3111

Sampler _____ Date _____ Time _____

Sample Identification: _____

_____

_____

Preservatives: _____

_____

_____

Analysis: _____

_____

FIGURE QAPP 7-5

ENSECO CHAIN OF CUSTODY RECORD

Enseco

CHAIN-OF-CUSTODY RECORD

SAMPLER: *(Signature)* _____ Date Shipped _____ Carrier _____

Phone _____ Airbill No. _____ Cooler No. _____

SHIP TO:
Enseco-ERCO Lab
205 Alewife Brook Parkway
Cambridge, MA 02138
(617) 661-3111
ATTENTION: _____

SEND RESULTS TO:
Client Name _____
Company _____
Address _____
Phone _____

PROJECT NAME _____ PROJECT NO. _____ P.O. NO. _____

Relinquished by: *(Signature)*	Received by: *(Signature)*	Date	Time
Relinquished by: *(Signature)*	Received by: *(Signature)*	Date	Time
Relinquished by: *(Signature)*	Received at lab by: *(Signature)*	Date	Time
Relinquished from lab by: *(Signature)*	Received by: *(Signature)*	Date	Time

ANALYSIS REQUEST

Sample ID Number	Sample Description	Date/Time Sampled	Analysis Requested	Sample Condition Upon Receipt

Special Instructions/Comments:

NOTE: UNUSED PORTIONS OF NON-AQUEOUS SAMPLES WILL BE RETURNED TO CLIENT

Expected Analytical T.A.T's: _____ Immediate Attention (200% surcharge) _____ RUSH (50-100% surcharge) _____ Standard

ERCO Booking Log Number: *(for lab use only)*

Client Retains White Copy Only (Revised 1/87)

FIGURE QAPP 7-6

Erco Shipping Log - Incoming

Page ___

Date	Time	Carrier	Airbill Number	Name of Company	Shipment for	Received by (signature)	Item	COD

FIGURE QAPP 7-7

SAMPLE RECEIVING FORM

Booking Log No.: _____

Enseco - Erco Laboratory

Client: _____

Client Project: _____ Date Received: _____

Program Mgr.: _____ Date Due: _____

Reporting Format (Please Circle)

CLP Tier I A, B Tier II A, B ECRA RCRA

HSL PP Unknowns ☐ Yes ☐ No

Other _____

Date Due To Document Control: _____

Comments: _____

Received by		Date
_____	Metals	_____
_____	GWQ	_____
_____	VOA/GC	_____
_____	VOA/MS	_____
_____	Prep.	_____
_____	SEA	_____

Surcharge (Please Circle)

50% 100% 200%

Logged by: _____ Date: _____

Approved by: _____ Date: _____

Project Information

Client Contact: _____

Account No.: _____

Bill No.: _____

Project No.: _____

P.O. No.: _____

Program: _____

Page 1 of ___

FIGURE QAPP 7-7 (cont'd)

SAMPLE RECEIVING FORM

Booking Log No.: _____

Enseco - Erco Laboratory

Erco ID	Client ID	Date Sampled	Matrix	Condition	Analyses

Page ___ of ___

sample. Once it is approved by the project manager, the sample receiving form is copied and distributed to the appropriate laboratory managers.

In the event that samples are not properly received, the applicable laboratory's sample custodian will note this on the chain-of-custody, and inform ERCO's project manager. The samples will be rejected, and ERCO's project manager will notify the project manager at ERM verbally and in writing, and necessary corrective action will be taken.

When all the log-in procedures have been completed, the Sample Custodian contacts the appropriate laboratory managers and delivers the samples to a refrigerator where they will be stored under limited access. The designated sample custodians are presented in Section 4.2.7 of this QAPP. Maximum holding times, bottle types and preservatives are specified in Table 6-1.

Each Enseco laboratory maintains an internal laboratory logbook consisting of chronologically ordered sample tracking report sheets. The samples delivered by the sample custodian (or by an analyst in the case of an interlaboratory transfer) will be signed into the internal logbooks prior to storage and or analysis. Examples of sample receipt, tracking, and instrument log sheets are included as Figures 7-8 through 7-18.

Finally, all samples and extracts will be retained after analysis is complete. These samples will only be disposed of upon orders of the Project Manager and only after all tags and identification have been removed for the permanent file.

FIGURE QAPP 7-8

Enseco - Erco Laboratory B/N/A Sample Transfer Log

Erco ID	Client	Date Holding Time Expires	Sample Type	BNA Comb.	BNA Not Comb.	Extraction Used	Extraction Data	Surrogate Matrix Spike	Analyst Extr. Date	Associated Blank	Comments	Date Transferred	Re-extraction
						Sep. F. ___ Liq/Liq ___ Soxhlet ___ Sonication ___	Vol. Extr. ___ Wt. Extr. ___ % Dryness ___ Conc. by ___	BNA Surr. ___ Acid MS ___ B/N MS ___					
						Sep. F. ___ Liq/Liq ___ Soxhlet ___ Sonication ___	Vol. Extr. ___ Wt. Extr. ___ % Dryness ___ Conc. by ___	BNA Surr. ___ Acid MS ___ B/N MS ___					
						Sep. F. ___ Liq/Liq ___ Soxhlet ___ Sonication ___	Vol. Extr. ___ Wt. Extr. ___ % Dryness ___ Conc. by ___	BNA Surr. ___ Acid MS ___ B/N MS ___					
						Sep. F. ___ Liq/Liq ___ Soxhlet ___ Sonication ___	Vol. Extr. ___ Wt. Extr. ___ % Dryness ___ Conc. by ___	BNA Surr. ___ Acid MS ___ B/N MS ___					
						Sep. F. ___ Liq/Liq ___ Soxhlet ___ Sonication ___	Vol. Extr. ___ Wt. Extr. ___ % Dryness ___ Conc. by ___	BNA Surr. ___ Acid MS ___ B/N MS ___					
						Sep. F. ___ Liq/Liq ___ Soxhlet ___ Sonication ___	Vol. Extr. ___ Wt. Extr. ___ % Dryness ___ Conc. by ___	BNA Surr. ___ Acid MS ___ B/N MS ___					
						Sep. F. ___ Liq/Liq ___ Soxhlet ___ Sonication ___	Vol. Extr. ___ Wt. Extr. ___ % Dryness ___ Conc. by ___	BNA Surr. ___ Acid MS ___ B/N MS ___					
						Sep. F. ___ Liq/Liq ___ Soxhlet ___ Sonication ___	Vol. Extr. ___ Wt. Extr. ___ % Dryness ___ Conc. by ___	BNA Surr. ___ Acid MS ___ B/N MS ___					

FIGURE QAPP 7-9

Enseco - Erco Laboratory PESTICIDE SAMPLE TRACKING LOG

Erco ID	Client	Date Holding Time Expires	Sample Type	Analysis	Extraction Used	Extraction Data	Surrogate Matrix Spike	Analyst Extr. Date	Associated Blank	Comments	Date Transferred	Re-extraction
					Sep. F. ___ Liq/Liq ___ Soxhlet ___ Sonication ___	Vol. Extr. ___ Wt. Extr. ___ % Dryness ___ Conc. by ___	DBC ___ Matrix Spike ___					
					Sep. F. ___ Liq/Liq ___ Soxhlet ___ Sonication ___	Vol. Extr. ___ Wt. Extr. ___ % Dryness ___ Conc. by ___	DBC ___ Matrix Spike ___					
					Sep. F. ___ Liq/Liq ___ Soxhlet ___ Sonication ___	Vol. Extr. ___ Wt. Extr. ___ % Dryness ___ Conc. by ___	DBC ___ Matrix Spike ___					
					Sep. F. ___ Liq/Liq ___ Soxhlet ___ Sonication ___	Vol. Extr. ___ Wt. Extr. ___ % Dryness ___ Conc. by ___	DBC ___ Matrix Spike ___					
					Sep. F. ___ Liq/Liq ___ Soxhlet ___ Sonication ___	Vol. Extr. ___ Wt. Extr. ___ % Dryness ___ Conc. by ___	DBC ___ Matrix Spike ___					
					Sep. F. ___ Liq/Liq ___ Soxhlet ___ Sonication ___	Vol. Extr. ___ Wt. Extr. ___ % Dryness ___ Conc. by ___	DBC ___ Matrix Spike ___					
					Sep. F. ___ Liq/Liq ___ Soxhlet ___ Sonication ___	Vol. Extr. ___ Wt. Extr. ___ % Dryness ___ Conc. by ___	DBC ___ Matrix Spike ___					
					Sep. F. ___ Liq/Liq ___ Soxhlet ___	Vol. Extr. ___ Wt. Extr. ___ % Dryness ___	DBC ___ Matrix Spike ___					

FIGURE QAPP 7-10

PESTICIDE TRANSFER & ANALYSIS LOG

Enseco - Erco Laboratory

Pesticide Laboratory

Gas Chromatograph Log

Instrument Number: _____ **Detector Type:** _____

Date	Auto-sampler Position	Volume Inj.	Sample ID	Type of Run	GC Standard Number	Packing	Temp.	Computer File Name	Analyst

FIGURE QAPP 7-11

SEMIVOLATILE GC/MS SAMPLE TRANSFER LOG

SAMPLE TRANSFER LOG

I.D #	DATE	TIME	ACID	BN	BNA	delivered by	received

FIGURE QAPP 7-12

SEMIVOLATILE GC/MS INSTRUMENT LOG

ERCO A Division of ENSECO

ANALYSIS LOG

CLIENT		DISK	SAMPLE ID	CONC. or VOL	VOLT. or GAIN	COLUMN	Analyst	DATE	Searched	SDump	Unk.s	COMMENTS

FIGURE QAPP 7-13

ERCO/A Division of ENSECO Incorporated VOA SAMPLE RECEIVING AND TRACKING LOG

Date rec'd.	Client (sample lot size) ERCO IDs	Client ID	Run deadline	Matrix	Analysis	Screen info.	Rerun amount	Good run	# vials rec'd.	Comments	Project due date	Out	Sample Custodian

FIGURE QAPP 7-14

VOA INSTRUMENT LOG

Erco Laboratory | Volatile Organics Laboratory | GC/MS Analysis Log

FRN	Arcv. Tape	Ch. No.	Sample ID	Conc. or Vol.	E.M. Volt	Column		Analyst	Date	Check if Good Run	Comments
						Length	Type				
>											
>											
>											
>											
>											
>											
>											
>											
>											
>											
>											
>											
>											

FIGURE QAPP 7-15

METALS AQUEOUS SAMPLE RECEIPT LOG

Sample ID	pH	Acidified by	Qual. Filtered	0.45 µ Filtered	Date Received by Lab	Comments

FIGURE QAPP 7-16

METALS SOLID SAMPLE RECEIPT LOG

Sample ID	Date Received by Lab	Transferred from	Comments

FIGURE QAPP 7-17

ERCO/A DIVISION OF ENSECO INC.
INORGANIC LAB

CLIENT:_____
ERCO ID(s):_____
MATRIX:_____
Samples Relinquished by:_____
Samples Received by:_____
Date:_____ Time:_____

	Preparation (if necessary)			Analysis		
Analyte	(✓)	Chemist	date/ time	(✓)	Analyst	date/ time

FIGURE QAPP 7-18

INORGANIC SAMPLE RECEIPT FORM

Analysis

General Location

MATRIX – A = Aqueous, S = Solid, SL = Sludge

Sample ID Client	Matrix	Due Date Comments	Sample ID Client	Matrix	Due Date Comments	Sample ID Client	Matrix	Due Date Comments

7.3 Final Evidence Files

7.3.1 ERM's Final Evidence File

ERM will maintain all original documentation including but not limited to: field chain-of-custody forms, laboratory analytical reports, field notes, sample logs, telecons, photographs, memoranda, logbooks and letters in a designated secure, limited access area at the ERM-Midwest office. All items being placed into this file will be assigned a document control identification number and a document description. All items will then be logged onto the file "Table of Contents." All items added or removed from the file will be signed and dated by the person making the entry. All records will be maintained in this fashion for a period of ten years in accordance with Paragraph XXIII of the Consent Order.

7.3.2 Enseco's Final Evidence File

The final project report, which includes the raw data, is retained in Enseco's laboratory archives. These are permanent, locked archives which are not purged. All associated original logbooks are also retained in these archives.

8.0 CALIBRATION PROCEDURES AND FREQUENCY

8.1 Field Instruments

To ensure that measurements during the investigation have been collected with properly calibrated instruments, field personnel will follow the procedures described in the Equipment Owners Manual. All field equipment will be calibrated, at a minimum, twice daily prior to and after use (with the exception of the geophysical instrumentation), maintained, and repaired in accordance with manufacturer's specifications. In addition, prior to use, each major piece of equipment will be cleaned, decontaminated, checked for damages, and repaired as needed. These activities will be noted in a field log notebook.

Despite even the most rigorous maintenance program, equipment failures do occur. When equipment cannot be repaired, it will be returned to the manufacturer for repairs.

Quality control efforts and accuracy and precision objectives for field water quality measurements are summarized below. Calibration procedures and frequency for all field instruments are summarized in Table 8-1. Section 8.1.1 provides detailed calibration procedures for all air and particulate equipment.

8.1.1 Century OVA Meter 128

The OVA 128 is capable of responding to nearly all site specific organic compounds.

QAPP TABLE 8-1

EQUIPMENT MAINTENANCE AND CALIBRATION PROTOCOLS
RUETGERS-NEASE SALEM SITE RI/FS

Equipment	Maintenance/Calibration	Frequency
EM34 and 31 conductivity meters	Internal instrumentation is factory calibrated/ routinely maintained.	Every 5 yrs.
	A background conductivity survey will be performed to calibrate the equipment.	Prior to initiation of the geo-physical survey.
Resistivity meter	Internal instrumentation is factory calibrated/ routinely maintained.	Every 5 yrs.
	A background conductivity survey will be performed to calibrate the equipment.	Prior to initiation of the geo-physical survey.
Seismic	Internal instrumentation is factory calibrated/ routinely maintained.	As Required.
PID photoionization detector	Calibrate with isobutylene gas.	Start and end of each. day.
FID flame ionization detector	Calibrate with methane and/or benzene gas.	Start and end of each day.
Explosimeter	Calibrate with methane and carbon monoxide.	Once per month.
	Zero instrument in air.	Start of each day in clean area (e.g., support zone)
pH meters	Calibrate with three pH buffer solutions.	Before and after use, and after every 20 samples

QAPP TABLE 8-1 (cont'd)

EQUIPMENT MAINTENANCE AND CALIBRATION PROTOCOLS
RUETGERS-NEASE SALEM SITE RI/FS

Equipment	Maintenance/Calibration	Frequency
Temperature	Check against a mercury thermometer.	Every 10 samples.
Sp. conductance	Calibrate with one calibration solution.	Before and each use.
Dissolved oxygen meter	Calibration according to manufacturer's recommendations with ambient air.	At the beginning of each day, and every 30 minutes.
Rechargable equipment batteries	Charge	After use as, required.
Sampling accessories (tubing, submersible pumps)	Periodic maintenance performed and recorded in equipment maintenance log.	As required.

The OVA will be calibrated using a mixture of a specific vapor in air, with known concentration. After the instrument is in operation and zeroed by using ambient air that has passed through vapor phase granular activated charcoal, a sample of calibration gas will be drawn into the instrument and calibration will proceed by adjusting the readout to the known concentration. Calibration will be accomplished using a single known sample of methane in air in the range of 90 to 100 ppm. After this calibration, the setting on the "digidial" will be recorded. Calibration on any one range automatically calibrates the other two ranges.

8.1.2 HNU Model P1-101 Photoionization Analyzer

The HNU calibration gas consists of approximately 100 ppm isobutylene in an air matrix. This gas has been referenced to benzene. The calibration gas canister is connected to the HNU inlet probe and the canister is opened. The function switch is then turned to the 0-200 range position (X10). The meter is adjusted to the ppm number on the calibration gas canister. The valve on the calibration gas regulator is disconnected from the probe inlet.

8.1.3 High Volume Particulate Sampling

The calibration of high volume particulate samplers is fairly complicated but is briefly summarized below:

1. A hi-vol sampler will be assembled with a clean filter and operated for at least five minutes at normal line voltage.

2. The rotameter number and date on three gummed labels will be recorded. One label will be affixed to the top of the metal on the front of

the rotameter, another to the middle of the vacuum hose and the third to the side of the hi-vol motor.

3. The rotameter is now attached to the hi-vol motor.

4. The rotameter is adjusted so that the center of the ball reads 60 on the rotameter scale.

   a. The locking nut is now loosened by turning it from left to right.

   b. The screw is now adjusted by turning it clockwise to lower the ball and counterclockwise to raise the ball. After this adjustment, the locknut is tightened and the hi-vol motor is turned off.

5. The filter holder is now removed.

6. The calibrated orifice is now attached with one of the load plates between the motor and the orifice.

7. The motor is turned on and the water manometer and rotameter readings are recorded after they stabilize.

8. The motor is turned off.

9. Steps 6-8 are repeated for each load plate.

10. Steps 6-9 are repeated once more.

11. The air flow rate from the hi-vol orifice calibration curve for each manometer reading will be recorded.

12. The barometric pressure in mm of Hg, and the temperature in ^{o}C will be recorded.

13. The temperatures measured when the hi-vol orifice was calibrated (T_2) are converted to absolute values (K).

14. The true flow rates corrected to the barometric pressure and temperature are determined by substituting each flow rate for Q_1 in the following equation and by solving for Q_2.

$$Q_2 = Q_1 \left(\frac{T_2 P_1}{T_1 P_2}\right)^{1/2}$$

where

Q_2 = corrected flow rate, m^3/min,
Q_1 = flow rate, m^3/min,
T_2 = absolute temperature (during sampling), K
P_1 = barometric pressure when hi-vol orifice was calibrated, mm Hg,
T_1 = absolute temperature when hi-vol orifice was calibrated, K^o
P_2 = barometric pressure (during sampling), mm Hg.

The actual flow rate during sampling will be determined by comparing the rotameter with the calibration curve of the rotameter reading vs flow rate. A flow rate of approximately 60 ACFM will be maintained by adjusting the flow controller on the actual hi-volume particulate sampler.

8.1.4 PUF Sampler

The airflow through the sampling system is monitored by a venturi/magnehelic assembly. A multipoint calibration of the venturi/magnehelic assembly must be conducted every six months using an audit calibration orifice, as described in the U.S. EPA High Volume Sampling Method (Section 8.1.3). A single point calibration must be performed before and after each sample collection, using the procedure described below.

Prior to calibration a "dummy" PUF cartridge and filter are placed in the sampling head and the sampling motor is activated. The flow control valve is fully opened and the voltage aviator is adjusted so that a sample flow rate corresponding to approximately 110% of the desired flow rate is indicated on the magnehelic. The motor is allowed to warmup for approximately 10 minutes and then the flow control valve is adjusted to achieve the desired flow rate. The ambient temperature and barometric pressure will be recorded. The calibration orifice is then placed on the sampling head and a manometer attached to the tap on the calibration orifice. The sampler is momentarily turned off to set the zero level of the manometer. The sampler is then switched on and the manometer reading recorded, once a stable reading is achieved. Calibration is now complete. The calibration curve for the orifice is then used to calculate sample flow using the just obtained manometer reading.

The calibration curve for the venturi/magnehelic assembly is used to calculate sample flow from the manometer measurement. The calibration data will be recorded. If the manometer and venturi/magnehelic values do not agree within 10%, the sampler will be inspected for damage, flow blockage, etc. If no obvious problems are found the sampler will be recalibrated (multi-point) according to the U.S. EPA High

Volume Sampling procedure. If the values agree within 10%, calibration will proceed as follows:

1. Calibration of the PUF Sampler is performed <u>without</u> a foam slug or filter paper in the sampling module. However, the empty glass cartridge will remain in the module to insure a good seal through the module.

2. The GMW-40 Calibrator is installed on top of the 4" filter hole.

3. An 8" water manometer is connected to the Calibrator.

4. The ball valve is fully opened.

5. The system is turned on by tripping the manual switch on the timer.

6. The voltage control screw is adjusted to obtain a reading of 70 inches on the dial gage (magnehelic gage).

7. With 70 inches on the dial gage as the first calibration point, the manometer reading will be recorded.

8. The ball valve is slightly closed to readjust the dial gage down to 60 inches. The manometer reading will be recorded.

9. Using the above procedure, the ball valve will be adjusted for dial gage readings at 50, 40, and 30 inches and recorded.

10. Using these two sets of readings, a curve will be plotted. This curve will be used for determining the actual flow rate in the field.

11. The voltage control is then readjusted fully clockwise to its maximum setting.

8.1.5 Pump Flow Rate Calibration for the Tenax, XAD-2 and CMS Traps

A mass flow meter will be utilized to measure and control the flow rate through each sampling train. A wet test meter will be utilized to verify the flow through each mass flow meter. A pump, mass flow meter, and mass controller will be connected in series. The system will operate for a fixed period of time. Each revolution of the dial on the wet test meter represents the displacement of a known volume of water by air. By knowing the volume of air passing through the system per unit of time, the mass flow meter will be calibrated. An initial calibration will be performed upon each mass flow meter. The mass flow meters will be recalibrated weekly or whenever abnormalities become apparent.

8.2 Enseco Analytical Instrumentation

Enseco will employ analytical methods found in the U.S. EPA Contract Laboratory Program Statement of Work for Organic Analysis (Rev. 8/87) and Inorganic Analysis (Rev. 8/87) EPA CLP (SOW). These methods cite the appropriate calibration and check procedures that are required to conduct the analyses properly. An outline of these procedures follows:

8.2.1 GC/MS -- Volatiles (VOA)

The instrument is hardware tuned using 50 ng Bromofluoro benzene (BFB). Ion Abundance criteria must meet that of EPA CLP. Initial calibration is required at 20, 50, 100, 150, and 200 ug/L. Average Response Factors (RF) and Relative Standard Deviations (RSD) are calculated for each compound (see Table 9-2 in Section 9.1.1 for Target Compound List [TCL]). Calibration check compounds (CCC) are: 1,1-dichloroethene, chloroform, 1,2-dichloropropane, toluene, ethylbenzene, and vinyl chloride. System Performance Check Compounds (SPCC) are chloromethane, 1,1-dichloroethane, bromoform, 1,1,2,2- tetrachloroethane, and chlorobenzene. For the initial calibration to be considered valid, the relative percent difference (RSD) must be less than or equal to 30.0 percent for CCC's. The RF for SPCC's must be 0.300 or greater; 0.250 or greater for bromoform. Analysis of samples can proceed for twelve hours following the time of the BFB injection once these criteria are met. Continuing calibration is achieved through 50 ng BFB injection and criteria, and a calibration standard containing all of the TCL compounds. For a continuing calibration to be valid, SPCCs must meet the same criteria as that for the curve, and CCCs RF's less than or equal to 25.0% difference from those of the curve. Sample analysis can proceed for twelve hours from the time of the BFB injection once these criteria are met.

8.2.2 GC/MS -- Semivolatiles and Diphenyl Sulfone and Dichloronitrobenzene

Semivolatiles calibration procedures follow the same analytical calibration scheme as that of volatiles with the following differences: DFTPP is used to meet ion abundance criteria. The initial calibration is required at 20, 50, 80, 120, and 160 ng/ul. Nine compounds contained on the TCL list

are not required in the 20 ng/ul standard. There are 13 CCC's and 4 SPCC's which are listed in the method. The minimum acceptable RF for SPCC's is 0.050. The maximum % RSD for the CCC's in the initial calibration is 30.0%. The percent difference required for continuing calibrations is 25.0%. Procedures for preparing calibration standards, their concentrations, and internal standards which will be used to quantitate DPS and 3,4 DCNB have been provided in the final method validation study.

8.2.3 GC/MS -- Mirex, Kepone and Photomirex

The specific procedures for preparing calibration standards and their concentrations, the levels for matrix spikes and surrogates, and recovery standards have been presented in the final method validation study. Calibration procedures are also provided in this document.

8.2.4 GC -- Pesticides/PCBs

Calibration is initially established for each GC and column used. Three concentrations of evaluation standards are run containing aldrin, endrin, dibutylchlorendate and 4-4'-DDT. Calibration factors (CF) and %RSDs are calculated for each compound. %RSD must be less than 10.0% for quantitation. The total breakdown of Endrin and 4-4'-DDT is calculated and must be less than 20.0%. An initial calibration is then run containing each of the single and multi-component pesticides. This establishes initial calibration. Continuing calibration standards are run every five samples and at the end of the 72 hour period. Percent differences must not exceed 15.0% for quantitation or 20.0% for confirmation. A confirmation analysis is run to confirm any compound tentatively identified in the primary analysis. Quantitation can be done using the secondary analysis.

Criteria for a second column confirmation and the 72 hour sequence are found in the EPA CLP method.

8.2.5 Metals

8.2.5.1 Inductively Coupled Plasma (ICP)

A five point multi-element calibration curve is run to establish initial calibration. An initial calibration verification (ICV) is then run. All elements in the ICV must agree with the true values to ±10%. An interference check standard (ICS) is then run and elements must agree with the true values to ± 20%. Sample analysis can then proceed. A continuing calibration verification (CCV) is run after every 10 samples and all elements must be within ±10% of the true value. Sample analysis can continue for up to 8 hours or until the CCV is out, at which point the instrument must be recalibrated.

8.2.5.2 Graphite Furnace AA

The same analytical scheme as that for ICP is used. A five point calibration is run and verified with an ICV which must fall within ±10% of the true value. A CCV is run every 10 samples and must agree to ±10%. Additionally, samples are double-burned and the RPD must be 20% or less.

8.2.5.3 Cold Vapor AA-Mercury

A four point initial calibration is used. The resulting correlation coefficient derived from the calibration curve must be greater than or equal to 0.995. An ICV is then run and must be within ±15% of the true value. A CCV is run every 10 samples and must agree with the true value ±15%.

FIGURE QAPP 8-1

STANDARDS LOG SHEET

Parent	Compound	Date	Chemist	Parent (stock) Concentration (ng/µl)	Dilution parent. stock	Vol. Parent Solution	Vol. Solvent Added	Final Vol.	Solvent	Conc. ng/µl

8.2.5.4 Cyanide - Distillation

A five point initial calibration is used. The resulting correlation coefficient derived from this calibration curve must be greater than or equal to 0.995. An ICV is then run and must be within ±15% of the true value. A CCV is run every 10 samples and must agree with the true value within ±15%.

8.2.6 Standards

Enseco's primary sources of standard reference materials for calibrations and calibration checks are the U.S. EPA and National Bureau of Standards repositories. Secondary sources are reliable commercial supply houses, such as Supelco, Aldrich, and Chem Service. Specific projects, such as pesticide certification programs, may require the use of client-specified reference standards. New standards are routinely checked against known standards, traceable to EPA or NBS reference materials, if available. Each of Enseco's laboratories maintains a Standards Preparation Logbook in which all pertinent information regarding the source and preparation of each analytical standard is recorded. Examples are shown in Figures 8-1 and 8-2.

Reagents used in the preparation of matrix spike and matrix spike duplicate solutions and in surrogate standard spiking solutions are obtained directly from the U.S. EPA or are validated using EPA-traceable standards. Quality Control Check Samples are requested and received on a frequent basis from EPA-EMSL in Cincinnati.

Reagents that are used in large quantities are analyzed for purity at Enseco's laboratories prior to purchase, according to lot number.

FIGURE QAPP 8-2

STANDARDS LOG SHEET

Mix I.D.									
Date				Date					
parent vol.				Parent ID					
Sol. added				Vol. parent					
Total vol.				Vol. solvent added					
Solvent				Final vol.					
				Solvent					
				Dilution					
			Mix ID:						
Compound	Parent ID	Parent conc. ng/µl	vol. of parent	ng/µl	ng/µl	ng/µl	ng/µl	ng/µl	ng/µl

8.2.7 Instrumentation

Enseco instrumentation includes:

8.2.7.1 GC/MS/DS Systems -- Semivolatile Organics

- Finnigan MAT 4600/Super INCOS GC/LC/MS/DS
- Finnigan MAT 4500/Super INCOS GC/MS/DS
- Finnigan MAT 4521/INCOS GC/MS/DS
- Hewlett Packard 7970B magnetic tape unit
- Cipher magnetic tape unit

8.2.7.2 GC/MS/DS Systems -- Volatile Organics

- Three Hewlett Packard 5996 GC/MS systems with HP RTE-6 Data Systems and Tekmar LSC-2 purge and traps with Tekmar ALS automatic samplers
- Hewlett Packard 5985 GC/MS/DS with HP RTE-6 Data Systems and Tekmar LSC-2 purge and traps with Tekmar ALS automatic samplers
- Nutech #8533 thermal desorption unit

8.2.7.3 GC Systems -- Semivolatile Organics

- Three Hewlett Packard 5890A GCs with dual electrol capture detectors and HP 7673A autosamplers
- Hewlett Packard 5890A with single flame ionization detector and single nitrogen photometric detector and HP 7673A autosampler

- Hewlett Packard 5880A with single flame ionization detector and HP 7671A autosampler

- Hewlett Packard 5880A with dual electron capture detectors and flame-photometric detector and HP 7673A autosampler

- Hewlett Packard 5880A with dual electron capture detectors and HP 7671A autosampler

- Hewlett Packard 5840A with single flame ionization detector and HP 7672A autosampler

- Hewlett Packard 5840A with single flame ionization detector and HP 7671A autosampler

- Hewlett Packard 5840A with single electron capture detector and HP 7671A autosampler

- Hewlett Packard 5840A with single electron capture detector and single flame ionization detector and HP 7671A autosampler

- Hewlett Packard 5840A with single electron capture detector and HP 7671A autosampler

- Hewlett Packard 5840A with single electron capture detector and single flame ionization detector and HP 7671A autosampler

8.2.7.4 Other Chromatographic Instrumentation

- Waters HPLC with WISP automated sample processor and UV (variable wavelength), fluorescence, and refractive index detectors

- Farrand Mark I Spectrofluorometer with corrected excitation and emission

- ABC 1002 gel permeation chromatograph for preparative cleanup of sludges and fatty tissues

8.2.7.5 Spectrophotometric Instrumentation

- Perkin-Elmer 710B infrared spectrophotometer

8.2.7.6 Inorganic Instrumentation -- Metals

- Jarrell-Ash 9000 Inductively Coupled Argon Plasma Emission Spectrometer with 33 analytical channels, background correction capability, and Apple software and graphics

- Jarrell-Ash 970 Inductively Coupled Argon Plasma Emission Spectrometer with 40 fixed and 1 variable wavelength analytical channels, dual discs, and background correction capability

- Three Perkin-Elmer 3030 Zeeman Atomic Absorption Spectrophotometers with HGA-600 graphite furnaces, AS-60 automatic samplers, and HGA graphics software

- Perkin-Elmer 5100 Zeeman Atomic Absorption Spectrophotometer with deuterium background correction, HGA-600 graphite furnace, AS-60 automatic sampler, and HGA graphics software

- Perkin-Elmer 3030B Atomic Absorption spectrophotometer with deuterium background correction and graphics software

- Perkin-Elmer 603 Atomic Absorption Spectrophotometer with graphic, digital, and computer-compatible output;

deuterium background correction; electrode discharge lamps; and Hg, As, Sb, and Se accessories

- Two Millipore Super Q ultrapure water systems
- Two Fisher Mercury Analyzers

8.2.7.7 Inorganic Instrumentation -- Wet Chemistry

- Alpkem RFA-300 Rapid Flow Analyzer
- Two Technicon AutoAnalyzers - 1 channel
- Dohrmann DC80 TOC Analyzer
- Mitsubishi Model MCI 10 TOX Analyzer
- Labconco Rapid Digestor 25

8.2.7.8 Analytical Balances

- Metler PE600
- Metler H51
- Metler PN1210
- Cahn 25 Electrobalance
- Cahn TA450
- Ohaus B1500D
- Sartorious 2462
- Metler PM4600

9.0 ANALYTICAL PROCEDURES

In order to accomplish the objectives of the RI/FS, analysis for Volatile Organics, Semivolatile Organics, Dioxins and Furans, Inorganics, and various non-target compounds (mirex, photomirex, kepone, dichloronitrobenzene, and diphenyl sulfone) will be performed. All standard TCL analyses will be performed using the most recent revision of the U.S. EPA's Statement of Work for Organics, Inorganics and Dioxins/Furans (U.S. EPA PCDF/PCDD protocol, exhibit D, analytical methods, draft April 19, 1989 and revised in November, 1989 and EPA CLP SOW 8/87). Enseco currently holds contracts with the U.S. EPA for each of these categories. The following sections provide analytical references and method descriptions for each of the parameters in the different matrices.

The non-target compounds are also discussed in this section with detailed descriptions of the methods developed for this particular program. A method validation study will be performed on each of the non-standard components and will determine method detection limits, precision, and accuracy.

9.1 Sample Preparation

9.1.1 TCL Analyses: Volatiles, Semivolatiles, Pesticides, PCBs, Metals, Dioxins and Furans

Waters and soils will be prepared according to EPA CLP guidelines. These can be found in the following publications: Organics; U.S. EPA Contract Laboratory Program Statement of Work for Organic Analyses (Rev. 8/87); Inorganics: U.S. EPA Contract Laboratory Program Statement of Work for Inorganics Analysis, SOW No. 787, (Rev 12/87); Dioxins and Furans; U.S. EPA PCDF/PCDD protocol, exhibit D, analytical methods, draft April 19, 1989 and revised in November, 1989 and EPA CLP SOW 8/87.

For semivolatile, organics this method involves solvent extraction of the water sample using a separatory funnel and Kuderna-Danish (KD) concentration. Soil samples are sonicated and concentrated to 1 ml. using KD concentrations. For soils with high level non-TCL interferences, GPC cleanup will be employed, if needed.

Pesticides and PCBs in water are solvent extracted, concentrated, and subject to alumina column cleanup. Soil samples are sonicated and concentrated, and cleaned up on an alumina column.

For dioxins and furans in soil and sediments, samples are extracted with methanol/petroleum ether, solvent-exchanged into hexane, and subjected to multiple cleanup procedures. Water samples are extracted with methylene chloride, solvent-exchanged into hexane, and washed with aqueous potassium hydroxide, sodium chloride, and sulfuric acid to remove gross interferences. Dioxin and furan analysis will be performed at Enseco-California Analytical Laboratory using U.S. EPA PCDF/PCDD protocol, exhibit D, analytical methods, draft April 19, 1989 and revised in November, 1989 and CLP-SOW 8/87. California Analytical currently performs these analyses under SAS contracts with the U.S. EPA.

Volatile organics in water and low-level soils require no preparation step other than using appropriate dilution volumes and adding surrogate and internal standard compounds prior to analysis. Medium level soils are methanol extracted, injected into water, and purged.

Inorganics in water and soil are acid digested prior to analysis.

For all preparations, Enseco Laboratory will strictly adhere to the EPA CLP protocol. Target Compound Lists and Method Detection Limits for each analysis are listed in Tables 9-1 through 9-6.

Table 9-1

Target Compound List and Detection Limits for Semivolatile Organics

Parameter	Detection Limits[a] Water (µg/L)	Selected Water (µg/L)	Soil[b,c] (µg/kg)
Phenol	10	2	330
bis(2-Chloroethyl)ether	10	1.5	330
2-Chlorophenol	10	1.5	330
1,3-Dichlorobenzene	10	1.5	330
1,4-Dichlorobenzene	10	1.5	330
Benzyl alcohol	10	2	330
1,2-Dichlorobenzene	10	2.5	330
2-Methylphenol	10	1	330
bis(2-Chloroisopropyl)ether	10	2.5	330
4-Methylphenol	10	1	330
N-nitroso-di-n-dipropylamine	10	1.5	330
Hexachloroethane	10	2	330
Nitrobenzene	10	2.5	330
Isophorone	10	2.5	330
2-Nitrophenol	10	2	330
2,4-Dimethylphenol	10	2	330
Benzoic acid	50	25	1,600
bis(2-Chloroethoxy) methane	10	2.5	330
2,4-Dichlorophenol	10	2	330
1,2,4-Trichlorobenzene	10	2	330
Naphthalene	10	2	330
4-Chloroaniline	10	3.3	330
Hexachlorobutadiene	10	2.5	330
4-Chloro-3-methylphenol (para-chloro-meta-cresol)	10	1.5	330
2-Methylnaphthalene	10	2	330
Hexachlorocyclopentadiene	10	2	330
2,4,6-Trichlorophenol	10	4.7	330
2,4,5-Trichlorophenol	50	1.5	1,600
2-Chloronaphthalene	10	1.5	330
2-Nitroaniline	50	10	1,600
Dimethylphthalate	10	1.5	330
Acenaphthylene	10	1.5	330
2,6-Dinitrotoluene	10	1	330
3-Nitroaniline	50	12.2	1,600
Acenaphthene	10	1.5	330
2,4-Dinitrophenol	50	23	1,600
4-Nitrophenol	50	15	1,600
Dibenzofuran	10	1	330

See next page for footnote references.

Table 9-1 (Continued)

Target Compound List and Detection Limits for Semivolatile Organics

Parameter	Detection Limits[a] Water (µg/L)	Selected Water (µg/L)	Soil[b] (µg/kg)
2,4-Dinitrotoluene	10	1	330
Diethylphthalate	10	1	330
4-Chlorophenyl-phenyl ether	10	1.6	330
Fluorene	10	1	330
4-Nitroaniline	50	20	1,600
4,6-Dinitro-2-methylphenol	50	15	1,600
N-nitrosodiphenylamine[d]	10	1.5	330
4-Bromophenyl-phenylether	10	1.5	330
Hexachlorobenzene	10	1.5	330
Pentachlorophenol	50	2	1,600
Phenanthrene	10	1	330
Anthracene	10	2.5	330
Di-n-butylphthalate	10	2	330
Fluoranthene	10	1.5	330
Pyrene	10	1.5	330
Butylbenzylphthalate	10	3.5	330
3,3'-Dichlorobenzidine	20		660
Benzo(a)anthracene	10	1.5	330
Chrysene	10	1.5	330
bis(2-Ethylhexyl)phthalate	10	1	330
Di-n-octylphthalate	10	1	330
Benzo(b)fluoranthene	10	1.5	330
Benzo(k)fluoranthene	10	1.5	330
Benzo(a)pyrene	10	2	330
Indeno(1,2,3-cd)pyrene	10	3.5	330
Dibenz(a,h)anthracene	10	2.5	330
Benzo(g,h,i)perylene	10	4	330
Diphenyl sulfone	10	4	1000
Dichloronitrobenzene	25	12	1700

[a]Specific quantitation limits are highly matrix dependent. The quantification limits are provided for guidance and may not always be achievable.

[b]Quantitation limits listed for soil are based on wet weight. The quantitation limits calculated by the laboratory for soil (calculated on a dry weight basis) will be somewhat higher.

[c]Detection limits for medium level soil samples are 60 times the low level detection limits.

[d]Reported as diphenylamine.

Table 9-2

Target Compound List and Detection Limits for Volatile Organics

Parameter	Detection Limits[a] Water (µg/L)	Selected Water (µg/L)	Soil[b,c] (µg/kg)
Chloromethane	10	1.5	10
Bromomethane	10	1.5	10
Vinyl chloride	10	1.5	10
Chloroethane	10	1.5	10
Methylene chloride	5	1	5
Acetone	10	5	10
Carbon disulfide	5	3	5
1,1-Dichloroethene	5	1.5	5
1,1-Dichloroethane	5	1.5	5
1,2-Dichloroethene (total)	5	1.5	5
Chloroform	5	1.5	5
1,2-Dichloroethane	5	1.5	5
2-Butanone	10	5	10
1,1,1-Trichloroethane	5	1.5	5
Carbon tetrachloride	5	1.5	5
Vinyl acetate	10	5	10
Bromodichloromethane	5	1.5	5
1,2-Dichloropropane	5	1.5	5
cis-1,3-Dichloropropene	5	1	5
Trichloroethene	5	1.5	5
Dibromochloromethane	5	1.5	5
1,1,2-Trichloroethane	5	1.5	5
Benzene	5	1.5	5
trans-1,3-Dichloropropene	5	2	5
Bromoform	5	1.5	5
4-Methyl-2-pentanone	10	3	10
2-Hexanone	10	5	10
Tetrachloroethene	5	1.5	5
Toluene	5	1.5	5
1,1,2,2-Tetrachloroethane	5	1.5	5
Chlorobenzene	5	1.5	5
Ethyl benzene	5	1.5	5
Styrene	5	1	5
Total xylenes	5	2	5

[a]Specific quantitation limits are highly matrix dependent. The quantification limits are provided for guidance and may not always be achievable.

[b]Quantitation limits listed for soil are based on wet weight. The quantitation limits calculated by the laboratory for soil (calculated on a dry weight basis) will be somewhat higher.

[c]Detection limits for medium level soil samples are 125 times the low level detection limits.

Table 9-3

Target Compound List and Detection Limits for Pesticides/PCBs

Parameter	Detection Limits[a] Water (µg/L)	Selected Water (µg/L)	Soil[b,c] (µg/kg)
alpha-BHC	0.05	0.010	8.0
beta-BHC	0.05	0.005	8.0
delta-BHC	0.05	0.005	8.0
gamma-BHC (Lindane)	0.05	0.005	8.0
Heptachlor	0.05	0.030	8.0
Aldrin	0.05	0.005	8.0
Heptachlor epoxide	0.05	0.005	8.0
Endosulfan I	0.05	0.010	8.0
Dieldrin	0.10	0.010	16.0
4,4'-DDE	0.10	0.005	16.0
Endrin	0.10	0.010	16.0
Endosulfan II	0.10	0.010	16.0
4,4'-DDD	0.10	0.020	16.0
Endosulfan sulfate	0.10	0.10	16.0
4,4'-DDT	0.10	0.020	16.0
Methoxychlor	0.5	0.005	80.0
Endrin ketone	0.10	0.030	16.0
alpha-Chlordane	0.5	0.020	80.0
gamma-Chlordane	0.5	0.020	80.0
Toxaphene	1.0	0.25	160.0
Aroclor-1016	0.5	0.10	80.0
Aroclor-1221	0.5	0.10	80.0
Aroclor-1232	0.5	0.10	80.0
Aroclor-1242	0.5	0.10	80.0
Aroclor-1248	0.5	0.10	80.0
Aroclor-1254	1.0	0.10	160.0
Aroclor-1260	1.0	0.10	160.0

[a]Specific quantitation limits are highly matrix dependent. The quantification limits are provided for guidance and may not always be achievable.

[b]Quantitation limits listed for soil are based on wet weight. The quantitation limits calculated by the laboratory for soil (calculated on a dry weight basis) will be somewhat higher.

[c]Detection limits for medium level soil samples are 15 times the low level detection limits.

Table 9-4

Target Compound List and Detection Limits for Inorganics

Parameter	Detection Limits[a] Water (µg/L)	Soil[b] (mg/kg)
Aluminum	200	40
Antimony	60	12
Arsenic	10	2
Barium	200	40
Beryllium	5	1
Cadmium	5	1
Calcium	5,000	1,000
Chromium	10	2
Cobalt	50	10
Copper	25	5
Cyanide	10	2
Iron	100	20
Lead	5	1
Magnesium	5,000	1,000
Manganese	15	3
Mercury	0.2	0.04
Nickel	40	8
Potassium	5,000	1,000
Selenium	5	1
Silver	10	2
Sodium	5,000	1,000
Thallium	10	2
Vanadium	50	10
Zinc	20	4

[a]Specific quantitation limits are highly matrix dependent. The quantification limits are provided for guidance and may not always be achievable.

[b]Quantitation limits listed for soil are based on wet weight. The quantitation limits calculated by the laboratory for soil (calculated on a dry weight basis) will be somewhat higher.

Table 9-5

Target Compound List and Detection Limits for Polychlorinated Dibenzofurans and Dibenzo-p-dioxins

Parameter	Detection Limits[a] Water (ng/L)	Soil[b] (µg/kg)
Tetra (total)	1	0.1
Penta	1	0.5
Hexa	1	0.5
Hepta	5	1.0
Octa	10	5.0

[a]Specific quantitation limits are highly matrix dependent. The quantification limits are provided for guidance and may not always be achievable.

[b]Quantitation limits listed for soil are based on wet weight. The quantitation limits calculated by the laboratory for soil (calculated on a dry weight basis) will be somewhat higher.

TABLE 9-6

Target Compound List and Detection Limits for Mirex, Photomirex, and Kepone

	Detection Limits[ab]			
Parameter	Water (µg/L)	Soil[c] (µg/kg)	Fish (µg/kg)	Air[d] (µg/m^3)
Mirex	0.003	9	13	0.0334
Photomirex	0.024	10	8	0.0522
Kepone	0.066	34	97	0.544

[a]Specific quantitation limits are highly matrix dependent. The quantitation limits are provided for guidance and may not always be achievable.

[b]Practical quantitation limits, which are two times the method detection limits, will be used for reporting purposes.

[c]Quantitation limits listed for soil are based on wet weight. The quantitation limits calculated by the laboratory for soil (calculated on a dry weight basis) will be somewhat higher.

[d]Detection limits for these compounds in air are based on the assumption of one cubic meter of air collected.

9.2 Instrument Start-Up Performance Check

All instruments will be calibrated according to the EPA CLP methods. This involves mass spectrometer performance and sensitivity checks, and multilevel calibrations, with tabulations of %RSD, average responsibility factors, and %D. For a detailed description of Instrument Calibration see Section 8.

Bromofluorobenzene (BFB) is used to tune the GC/MS for volatile organics and decafluorotriphenylphosphine (DFTPP) for semivolatile organics. Tunes are established every 12 hours and standard and/or sample analysis cannot proceed unless EPA CLP criteria is met. Tabulations of tunes and the associated raw data is included in the final project report.

9.3 Establishment of Detection Limits for Parameters

Detection Limits for all organic and inorganic TCL parameters in soil and water are listed in Tables 9-1 through 9-5. Enseco Laboratory anticipates achievement of these detection limits; however, highly contaminated samples may have somewhat higher quantitation limits based upon the best available technology. All efforts will be made to achieve the lowest possible detection limits on all samples, including highly contaminated samples. Various cleanup procedures, such as alumina column cleanup and gel permeation chromatography, will be employed if needed.

Enseco Laboratory expects to meet all of the QC criteria, including sensitivity, precision, accuracy, and completeness as listed in Section 5.0. If outliers exist, reanalyses will be performed and documented as per the EPA CLP protocol. The case narrative will note any of these occurrences.

9.4 Initial and Continuing Calibrations

The EPA CLP criteria for initial and continuing calibrations will be strictly followed. This criteria includes multilevel calibrations for organic and inorganic analytes. Sample analysis cannot proceed until this specific criteria is met.

Specific calibration procedures for each of the TCL analyses is contained in Section 8.0. A standards package, including tabulation reports of response factors, %RSD and %D, will be provided in addition to the raw data.

9.5 Analytical Procedures for Each Sample Matrix and Parameter

Enseco's SOP for the analysis for TCL VOA, BNA and pesticide/PCB analytes in fish and the SOP for achieving low-level detection limits in drinking water for TCL VOA, BNA, pesticide/PCB analytes are presented as Attachments 1 and 2, respectively, of this QAPP. The SOP for the analysis of DPS and DCNB will be provided with the final method validation study.

9.5.1 Volatile Organics in Water and Soil

Analyses are conducted using the purge-and-trap GC/MS procedure in accordance with EPA CLP methods. All procedures, including purge-and-trap, tuning, calibration of the GC/MS system, quantitation, quality control, and reporting protocols, are in strict adherence with the August 1987 revision of the EPA's CLP protocols. A target compound list and detection limits for all volatile compounds are listed in Table 9-2. This table also contains low-level

detection lists requested on selected water samples. The methods that the laboratory will use to achieve these detection limits are covered in Section 9-7.

9.5.2 Semivolatile Organics in Water and Soil

The extraction and analysis procedures are conducted in accordance with the EPA CLP protocol. All procedures, including extraction, tuning calibration of the GC/MS system, quantitation, quality control, and reporting protocols, are in strict adherence with the August revision of the EPA's CLP protocols. Detection limits for semivolatiles in water and soil are listed in Table 9-1. This table also contains low-level detection lists requested on selected water samples. The methods that the laboratory will use to achieve these detection limits are covered in Section 9-7.

9.5.3 Pesticides/PCBs in Water and Soil

The analyses of organochlorine pesticides and polychlorinated biphenyls are conducted in accordance with EPA CLP methods.

The extract is run on a GC/ECD using a packed column. The analyte is quantitated on a GC/ECD after a second column confirmation. All procedures, including extraction, analysis, quantitation, quality control, and reporting protocols, are in strict adherence with the August 1987 revision of the EPA's CLP protocols. Detection limits for pesticides and PCBs in water and soil are listed in Table 9-3.

9.5.4 Polychlorinated Dibenzo-Dioxins and Polychlorinated Dibenzofurans in Water and Soils

Analyses of polychlorinated dibenzo-dioxins and polychlorinated dibenzofurans will be performed by Enseco at California Analytical Laboratory using U.S. EPA PCDF/PCDD protocol, exhibit D, analytical methods, draft April 19, 1989 and revised in November 1989. This procedure uses a matrix-specific extraction, an analyte-specific cleanup, and a high resolution capillary column GC/low resolution mass spectrometry. If interferences are encountered, the method defines several cleanup procedures to minimize the interference.

If tetrachlorodioxins are found, the extract will be analyzed for 2,3,7,8-TCDD by EPA CLP SOW 8/87. Subsequent column cleanup procedures are utilized as needed. Detection limits for polychlorinated dibenzo-dioxins and polychlorinated dibenzofurans in water and soil are listed in Table 9-5.

9.5.5 Inorganics in Water and Soil

To determine the metal content of a sample, Enseco uses either atomic absorption spectroscopy or inductively-coupled plasma (ICP). Acid digestion and analytical procedures are in strict adherence with the December 1987 revision of the EPA's CLP protocols. Detection limits for inorganics in water and soil are listed in Table 9-4.

9.5.6 Cyanides in Water and Soils

Analysis of cyanides will be performed using the December 1987 revision of the EPA CLP protocols. Detection limits are listed in Table 9-4.

9.6 Chain-of-Custody

Chain-of-custody in the laboratory starts with the Sample Custodian, who receives the samples, coordinates them with chain-of-custody records, logs the samples into the computerized tracking system, and assigns a unique Enseco identification number to them. A booking log form is filled out with documents Enseco Sample ID number, Client ID, date sampled and test request. An internal case number will be assigned. This case number will correspond to all samples received within one sampling week. Matrix spikes, matrix spike duplicates, duplicates, and blanks will be assigned upon log-in.

Once the project assignment is completed and approved by the project manager, the samples are delivered to the appropriate labs, where the personnel sign and date the internal chain-of-custody forms. If there is no preparation step required, the analysis proceeds, and unused portions of the sample are stored. If there is a preparation step involved, the resulting extract is transferred to the appropriate analytical laboratory and an extract transfer logbook is filled out, signed, and dated to document internal extract chain-of-custody.

Additional information on sample custody and chain-of-custody is provided in Section 7.

9.7 Analytical Procedures for Non-CLP Parameters

9.7.1 Mirex, Photomirex and Kepone in Water and Soil

The analytical procedure for mirex, photomirex, and kepone utilizes GC/MS with the Pulsed Positive Negative Ion Chemical Ionization (PPNICI) detection system. The

extraction and analysis procedures were developed at Enseco to achieve low level detection limits in the various matrices. These methods have been validated at Enseco. The following is a detailed description of preparation, analysis and quality control procedures for these compounds. Detection limits are listed in Table 9-6.

9.7.2 Sample Preparation for Mirex, Photomirex and Kepone in Water

The following extraction procedure will be followed when analyzing for mirex, photomirex, and kepone in water.

1. Add 1,000 ml of sample to a separatory funnel.

2. Add approximately 0.5 ml concentrated HCL.

3. Add 100 ng of ^{13}C-mirex.

4. Add 60 ml methylene chloride and shake for 2 minutes.

5. Allow the layers to separate for 10 minutes.

6. Drain the methylene chloride layer into an Erlenmeyer flask.

7. Repeat steps 4-6 two more times.

8. After the third extraction, pour the combined extract into a KD flask through a funnel containing anhydrous sodium sulfate. Rinse the sodium sulfate with an additional 50 ml methylene chloride.

9. Add 200 ul toluene to each flask.

10. Add a boiling chip to each flask and insert a Snyder condenser column into the flask. Concentrate the extract to a volume of 5 ml in a water bath at 85-90°C.

11. Replace the macro Snyder column with a micro Snyder column and concentrate further to 1 ml.

12. Remove the KD apparatus and blowdown the extract to 200 ul under a stream of nitrogen.

13. Add 5 ul of isopropanol to the 200 ul extract.

9.7.3 Sample Preparation for Mirex, Photomirex and Kepone in Soil

The following extraction procedure will be followed when analyzing for kepone, mirex, and photomirex in soil.

1. Decant and discard any water layer.

2. Add 30 g of homogenized soil to an acid-cleaned Teflon jar with 60 g anhydrous sodium sulfate and mix well.

3. Add 1000 ng of ^{13}C-mirex.

4. Add 100 ml of methylene chloride/acetone (a 70:30 mixture).

5. Agitate on an automatic shaker for 30 minutes.

6. Centrifuge for 3 minutes.

7. Decant liquid into an Erlenmeyer flask.

8. Repeat steps 4-7 two more times.

9. Quantitatively transfer combined extracts through sodium sulfate into KD apparatus. Wash the remaining soil/sodium sulfate mixture with 60 ml methylene chloride/acetone and transfer liquid into KD apparatus also.

10. Add 1 ml toluene.

11. Add a boiling chip to each flask and insert a Snyder condenser column into the flask. Concentrate the methylene chloride/acetone extract to a volume of 5 ml in a water bath at 85-90°C.

12. Replace the macro Snyder column with a micro Snyder column and concentrate further to 1 ml.

13. Remove the KD apparatus and blowdown the extract to 1 ml under a stream of nitrogen.

14. Add 25 ul of isopropanol to the 1 ml extract.

9.7.4 Analysis for Mirex, Photomirex and Kepone

9.7.4.1 GC/MS Operating Conditions

Column: J&W DB-5, 0.32 mm ID, 30 m

Initial Temp: 30°C

Initial Time: 4 min

Ramp Rate: 10°C/min

Final Temp: 290°C

Final Time: 10 min

Injection Volume: 2 ul water, air matrices
1 ul solid, fish matrices

Injection Port Temp: 240°C

Injector: Grob-type, splitless

Carrier Gas: Helium

Reagant Gas: Methane

A mass spectrometer utilizing the pulsed positive negative ion chemical ionization (PPNICI) detection system is used. The ion ranges that will be monitored for mirex, photomirex, and kepone are presented in Table 9-7.

9.7.4.2 Calibration Procedure

Initially, the GC/MS system is calibrated with a five-level calibration curve containing the following compounds: mirex, photomirex, and kepone. Calibration factors will be generated from this curve. The %RSD for mirex and photomirex must be less than 30%. For kepone it must be less than 40%. The calibration curve will establish a 12 hour window in which samples can be run. The levels of the calibration standards will be specified in the final method validation plan.

A daily continuing calibration check will be run subsequent to the initial calibration. The daily calibration will consist of the three target compounds at a mid-

TABLE 9-7

Major Ions in the Monitored Clusters for Mirex, Photomirex, Kepone and ^{13}C-Mirex.

Analyte	Quantitation Ions (First Cluster)	Confirmation Ions (Second Cluster)	Confirmation Ions (Third Cluster)
Mirex	368, 370	402, 404	437, 439
Photomirex	368, 370	402, 404	437, 439
Kepone	383, 385	418, 420	-
Internal Standard			
$^{13}C_8$-Mirex	376, 378	410, 412	445, 447

concentration range: Response factors for mirex and photomirex must be within 30% for the daily calibration standard to be used. The daily calibration will initiate a 12-hour analytical time period in which samples can be run. Calibration curves will be rerun if the daily calibration does not meet the preceding criteria.

9.7.4.3 Sample Analysis and Quantitation

Samples will be quantitated using an isotope dilution internal standard quantitation procedure. The total integrated ion current of the mass ranges will be used for quantitation unless it is found there are interferences present. In this case, one selected ion current will be used for quantitation.

Detection limits for mirex, photomirex and kepone are listed in Table 9-6. The levels of the matrix spikes and recovery standards, along with the recovery acceptance criteria, are based on the results of the method validation study and are listed in Table 5-2. Full details on the analysis of mirex, photomirex and kepone are presented in Sections 9.7.1 through 9.7.4.2 of this QAPP and in Section 4 of the Method Validation Study.

9.7.4.4 Reporting Format

All raw data, including standards packages, chromatograms, extracted ion current profiles (EICPs), quantitation reports, and spectra will be provided in the final report. Tabulations of standards and recoveries will also be provided.

9.7.4.5 Quality Control Procedures

One method blank will be extracted with every 20 samples or every time samples are set up, whichever is more frequent. Holding times are listed in Section 6. A matrix spike and matrix spike duplicate, containing each of the three

compounds, will be extracted with every set of 20 samples, or with every case, whichever is more frequent. The recovery percent and difference relative percent will be calculated. Control limits will be set at ±3 SD of the mean recovery value derived from the method validation study.

9.7.5 Analysis of Diphenyl sulfone (DPS) and Dichloronitrobenzene (DCNB) in Water and Soil

The diphenyl sulfone (DPS) and dichloronitrobenzene (DCNB) will be analyzed as part of the semivolatile analysis. The GC/MS system will be calibrated for all of the compounds indicated in the August 1987 EPA contract. In addition to this, a five-point calibration will be run for DPS and DCNB, and average response factors will be generated. These compounds will also be run on a daily basis as a continuing calibration standard. The quantitation ion mass for DPS will be m/z 125 and for DCNB m/z 145. Quantitation, identification, and reporting protocols are in strict adherence with the August 1987 revision of the EPA's CLP protocols. Detection limits for DPS and DCNB in water and soil are listed in Table 9-1. Full details on the analysis of DPS and DCNB are presented in Section 9.7.5.1 through 9.7.5.4 of this QAPP and in Section 3 of the Method Validation Study.

9.7.5.1 Sample Preparation for DPS and DCNB

DPS and DCNB will be extracted out of water and soil along with the other semivolatile organics. Therefore, a separate extraction for the two compounds will not be done. The EPA CLP semivolatiles extraction procedure will be followed (EPA CLP SOW 8/87).

9.7.5.2 Analysis of DPS and DCNB

DPS and DCNB will be analyzed for in the semivolatile GC/MS fraction. GC conditions and MS scan ranges in the EPA

CLP method will detect the presence of either of the compounds.

9.7.5.3 Calibration Procedures

An initial five-point calibration will be run for DPS and DCNB at 20-160 ppb. The %RSD for the compounds must be less than 30%. A daily continuing calibration will be run. For the continuing calibration standard, %D for the two compounds must be less than 30%. The same analytical procedure (i.e. DFTPP tune requirements 12 hour analytical time window) will be followed as that for the EPA CLP semivolatiles.

9.7.5.4 Quality Control Procedures

One method blank will be extracted with every 20 samples or every time samples are set up, whichever is more frequent. Holding times are listed in Table 6-1. DPS and DCNB will be added to the semivolatiles matrix spike. Control limits derived from the method validation study are listed in Table 5-2.

9.7.6 Fish Tissue Analysis

Preparative extraction and cleanup procedures used by Enseco for analysis of fish tissue can be found in "Interim Methods for the Sampling and Analysis of Priority Pollutants in Sediments and Fish Tissue," EPA 600/4-81-055 (Revised October 1980). Holding times are listed in Section 6. A brief method description of each of these procedures will follow. Any modifications to the above referenced procedures would be in the area of aliquot size, which is dictated by the size of the sample available.

Detection limits that can be expected for the various analyses in fish tissue are approximately the same as those for soil but may be somewhat higher due to sample size and matrix interferences. Results will be reported on a wet weight basis.

Once sample extracts have been generated through the above referenced methods, EPA CLP methods will be used to analyze the extracts. Therefore for fish analysis, refer to the appropriate soil analysis for method descriptions. The only deviation from the EPA CLP methods will be control limits.

9.7.6.1 Semivolatiles, Pesticides, PCBs, Mirex, Photomirex, and Kepone in Fish Tissue

For the larger upper trophic level fish, the laboratory will fillet the fish prior to analysis. In the case of all lower trophic level fish and smaller upper trophic level fish, the laboratory will use the entire fish as the sample. The frozen tissue or fish is first homogenized in a Tissuemizer. Four aliquots of this fish are taken, using a 30 gm sample size for each aliquot of semivolatiles, and a 5 gm sample aliquot for volatiles. For semivolatiles, pesticides, and PCBs the aliquot is mixed with sodium sulfate and soxhlet extracted using a methylene chloride/methanol mixture. The semivolatiles and pesticide/PCB aliquots are spiked, prior to extraction, with EPA CLP surrogate mixtures. The extracts are then washed with an acidic sodium sulfate saturated water solution, and the acidic water is reextracted and the resulting extract is combined with the initial extract. The extract is then concentrated and subjected to GPC cleanup prior to analysis.

9.7.6.2 Volatile Organics in Fish Tissue

The homogenized tissue will be directly purged in water. EPA CLP internal and surrogate standards will be used to monitor purging efficiency and recovery. Instrument tuning, calibration, and quantitation procedures will be EPA CLP methods for volatile organics in soil. It should be noted that some fish tissue absorbs internal and surrogate standards, therefore purging efficiency and recoveries could be affected.

9.7.6.3 Quality Control Procedures

One method blank will be extracted with every 20 samples or every time samples are set up, whichever is more frequent. A matrix spike and matrix spike duplicate will be performed for every 20 samples. The five non-TCL extractable compounds will be added to the matrix spike. Surrogate spikes will be added according to CLP protocol. The control limits are listed in Tables 5-2 and 5-3.

9.7.7 Low-Level Detection Limits for Selected Water Samples

Detection Limits on selected water samples are listed in Tables 9-1 through 9-6. The following methods will be used to achieve these detection limits.

9.7.7.1 Semivolatiles and DPS, DCNB

To achieve the detection limits requested for the selected water samples, only minor adjustments to the EPA CLP method will be made. Most compounds on the TCL list are detected at the levels referenced in the Selected Water Column in Table 9-1 using the CLP method. To decrease the detection limits, the extracts will be concentrated to a final volume of 0.5 ml, which will yield of a four time sensitivity increase over the standard EPA CLP method, which calls for a final volume of 2.0 ml.

EPA CLP surrogate and matrix spikes will be used at 50% concentration.

All other procedures including tuning, calibration, quantitation, QC, and documentation will be according to EPA CLP methods.

9.7.7.2 Pesticides/PCBs

To achieve the detection limits on selected water for pesticides/ PCBs, the EPA CLP method will be employed.

Detection limits for this method, listed in Table 9-3, can be achieved using standard CLP methods.

9.7.7.3 Volatile Organics

EPA Method 524.2 will be used to achieve the detection limits listed in Table 9-2. Method 524.2 can be found in "Methods for the Determination of Organic Compounds in Finished Drinking Water and Raw Source Water", U.S. EPA, September 1986.

EPA CLP calibration, quantitation, QC, and reporting protocols will be followed. All raw data will be provided in the final report.

9.7.7.4 Mirex, Photomirex and Kepone

The extraction and PPNICI GC/MS procedure outlined in Section 9.7.1 was developed to achieve the detection limits listed in Table 9-1 for Selected Water Samples.

9.7.8 Air Analysis

9.7.8.1 Semivolatiles, DPS and DCNB

The samples will be taken in XAD-2 tubes. The tubes will be desorped as follows:

1. Break the tube. Empty the material into methylene chloride rinsed Vortex test tubes. Do not empty the glass wool into the Vortex tube. A blank tube will be provided.

2. Spike with 1 ml of BNA surrogate.

3. If the tube provided is for a matrix spike/matrix spike duplicate, spike with 10 ml of a 10 ng/ul solution of EPA CLP matrix spike solution before breaking the tube.

4. Add 8-9 mls of methylene chloride.

5. Mix on the Vortex 1-2 minutes.

6. Decant the methylene chloride into a methylene chloride rinsed 40 ml Teflon screw top vial.

7. Repeat steps 4-6 two more times.

8. The final extracts volume will be concentrated to 1 ml.

For analysis, EPA CLP tuning, calibration, and quantitation procedures will be followed. EPA CLP Quality Control will be followed, including a method blank with every set of samples extracted, and a matrix spike/matrix spike duplicate with every set of 20 samples or every case, whichever is more frequent. Control limits for surrogate and matrix spike recoveries are presented in Table 5-2. All raw data will be provided in the final report.

9.7.8.2 Organochlorine Pesticides/PCBs

EPA Method TO4 will be used for the Organochlorine pesticides/PCB analysis in air. This method involved soxhlet extraction of a glass fiber filter with a polyurethane foam absorbent cartridge. The extraction solvent is 5% diethylether in hexane, and the extraction time is 14-24 hours.

The resulting extract is concentrated to 10 mls, and a 5 ml portion is taken for the pesticide/PCB analysis. This portion is concentrated to 1 ml and analyzed in accordance with EPA CLP protocols.

The other portion of the extract will be analyzed for mirex, photomirex, and kepone. Because of possible interferences in the negative CI spectrum, DBC will not be spiked into the samples.

EPA CLP QC will be followed, including a method blank, and a matrix spike/matrix spike duplicate per every 20 samples. (EPA method TO4 is contained in Compendium Methods for Determination of Toxic Organic Compounds in Ambient Air, EPA 600/4-84-040.) Control limits are presented in Table 5-2.

9.7.8.3 Volatile Organics

EPA Methods TO1 and TO2 will be used for VOA priority pollutant analysis in air. Method TO1 involves thermal desorption of the Tenax cartridge directly onto a cold trap. The organic compounds are then transferred onto the front of a GC column and analyzed by GC/MS. (EPA Methods TO1 and TO2 are contained in Compendium Methods for Determination of Toxic Organic Compounds in Ambient Air, EPA 600/4-84-040).

Method TO2 involves thermal desorption of Carbon Molecular Seive Tubes onto a trap.

The TO1 and TO2 thermal desorption analyses for the volatile organic compounds will be performed at the Enseco-CRL/El Monte Air Toxics Laboratory. Calibration protocols from EPA Methods TO1 and TO2 will be followed. Reporting protocols and deliverables will be EPA CLP.

Presently, there is no technically acceptable method for preparing and analyzing matrix spikes utilizing these methods, thus matrix spike analysis will not be done. Method blank analysis will be performed with every set of samples taken, or every 20 samples, whichever is more frequent.

9.7.8.4 Mirex, Photomirex and Kepone

A portion of the extract from the Organochlorine pesticides/PCB air sample will be analyzed for mirex, photomirex, and kepone. The extract will be concentrated to 200 ul and analyzed according to Section 9.7.1.

Control limits will be established based on the results of the method validation study and are listed in Table 5-2.

9.7.9 Physical Parameters

GZA will perform the analysis of the physical parameters on the specified soil borings. The methods that will be utilized for each parameter are as follows:

Parameter	Method
Moisture	ASTM*-D-2216
Soil Classification	ASTM*-D-2487
Atterburg Limits	ASTM*-D-4318
Specific Gravity	ASTM*-D-854
Permeability	US Army Corp EM1110-2-1906
Grain Size	ASTM*-422
Porosity	Calculated by density, specific gravity and unit weight

*ASTM - American Society of Testing and Materials

10.0 DATA REDUCTION, VALIDATION AND REPORTING

10.1 ERM's Data Reduction, Validation and Reporting

Data validation practices will be followed to insure that raw data are not altered and that an audit trail is developed for those data which require reduction. All the field data, such as those generated during field measurements, observations and field instrument calibrations, will be entered directly into a bound field notebook. Each project team member will be responsible for proofing all data transfers made, and the Field Operations Manager will proof at least 10 percent of all data transfers.

Upon receipt of the sample data packages, the laboratory data will be quantitatively and qualitatively validated by ERM's Project QA/QC Manager. The data will subsequently be submitted to Region 5 of the U.S. EPA. Data validation is discussed in detail in Section 14.

It is anticipated that ERM's data reduction for this investigation will be minimal and will consist primarily of tabulating analytical results from Enseco's Form 1s (Analytical Reports) onto summary tables through the use of computerized spreadsheet software. All reduced data will be assigned document control identification numbers and placed in the final evidence file maintained by the Project Manager.

All analytical data obtained during the course of the investigation for ground waters, surface waters, seeps and leachates will be reported as ug/1 (organics and inorganics). Solid sample results will be reported as ug/kg (organics) and mg/kg (inorganics) on a dry weight basis. Dioxin/furan, mirex, kepone and photomirex will be reported in parts-per-billion (ppb). Data packages associated with the analyses of samples collected during the RI will be prepared utilizing full CLP deliverables. The required deliverables are stated in Section B of EPA CLP SOW 8/87 for RAS organic, dioxin and inorganic IFB.

10.2 Laboratory Reduction, Validation and Reporting

10.2.1 Data Reduction

Raw data obtained from instrument readouts, chromatograms, strip chart recorders, and physical measurements are reduced to concentrations of analytes by use of the appropriate equations given in each of the methods referenced in Section 9.

10.2.2 Data Validation

Data validation is the process of reviewing data and accepting or rejecting it on the basis of sound criteria. The data generated receive both technical and editorial review. Technical review concerns itself with the analytical techniques and their effect on data validity. Editorial review ensures that the text is concise and lucid and that it contains no transpositional errors. Enseco scientists use appropriate validation methods and criteria to validate data. The criteria used in evaluating data include:

- Accuracy requirements
- Precision requirements
- Detection limit requirements
- Completeness
- Representativeness

For research and methods development-type parameters, the acceptable warning and control limits may be statistically defined as analytical results become available. Routine analytical procedures for which windows have not been established are subject to an acceptance limit of ± 3 SD (three standard deviations).

10.2.3 Data Reporting

Complete data packages inclusive of all raw data, will be submitted for each of the analyses. The data reporting protocols can be found in the EPA CLP methods. These data packages will include bar graphs and listings from BFB and DFTPP tones, raw data from standards including %RSD and %Difference tabulations, raw data from samples and blank runs, including mass spectra, and summary sheets with surrogate recoveries, matrix spike recoveries, and method blank data.

Qualifiers used when reporting data are found in the EPA CLP contract. Sample results will not be blank corrected. Case narratives will be provided which document any non-standard occurrences within the case. Any and all non-standard occurrences will be relayed to the project manager at ERM and the Project QA/QC Manger. They will then be responsible for notifying the appropriate personnel and implementing project level corrective action, such as resampling.

A "Case" will be considered one sample delivery group (samples received at the laboratory during a seven day period). Non-CLP parameters will be reported in the following manner. Dichloronitrobenzene and diphenyl sulfone will be added to the semivolatiles target compound list and reported in the semivolatiles sections. Mirex, photomirex, and kepone will be reported in a separate report section and subsections of this report will include all raw data, including standard chromatograms, EICPs, quantitation reports, associated sample and standard spectra, summary sheets of sample results for all samples, blanks and matrix spikes. Sample results will not be blank corrected. Qualifiers used will be those found in the EPA CLP contract.

The key individuals from Enseco, who will handle data reporting, are the following laboratory senior personnel:

Volatiles, W. Henry Camp, Charlene Livingston; Semivolatiles, DPS, DCNB, Peter Kane, Linda Cook; Pesticides and PCBs, Ellie Kwong, Mary Ellen Fitzpatrick; Mirex, Photomirex, and Kepone, Agnes Van Langenhove, Peter Kane; Inorganics, Susan Chapnick, Vaughn Pusey; Dioxins/Furans, William Lukjemburg, Steven Rodgers; and Air Methods T01 and T02, Steven Harris.

11.0 INTERNAL QUALITY CONTROL

11.1 Field Quality Control Checks

Field Quality Control Checks will be utilized during this investigation through the use of the following:

- Trip Blank -- One trip blank will be submitted for each cooler of aqueous samples for the analysis of volatile organics. One trip blank in 20 solids/biota will be submitted for volatile analysis. Trip blanks will be prepared by the laboratory with deionized water, preserved with HCL, transported to the site, handled like a sample, and returned to the laboratory for analysis. Trip blanks for air samples will consist of sealed Tenax and CMS cartridges transported to the site and returned unopened to the laboratory for analysis. One trip blank will be submitted for 6 investigation samples. Trip blanks are not opened until they are received by the laboratory for analysis.

- Field Blanks -- Field blanks are prepared in the field to ensure a sampling device (e.g., bailer or pump) has been effectively cleaned. The sampling device is filled with deionized water or deionized water is pumped through the device, transferred to the appropriate sample bottles, preserved and returned to the laboratory for analysis. Field blanks for solid matrix are prepared by pouring deionized water through the solid sampling equipment directly into appropriate solids bottleware and one will be submitted for every 20 solid samples collected. Field blanks for air analysis will be prepared by opening each

canister/cartridge on-site and allowing any ambient contaminants to passively diffuse onto the collection media. The field blank will be collected at an upwind location. One field blank will be submitted for the air samples. One field blank will be collected for every 20 groundwater samples. The one per 20 samples for field blanks for groundwater samples apply since dedicated sampling equipment will be utilized for the collection of samples from each monitoring well. One field blank will be collected for every 10 surface water samples or everyday of sampling (whichever is more frequent).

- Field Duplicates or Replicates -- Two sets of samples from a single sample location are obtained (replicates) or prepared (duplicates), labeled with unique sample numbers, and submitted to the laboratory to determine analytical precision and sample representativeness. Field duplicates/ replicates will be collected at a frequency of 1 per 10 samples per matrix.

- Performance Evaluation (PE) -- Samples submitted for dioxin/furan analysis will be accompanied by known concentration PE samples. These PE samples will be supplied by the U.S. EPA. The acceptable concentrations (within 95% CI) will be requested of the U.S. EPA after data arrives at ERM since this information is necessary to assess analytical accuracy. One PE sample will be submitted with each batch of samples for dioxin/furan analysis.

- Matrix Spike/Matrix Duplicate Samples -- Matrix Spike (MS) and Matrix Spike Duplicate (MSD) samples will be submitted as further QC checks. One MS and MSD in every twenty (20) ground water, surface water, soil, sediment and fish samples will be collected and submitted for organic

analysis. These will allow accuracy to be determined by the recovery rates of compounds (the matrix spike and/or surrogate spike compounds defined in the analytical methods). The purpose of these laboratory spikes is to monitor any possible matrix effects specific to samples collected from the site. The addition of known concentrations of compounds/constituents into the sample also monitors extraction/digestion efficiency.

The analysis of MS and MSD samples checks precision by comparison with the respective spiked recoveries. In addition, any non-spiked analytes present in an unspiked sample will be compared with the results of the non-spiked compounds in the MS and MSD. Therefore data will be assessed for precision for this triplicate analysis. The analysis of laboratory duplicates (inorganics) is an excellent assessment of analytical precision.

Duplicate/replicate and matrix spike sample aliquots will be acquired for ground water, surface waters, and fish by collecting sequential grab samples after the collecting of the initial sample aliquot. Therefore, the specific sample location which will be used for matrix spikes and duplicates/replicates will be chosen by the Field Operations Manager. Multiple air samples will be collected simultaneously at a given sample location for replicate samples. Soil duplicate or replicate and matrix spike samples will be collected by splitting the sample between the sample container, duplicate container, and matrix spike container. Homogenizing <u>will</u> <u>not</u> be performed for the aliquots designated for volatile organic compounds; therefore, they are designated as sample replicates. If insufficient soil sample is present at a particular location to collect the three sample volumes, a single sample bottle will be split at the laboratory for MS and MSD analysis.

11.2 Laboratory Quality Control Checks

All QC procedures employed at Enseco Laboratory are, at a minimum, equivalent to those described in the EPA CLP methods (referenced in Section 9). Each individual Enseco Laboratory maintains its own internal QC program as summarized below. The various control limits for continuing calibration checks for analysis of mirex, kepone, photomirex, DPS and DCNB will be specified in the final method validation study.

11.2.1 Trace Organic Analysis: Volatiles, Semivolatiles, Pesticides and PCBs, Dioxins, Mirex, Photomirex, and Kepone

General QC protocols for trace organic analyses include:

- Field blanks, when applicable, to detect contamination introduced during sampling, shipping and handling.
- A minimum of one procedural blank (method blank) in every 20 samples analyzed to detect contamination during analysis.
- One matrix spike and one matrix spike duplicate per every 20 samples, or per case, whichever is more frequent; to determine recovery, precision, and the presence of matrix effect.
- Surrogate standards and internal standards to quantitate results, determine recoveries and to account for sample-to-sample variation.
- Routine analysis of performance evaluation samples and blind spikes to document method accuracy.
- Multilevel initial calibrations of instruments to establish calibration curves.

- Continuing calibration standards at least once every 12 hours of instrumental analysis for accurate quantitation, and recalibration if these do not meet EPA CLP criteria.

- Calibration of GCs and GC/MSs according to the appropriate EPA CLP methods.

- Tuning of GC/MS systems every 12 hours to meet EPA criteria using BFB (bromofluorobenzene) for volatile organics analysis, and DFTPP (decafluorotriphenylphosphine) for semivolatile analysis, as described in Section 8.0.

11.2.2 Trace Metals Analysis

General QC protocols for trace metals analyses include:

- Analysis of moderate-to-high concentration levels by ICP (inductively coupled plasma spectroscopy) or flame atomic absorption spectroscopy.

- Analysis of low-level metal concentrations by graphite furnace atomic absorption spectroscopy.

- Calibration blanks and method blanks prior to and between the analysis of samples.

- Multilevel calibration curves generated by analyses of individual standards (AA) or mixed standards (ICP).

- Initial calibration verification (ICV) and Continuing Calibration Verification (CCV) at a minimum of one every ten samples to verify instrument calibration.

- ICP Interference Check Standards after initial calibration and after samples are analyzed.

- Recalibration and reanalysis of applicable samples if check standard response deviates by more than 10% (15% for mercury) from the initial calibration

- A minimum of one matrix spike in every 20 samples or one for each sample matrix type

- A minimum of one duplicate in every 20 samples analyzed.

- A minimum of one Laboratory Control Spike for every 20 samples analyzed.

- A minimum of one method blank for every 20 samples.

- Digestion of solid or sludge samples according to EPA procedures.

11.2.3 Control Limits

Control limits used will be those defined by the EPA-CLP methodology. For fish tissue and air samples, the control limits listed in Tables 5-2 and 5-3 will be used.

For the five non-routine analytes, control limits will be established from the method validation data. The control limits will be ± 3 SD from the mean. These will be presented in the final method validation study.

12.0 PERFORMANCE AND SYSTEM AUDITS

12.1 External Audit of Laboratory by U.S. EPA Region 5

Enseco participates in a wide variety of certifications, programs, and contracts, it is subjected to rigorous external audits by many government regulatory agencies and industrial clients. Tables 12-1 and 12-2 provide lists of such audits. EPA Quarterly Blind Performance Evaluation Studies are also performed; recent results of these studies are given in Table 12-3.

Enseco presently holds EPA CLP contracts for Organics and Inorganics, and is audited on a regular basis by the U.S. EPA under these contracts. Quarterly performance evaluations are also performed under each of these contracts. For this project, Enseco, CAL Analytical, and CRL/El Monte are available for an audit by U.S. EPA Region 5 Central Regional Laboratory (CRL).

12.2 Laboratory Internal Audits

Each laboratory is subjected to quarterly systems audits by Enseco's QA Director as well as by an external team consisting of the Corporate Vice President of QA and the QA Directors of the other Enseco laboratories. These audits are intended to serve two purposes: 1) to ensure that laboratories are complying with the procedures defined in laboratory manuals and contracts, and 2) to determine any sample flow or analytical problems. The frequency of the audits will be increased if problems are encountered. Figure 12-1 outlines the general form a laboratory audit takes and the items that may be covered.

Internal performance audits are also conducted on a quarterly basis. The Vice President of QA submits a spiked sample to each laboratory as a routine sample and reviews the data after the analyses are completed. This is one of

Table 12-1

Erco systems audits, January 1987 to December 1987

	Auditor	Date
1)	California Department of Health	Feb. 26, 1987
2)	Massachusetts DEQE	March 19, 1987
3)	Anthony S. Wong, Vice President Quality Assurance and Safety Enseco Incorporated	March 30, 1987
4)	New York Department of Health	April 6 & 7, 1987
5)	Geraghty & Miller, Inc.	April 13, 1987
6)	Anthony S. Wong, Vice President Quality Assurance and Safety Enseco Incorporated	April 20 & 21, 1987
7)	Florida Department of Health and Rehabilitative Services	May 4, 1987
8)	Woodward-Clyde Consultants (volatile data)	May 29, 1987
9)	Dr. Joseph O'Brien Certification Officer Massachusetts DEQE	June 11, 1987
10)	Dr. Anthony S. Wong, Vice President Quality Assurance and Safety Enseco Incorporated	July 21-24, 1987
11)	Edwin Perkins, Clive Morrick NYS DEC, Department of Law	August 4, 1987
12)	Browning-Ferris, Inc. (BFI)	August 13, 1987
13)	U.S. EPA Teklaw, Lockheed Inorganic CLP audit	August 31, 1987
14)	Julia Wilcox Enseco Incorporated Laboratory Walkthrough	October 5, 1987
15)	Edward Maser Commonwealth of Pennsylvania	November 6, 1987
16)	Martin Marietta Army Corps of Engineers	December 16, 1987

Table 12-2

Erco performance evaluations, January 1987 to December 1987

Agency/Program	Parameter(s)	Date
1) ERA Quarterly	VOA, metals, inorganics, semi-volatile organics, PCBs, pesticides	Feb. 13, 1987
2) U.S. EPA - New CLP Contract Preaward	Semivolatile organics, VOA, PCBs, pesticides	Feb. 14, 1987
3) NYS DOH Nonpotable Water	VOA, metals, pesticides, inorganics, semi-volatile organics, PCBs	March 15, 1987
4) U.S. EPA VOA Preaward	VOA	Feb. 17, 1987
5) U.S. EPA NEIC QB-2, FY-87 Case No. 6666	Semivolatile organics, VOA, PCBs, pesticides	Feb. 20, 1987
6) OSW Study IX	Semivolatile organics, (pesticides), metals	Feb. 23, 1987
7) NJ Check (WS-18)	Fluoride, endrin, methoxychlor, lindane	Feb. 18, 1987
8) U.S. EPA Water Pollution Study WP-018	VOA, metals, inorganics, pesticides, PCBs	Apr. 5, 1987
9) U.S. EPA Water Supply Study WP-020	VOA, metals, inorganics, pesticides, herbicides	May 18, 1987
10) NY DOH Potable Water Study (Drinking Water)	Trihalomethanes, metals, pesticides, herbicides,	May 26, 1987
11) U.S. EPA-OSW Study X	Semivolatile organics, (PAH) metals	May 18, 1987
12) Enseco Blind Sample Check	Semivolatile organics, VOA, metals, inorganics, PCBs, pesticides	May 8, 1987
13) ARCO	Petroleum hydrocarbons/ biomarkers	June 1, 1987

Table 12-2 (Continued)

Erco Performance Evaluations, January 1987 to December 1987

Agency/Program	Parameter(s)	Date
14) U.S. EPA NEIC QB-3, FY-87, Case No. 7144	VOA, semivolatile organics, pesticides, PCBs	May 28, 1987
15) New York Nonpotable Water	Semivolatile organics, VOA, inorganics, metals, PCBs, pesticides	Aug. 17, 1987
16) Enseco Quarterly PE (3rd Quarter)	Semivolatile organics, VOA, inorganics, metals, pesticides, PCBs	Aug. 24, 1987
17) U.S. EPA-OWS Study XI	Semivolatile organics, EP Tox metals	Aug. 24, 1987
18) U.S. EPA-CLP QB-4, FY87	Semivolatile organics, VOA, pesticides	Sept. 24, 1987
19) U.S. EPA Water Pollution Study WP-019	VOA, pesticides, PCBs, metals, inorganics	Sept. 28, 1987
20) Army Corps of Engineers		Oct. 26, 1987
21) NY DOH Potable Water Study (Drinking Water)	Trihalomethanes, metals, pesticides, herbicides, inorganics	Nov. 2, 1987
22) Enseco Quarterly PE (4rd Quarter)	Semivolatile organics, VOA, inorganics, metals, pesticides, PCBs	Nov. 11, 1987
23) U.S. EPA Water Supply Study WS-021	VOA, metals, inorganics, pesticides, herbicides	Nov. 16, 1987
24) U.S. EPA OSW	Semivolatile organics, EP Tox metals	Dec. 7, 1987

Table 12-3

EPA Quarterly Blind Performance Evaluation Studies

EPA ID	Date	Raw Score (%)
1) QB4, FY-84, Case #3061	Nov. 1984	82
2) QB1, FY-85, Case #3287	Feb. 1985	77
3) QB2, FY-85, Case #3821	May 1985	96
4) QB3, FY-85, Case #4183	Aug. 1985	90
5) QB4, FY-85, Case #4604	Dec. 1985	93
6) QB1, FY-86, Case #5076	March 1986	100
7) QB2, FY-86, Case #5423	May 1986	99
8) QB3, FY-86, Case #5772	Aug. 1986	94
9) QB4, FY-86, Case #6076	Nov. 1986	83.7
10) QB1, FY-87, Case #6437	Jan. 1987	96
11) QB2, FY-87, Case #6666	May 1987	96
12) QB3, FY-87, Case #7144	July 1987	100
13) QB4, FY-87, Case #7760	Dec. 1987	97

Figure QAPP 12-1

ENSECO-ERCO Audit Outline

- Laboratory Audit - General Considerations
  - Sample Flow through Lab
  - Chain of Custody
    - Usual
    - Strict
  - Sample Storage
    - Controlled Access
    - Proximity to Chemical Storage
    - Physical Conditions, e.g., Temperature
    - Holding Times
  - Sample Work-up and/or Analysis
    - SOPs
    - Logbooks
      - Standards Preparation
      - Instrument - Sample Analysis
      - Calibration/Tune
      - Standards Analyses
      - Check Samples
      - Balance
      - Temperature
    - Notebooks
      - Dates
      - Signature
      - Filled Pages
      - Initialed Errors with Single-line Crossouts
      - Units Recorded
    - QC Samples
      - Blanks
      - Spikes
      - Duplicates
      - Surrogates
      - Control Charts
  - Data File Storage
    - Hard Copies
    - Other Media - Magnetic Tape, Disk
  - Laboratory Safety
  - Organization, Order
- Sampling
  - Container Preparation
  - Preservative(s)
  - Techniques
- QA Access - Corrective Action Forms

the most valuable techniques for evaluating the quality of the entire analytical system, including personnel, instrumentation, methodology, and analytical procedures. Both the frequency and the number of spiked samples will be increased if problems are encountered or when the workload increases.

12.3 On-Site Field Audit Procedures

An on-site system audit will be performed monthly during major field activities to review all field-related quality assurance activities. The system audit will be conducted by the Project QA/QC Manager. Figure 12-2 presents ERM's Quality Assurance Audit forms. Deficiencies found during the audits will be brought to the attention of the responsible individuals and corrective action as per Section 15 of this QAPP will be initiated. Copies of the audits will be distributed to all appropriate project personnel.

Specific elements of the on-site audit include the verification of:

- Completeness and accuracy of sample Chain-of-Custody forms, including documentation of times, dates, transaction descriptions, and signatures.

- Completeness and accuracy of sample identification labels, including notation of time, date, location, type of sample, person collecting sample, preservation method used, and type of testing required.

- Completeness and accuracy of field notebooks, including documentation of times, dates, drillers' names, sampling method used, sampling locations, number of samples taken, name of person collecting samples, types of samples, results of field

FIGURE QAPP 12-2

ERM QUALITY ASSURANCE AUDIT

PROJECT _____ W.O.# _____

DATE _____ AUDIT CONDUCTED FROM ___ HR. TO __ HR.

AUDITOR(S): _____

_____

ON-SITE SAMPLING PERSONNEL: _____

_____

Audit Conducted on the Following:

_____ Soil Sampling
_____ Surface Water/Sediment
_____ Ground Water
_____ Decontamination
_____ Health & Safety

Sample Collection:

Do sampling locations agree with those specified in the Work Plan? _____

Is the sampling locating either documented sufficiently or marked to allow it to be found/sampled again in the future? _____

Are sampling times, ERM Traffic Report Numbers and sample description noted? _____

Is sampling proceeding from the suspected least contaminated area to the most contaminated area? _____

Have sample bottles been labeled properly? _____

Have proper containers and preservatives been used? _____

Are proper sample volumes procured? _____

Are samples being refrigerated/iced immediately after collection? _____

Does a travel blank exist for each matrix present? _____

Does the potential for sample cross-contamination exist based on procedures observed? _____

FIGURE QAPP 12-2 (cont'd)

Soil Sampling (Check if not applicable _____):
Type: _____ Hand _____ Auger or Rig

Are samples being collected at proper depths? _____

Are samples being screened with an OVA (if specified in Work Plan and applicable)? _____

Is a description of soils/materials being logged? _____

(Have soils been homogenized where applicable (specified by the Sampling Plan)? _____

Surface Water/Sediment Sampling (Check if not applicable _____):

Have stream flow and velocity parameters been noted? _____
Estimated _____ or Measured _____

Has sampling proceeded from downstream to upstream locations? _____

Has the sampler acquired the water sample upstream of his position to minimize suspended sediment from entering the sample? _____

Have water samples been collected in the mixing zone, not stagnant areas? _____

Have sediments been characterized as to type and size distribution? _____

Has the proper sediment fraction (fine, depth) been sampled for the analyses of interest? _____

Are the selected locations effectively monitoring effects of the potential source? _____

Ground Water Sampling (Check if not applicable _____):

Have the well specifications been noted properly (i.e., Total Depth, Casing diameter, Depth-to-water to the nearest one-hundredth of a foot, etc.)? _____

Has the purge volume been calculated properly? _____

What evacuation method has been used?
_____ Bailer _____ Submersible _____ Bladder Pump
_____ Other (_____)

If metals are being analyzed, have the samples been field filtered? _____

FIGURE QAPP 12-2 (cont'd)

Are field pH, conductivity, and temperature being measured? _____ Is there documentation of calibrating the instruments? _____

Has well yield been properly evaluated to determine when sample aquisition should take place (i.e., well goes dry and needs to recover). _____

Is bailer line and bailer dedicated to each well and line disposed of after use? _____
Bailer type _____ Line type _____

Have appropriate measures been taken to dispose of contaminated purge water, pump lines, bailers, etc? _____

For Domestic Wells - Has as much information on the well and distribution system been obtained, i.e., depth, casing type, diameter, treatment present, etc.? _____

Has the sample been collected prior to treatment and as close to the well head as possible? _____

Has the domestic well been purged sufficiently to reach temperature stabilization? _____

Have the weather conditions been recorded? _____

Decontamination:

Has sampling equipment been decontaminated properly for the given analytes? _____

Have the proper decontamination solutions been used? _____

For large equipment (backhoes, drill rigs), has decontamination taken place in an appropriate area? _____

Has decontaminated water/solution been collected for proper disposal? _____ Where disposed? _____

Safety:

Is the proper level of protective clothing being worn for the tasks? _____ Level A __ B __ C __ D __

Is the site Health and Safety Plan present with proper emergency contacts included? _____

Is monitoring equipment present? _____ OVA _____
H_2, O_2 meter _____ Explosimeter _____ Other _____

FIGURE QAPP 12-2 (cont'd)

Is the vehicle equipped with a First Aid Kit? _____

Is contaminated protective clothing being disposed of properly? _____

Are personnel aware of the contaminants present at the site? _____

General:

Are employees conducting the investigation in a professional manner? _____

Are the objectives of the sampling activities understood by the field personnel? _____

Are weather conditions affecting sample quality? _____

Audit Summary and Comments:

_____

Signed by: _____ Print: _____

_____ _____

_____ _____

Date: _____

The ERM Group

measurements, soil logs, and any problems encountered during sampling.

- Adherence to health and safety guidelines outlined in the Site Health and Safety Plan including wearing of proper protective clothing.

- Adherence to decontamination procedures outlined in Section 6 of this QAPP, including proper decontamination of pumps and pump tubing, bailers, and sampling equipment.

- Adherence to sample collection, preparation, preservation, and storage procedures.

13.0 PREVENTATIVE MAINTENANCE

13.1 Field Equipment Maintenance

ERM's field equipment is maintained through the use of a tracking system incorporating the tagging of each equipment item. This tag identifies its most recent maintenance, battery charge, and condition. When damaged or equipment in need of repair is returned to the equipment warehouse, it is appropriately flagged for the required maintenance to be performed. This process assures only operable and maintained equipment enters the field. Routine daily maintenance procedures conducted in the field will include:

- Removal of surface dirt and debris from exposed surfaces of the sampling equipment and measurement systems.
- Cleansing of filters in the organic vapor analyzer.
- Storage of equipment away from the elements.
- Daily inspections of sampling equipment and measurement systems for possible problems (e.g., cracked or clogged lines or tubing or weak batteries).
- Check instrument calibrations as described in Section 8.0 of this QAPP.
- Charging any battery packs for equipment when not in use.

Spare and replacement parts stored in the field to minimize downtime include, but are not limited to the following:

- Appropriately sized batteries
- Locks
- Extra sample containers and preservatives
- Bailer line
- Additional bailers
- OVA igniters and filters
- Calibration gases, battery charger, and support equipment
- Spare filters for filtration apparatus.
- Extra pH probes, conductivity probes, samples coolers, packing material, and sample location stakes.
- Additional supply of health and safety equipment i.e., respirator cartridges, boots, gloves, tyvek, etc.
- Additional equipment as necessary for the field tasks.

13.1.1 Century OVA 128 Meter

Primary filter-The primary filter will be cleaned after each day of use. This filter is located behind the sample

inlet connector (Fitting Assembly) on the Side Pack Assembly and is removed for cleaning by using a 7/16 inch thin wall socket to unscrew the Fitting Assembly. The filter cup, "O" ring and loading spring will then come out. The porous stainless filter cup will be cleaned by blowing out or heating the filter.

Secondary filter-A particle filter is located in each pick-up fixture. One of these filters must be in the sample line whenever the instrument is in use. The OVA 128 uses a porous metal filter which can be replaced or cleaned.

Mixer/burner Assembly Filter-A porous metal particle filter is incorporated in the Mixer/Burner Assembly which screws into the Preamp Assembly. This filter is used as the sample mixer and inlet flame arrestor in the chamber. This filter does not become contaminated under normal conditions but can be cleaned or the assembly replaced as necessary.

Access to this filter or output surface does not require removing the instrument from the case. For access, remove the safety cover using a hex key wrench (supplied) then unscrew the exhaust port. If necessary, the Filter Assembly will be cleaned with a small wire brush.

Exhaust Flame Arrestor-A porous metal flame arrestor is located in the exhaust port of the detector chamber (Preamp Assembly). It acts as a particle filter on the chamber output and restricts foreign matter from entering the chamber. This filter will be cleaned by removing the exhaust port. This filter is cleaned with a detergent and is completely baked out at 120°F before reinstalling.

Sampling Fixtures-Sampling fixtures will be cleaned with an air hose and/or detergent water to eliminate foreign particle matter.

The following procedures refer to maintenance for the gas chromatographic mode of operation:

Column-Any column can be contaminated with compounds having long retention times. This is evident by the occurrence of high background readings. If these are observed, the contaminated column will be baked at 100°C (212°F) for 3-4 hours in a drying oven while passing nitrogen through the column.

Charcoal Filter Assembly-After repeated use, the Charcoal Filter Assembly will become saturated. Periodically, the operator will check the effectiveness of the activated charcoal.

This will easily be done by operating the unit with the Sample Injection Valve "in" and passing the probe near a concentrated sample of the compound being analyzed. If the rise is more than 2 ppm, the old charcoal will be replaced with new activated charcoal.

The above mentioned filters will be maintained as spare parts. Additionally, the following items will be maintained:

ITEM DESCRIPTION	FOXBORO PART NUMBER
Quad Rings	510496-1
Tubing, .148 in ID .020 wall	12942
Tubing, teflon .120 in ID .030 in wall	12941
Activated charcoal	
"0" Ring for charcoal scrubber	U0118CE
Chart Paper (linear)	

13.1.2 PUF Sampling Equipment

Maintenance will be the same as for the high volume particulate equipment. The spare parts list will include

but are not limited to (Section 13) two (2) extra glass cartridges for the PUF plug and twenty (20) unexposed PUF plugs. Additional PUF plugs will be obtained from Enseco when it is determined that the number is not sufficient to complete the project. Enseco will provide the initial cleaning of the PUF plugs.

13.1.3 Tenax, XAD-2, and CMS Sampling Equipment

A spare pump will be maintained on site at all times. Any extra pumps may be obtained from the manufacturer within 24-hours. Should a malfunction occur, repairs will be accomplished by the factory. The same is true for the mass flow meters. A spare mass flow meter will not be maintained on site but will be available within 24 hours from the factory. Twenty traps each of Tenax, XAD-2, and CMS will initially be on-site at the start of the project. If it becomes apparent that this will not be sufficient to complete the project additional traps will be obtained from Enseco.

13.1.4 HNU Model P1 101 Photoionization Unit

This instrument contains only one moving part, and consumes no gases or reagents. The only routine maintenance procedure is cleaning the light source window. A spare lamp will be maintained on site.

13.1.5 High Volume Particulate Sampling Equipment

Sampler Motor-Motor brushes will be replaced any time a rotameter reading drops below 50 with a clean filter. All brushes must be replaced at least every six months. The hi-vol sampler will be recalibrated before and after the brushes are changed.

Face Plate Gasket-A worn face plate gasket is characterized by the gradually blending of the inter-face between collected particulates and the clean filter border. Any decrease in the original sharpness of this interface indicates the need for a new face plate gasket.

Rotameter-Small particles may become lodged in the air cavity of the rotameter, resulting in erratic behavior of the float. The rotameter will be cleaned and calibrated at any sign of foreign particles or moisture deposits in the air column. The rotameter will be cleaned prior to routine calibration. If any physical damage, such as a crack, is observed the rotameter will be discarded.

Other Maintenance-All other maintenance will be performed as required. All hi-volume samplers shall be calibrated at least once each Quarter. This should be done, if possible, the first week of each Quarter (first week of January, April, July and October). All maintenance and calibration records shall be recorded in a field log book, along with the date the work was performed and the initials of the person performing the work.

Supplies-The following list of parts will be maintained at all times. Additional parts or supplies shall be ordered any time inventory reaches the following values:

Hi-vol brushes - 5 sets
Face plate gaskets - 1
One complete hi-vol motor
Two extra rotameters
25 feet of 3/16-inch I.D. tubing
Five 15-amp fuses
50 filters

13.2 Laboratory Preventative Maintenance

Erco maintains a complete inventory of replacement parts needed for preventive maintenance and spare parts that routinely need replacement (e.g., gauges, detectors, etc.). If an instrument fails, the problem will be diagnosed as quickly as possible and either replacement parts will be ordered or a service call will be placed with the manufacturer. Service contracts are in effect for the data systems controlling operation of GC/MS systems.

Preventive maintenance scheduled for analytical instrumentation are included in Enseco's laboratory manuals and are given in Figures 13-1 through 13-6.

FIGURE QAPP 13-1

PREVENTIVE MAINTENANCE SCHEDULE FROM
ERCO'S TRACE METALS LABORATORY MANUAL (AA)

MAINTENANCE OF PERKIN ELMER 603 ATOMIC ABSORPTION SPECTROPHOTOMETERS, PERKIN ELMER 4000 ATOMIC ABSORPTION SPECTROPHOTOMETER, FISHER MERCURY ANALYZER

I. Nebulizer, Mixing Chamber, and Burner Maintenance

Cleaning of the nebulizer, mixing chamber, and burner system cannot be scheduled; the frequency of cleaning is left to the discretion of the analyst, who should keep the following guidelines in mind.

A) Following aspiration of organic solutions, cleaning should be done in the manner described in Section 10B,3 of the Perkin Elmer 603 instruction manual.

B) The burner and mixing chamber should be cleaned when analyzing for very low levels of an element and the previous sample lot has had the same element as a major constituent (Section 10B,2 and/or 10B,5).

C) Aspiration of high-solid samples (solid $\geq$ 4 percent for 10 cm and 1 percent for 5 cm burner head) requires a minimum of daily cleaning of the burner head. The burner will also require cleaning when discontinuities in the flame occur as a result of dry solids lodged in the burner slot (Section 10B,2).

D) The burner system must be cleaned following the aspiration of high concentrations of Ag, Cu, or Hg to prevent the build-up of explosive acetylide compounds (Section 10B,4).

E) Assuming the nebulizer calibration has not been altered, a decrease in sample uptake indicates a need to clean the nebulizer (Section 10C,1).

II. Drain-Trap and Waste-Vessel Maintenance

A) The waste vessel should be emptied when necessary, but in a manner that will leave enough residual water (5 inches) to submerge the end of the drain tube.

FIGURE QAPP 13-1 (cont'd)

PREVENTIVE MAINTENANCE SCHEDULE FROM
ERCO'S TRACE METALS LABORATORY MANUAL (AA)

B) The drainage system should be inspected each morning before flame ignition in order to prevent flashback explosions. The water trap should consist of a full-to-capacity 6-inch diameter loop. The end of the drain tube should be submerged in a minimum of 5 inches of liquid.

III. Sample Compartment-Window Maintenance

The sample compartment windows should be inspected and cleaned when any dirt is visible. The windows should be cleaned weekly even if they appear to be clean. Ultraviolet absorption losses can occur with optical components, even when visibly clean (Section 10D,4).

IV. Maintenance of Gas Supplies

A) WARNING - Linde-brand acetylene from Union Carbide cannot be used. The acetylene vehicle can dissolve internal plumbing and result in an explosion.

B) Only instrument-grade gases will be used.

C) Compressed air from an ambient air compressor utilizing water and oil traps is preferable to compressed cylinder gas that can suffer from alterations in O_2/N_2 ratio.

D) The nitrous oxide regulator should be monitored for condensation build-up on humid days. If condensation is noticed, a heated regulator or an IR heat lamp should be employed.

FIGURE QAPP 13-2

PREVENTIVE MAINTENANCE SCHEDULE FROM
ERCO'S TRACE METALS LABORATORY MANUAL (ICP)

MAINTENANCE OF THE JARRELL-ASH ATOMCOMP
INDUCTIVELY COUPLED PLASMA SPECTROPHOTOMETER

Timing of maintenance must be determined by the principal operator from instrument performance. Areas to keep under observation for routine cleaning are listed below. The majority of maintenance for this instrument and its associated computer will be nonroutine and logged in the appropriate maintenance log.

1. Mixing chamber, nebulizer, and torch assembly should be inspected and cleaned when necessary (i.e., after high dissolved solids or organic solutions are aspirated).
2. Fore-optic assemblies should be inspected for damage and corrosion.
3. Exhaust system should be checked.
4. Cooling fans and air filters should be checked and cleaned periodically (both for the computer and RF generator).
5. Instrument housing, RF generator housing, and teletype should be wiped with a damp cloth periodically.

FIGURE QAPP 13-3

PREVENTIVE MAINTENANCE SCHEDULE FROM ERCO'S
TRACE METALS LABORATORY MANUAL (Graphite Furnace)

MAINTENANCE OF HEATED GRAPHITE FURNACE (HGA-2100)

A) Plumbing should be checked for possible leaks when cooling water is turned on.

B) Power cables should be hand-tight before power to HGA control unit is turned on.

C) Graphite tubes should be cleaned of any residue (Section 4-1, HGA manual). If the graphite surfaces appear excessively burnt, are cracked, or, if after cleaning unacceptable precision persists, the tube should be replaced.

D) Graphite contact rings should be replaced when excessive wear is noted (Section 4.B).

E) Atomizer windows should be cleaned daily or more frequently if clouding is noted (Section 4.C).

F) Argon flow gas shall be used for all priority pollutant analyses. Proper flow of purge gas should be maintained to prevent excess wear of graphite components.

G) Sample compartment windows should be inspected and cleaned when any dirt is visible. The windows should be cleaned weekly even if they appear clean. Ultra-violet absorption losses can occur even when visibly clean (Section 10D, 4; PE 603 manual).

FIGURE QAPP 13-4

PREVENTIVE MAINTENANCE SCHEDULE FOR GAS CHROMATOGRAPHS
FROM ERCO'S ORGANIC LABORATORY MANUAL

Table B-1. Mainframe Maintenance Schedule

Area or Assembly	Type of Maintenance	Interval
Exterior surface	Cleaning	As conditions indicate
Air filter	Cleaning Replacement	6 months as required

Table B-2. Terminal Systems Maintenance Schedule

Area or Assembly	Type of Maintenance	Interval
Functional terminal		
Paper	Loading	As required
Printhead	Cleaning	As required
Cartridge tape unit		
Tapes	Storage	
Tapes	Conditioning	Every new tape
CTU	Cleaning	One week

Table B-3. Valve System Maintenance Schedule

Area or Assembly	Refer to
Valves	18900C Valve Manual (Part No. 18900-90130)

FIGURE QAPP 13-4 (cont'd)

PREVENTIVE MAINTENANCE SCHEDULE FOR GAS CHROMATOGRAPHS
FROM ERCO'S ORGANIC LABORATORY MANUAL

Table B-4. Flow System Maintenance Schedule

Area or Assembly	Type of Maintenance	Interval
Moisture Trap (between external supply & GC)	Conditioning	2 months, or when gas source is changed
Moisture Trap (between external supply & GC)	Repacking	Every 10 conditionings
Chemical Filter (between flow controller & injection port)	Conditioning	2 months, or when gas source is changed
Carrier Gases	Leak check	As required
Support Gases	Leak check	As required
Air Solenoid	Adjustment	Initial setup or when flow conditions change
H_2/air adjustment	Cleaning	As required
Column	Conditioning	When column is changed, or when chromatographic conditions or samples require
Column	Repacking	As required
Ni Catalyst	Conditioning	6 months, or as required

FIGURE QAPP 13-4 (cont'd)

PREVENTIVE MAINTENANCE SCHEDULE FOR GAS CHROMATOGRAPHS
FROM ERCO'S ORGANIC LABORATORY MANUAL

Table B-5. Injection System Maintenance Schedule

Area or Assembly	Type of Maintenance	Interval
Injection port		
Inj. port	Cleaning	As required
Septum	Replacement	Every 10 manual injections
Inj. port	Leak check	As required
Chem. filter	Conditioning	
Capillary inlet		
Cap. inlet	Leak test	As required
Septum	Replacement	1 week (more frequently when using large needle)
Insert	Cleaning	Whenever insert is changed
Insert seal	Replacement	Whenever leak occurs or damage is apparent
Inlet vent tube	Cleaning	3 months
Column seal	Replacement	Whenever leak occurs or damage is apparent
Autosampler		
Transport	Alignment Resetting stops	As required
Needle	Positioning	On installation, then as required

FIGURE QAPP 13-4 (cont'd)

PREVENTIVE MAINTENANCE SCHEDULE FOR GAS CHROMATOGRAPHS
FROM ERCO'S ORGANIC LABORATORY MANUAL

Table B-6. Detector System Maintenance Schedule

Area or Assembly	Type of Maintenance	Interval
FID/NPD	Leak check	As required
FID	Cleaning	As required
FID/NPD jet	Cleaning	2 months for capillary. As required for packed columns
FID collector	Cleaning	As required
FID/NPD Air/H_2 controls	Cleaning	As required
FID/NPD air solenoid	Adjustment	Initial setup or when flow conditions change
ECD	Freq. check	1 day
ECD	Carrier gas eval.	When carrier gas is changed
	Leak check	When column is changed
ECD	Thermal clean	1 month
ECD	NRC wipe test	6 months

FIGURE QAPP 13-5

S.O.P.
Preventive Maintenance

1. Traps -- Chloromethane response factors are monitored through the use of a Quality Control chart. Trap is replaced when chloromethane response factor falls below 0.3.

2. Chromatographic columns -- Resolution of peaks and column bleed are visually monitored on daily chromatograms. Retention time is monitored on Quality Control charts. When any of these factors causes or indicates a fall in chromatographic performance, the column is replaced.

3. Source cleaning -- BFB tunes are monitored daily to ensure that they meet EPA quality control requirements. When tune criteria cannot be met, the source is cleaned to restore instrument performance.

4. Electron multiplier -- As electron multiplier dynode surfaces wear, instrument sensitivity is reduced. This is compensated for by increasing multiplier gain. When maximum gain no longer provides required sensitivity, the electron multiplier is replaced.

5. Injection port septa -- Septa are changed every 10 injections to ensure against sample loss, carrier gas loss, and pressure drops.

6. Preventive maintenance -- Routine maintenance is performed on vacuum pump oils and GC/MS absorbent trap packings.

FIGURE QAPP 13-6

MAINTENANCE FOR GC/MS SYSTEMS

PREVENTIVE MAINTENANCE CHECKS

ITEM	SERVICE INTERVAL[1]						SERVICE LEVEL
	Dy	Wk	Mo	Qu	Bi	An	
HP 5840A GC							
• Septa		X					Inspect and replace as required.
• Injection port liner		X					Clean or replace.
• Carrier gas filter				X			Replace.
• Capillary column		X			X		Clip or replace as required.
HP 5985 MS							
• Ion gas tube	X						Operate Degas mode for 30 sec.
• Ion source filament				X			Replace as needed.
• Ion source					X		Inspect and clean.
• Electron multiplier					X		Replace as required.
• Teflon seal in direct introduction inlet				X			Replace.
• Vacuum pumps					X		Inspect belt and change oil.
HP 7900 Disc Drive					X		Service contract P.M.
Tektronix hardcopy unit			X				Clean and inspect.
					X		Service contract P.M.
Finnigan 9610 GC							
• Septa		X					Inspect and replace as required.
• Injection port liner		X					Clean or replace.
• Carrier gas filter				X			Replace.
• Capillary column		X			X		Clip or replace as required.
Printronix Printer							
• Interior			X				Clean and inspect.
• Printer ribbon			X				Change as needed.
• Preventive maintenance					X		
Finnigan 4530 MS							
• Quadrupole mass analyzer					X		Clean rod assembly.
• Ion source				X			Clean and inspect.
• Cal gas assembly					X		Inspect.
• Ion source filament				X			Replace as needed.
• Electron multiplier					X		Replace as needed.
• Vacuum pumps				X			Inspect and change oil.
Control Data CMD Disk Drive				X			Service contract preventive maintenance.
• Air filters				X			
• Power supply outputs					X		
• Actuator assembly					X		

[1]Da = daily; We = weekly; Mo = monthly; Qu = quarterly; Bi = biannually; An = annually.

14.0 SPECIFIC ROUTINE PROCEDURES USED TO ASSESS PRECISION, ACCURACY AND COMPLETENESS

14.1 Overall Project Assessment

Overall data quality will be assessed by a thorough understanding of the Project objectives and data quality objectives which are stated during the design phase of the investigation. By maintaining thorough documentation of all decisions made during each phase of sampling, performing field and laboratory audits, thoroughly reviewing (validating) the analytical data as it is generated by the laboratory, and providing appropriate feedback as problems arise in the field or at the laboratory, ERM will closely monitor data accuracy, precision and completeness.

14.2 Field Data Quality Assessment

To assure that all field data are collected accurately and correctly, specific written instructions will be issued to all personnel involved in field data acquisition by the Project Manager. The Project QA/QC Manager will perform field audit(s) during the investigation to document that the appropriate procedures are being followed with respect to sample (and blank) collection. These audits will include a thorough review of the field books used by the Project personnel to insure that all tasks were performed as specified in the instructions. The field audits will necessarily enable the data quality to be assessed with regard to the field operations.

The evaluation (data review) of field blanks, and other field QC samples will provide definitive indications of the

data quality. If a problem arises and can be isolated, corrective actions will be instituted for future field efforts.

14.3 Enseco's Procedures

For this project, the methods to determine precision and accuracy, and their acceptability is well defined in the EPA CLP contract. All other parameters requested will be subject to a method validation study and precision, accuracy, and completeness will be based on the results of this study. In general, for all non-routine parameters, accuracy is calculated through percentage recovery, and precision expressed in percentage difference or percentage relative standard deviation. Acceptance limits are $\pm$ 3 SD from the mean.

14.3.1 Precision

Precision is frequently determined by the comparison of duplicates, where duplicates result from an original sample that has been split for identical analyses. Standard deviation(s) of a sample is commonly used in estimating precision.

$$\text{Sample standard deviation(s)} = \sqrt{\frac{1}{n-1} \sum_{i=1}^{n} (x_i - \bar{x})^2}$$

where a quantity x (e.g., a concentration) is measured n times with a mean x.

The relative standard deviation, RSD (or sample coefficient of variation, CV), which expresses standard deviation as a percentage of the mean, is generally useful in the comparison of three or more duplicates.

$$RSD = 100\ (s/x)$$

or

$$CV = 100\ (s/\bar{x})$$

where RSD = relative standard deviation, or
CV = coefficient of variation
s = standard deviation
$\bar{x}$ = mean

In the case of duplicates -- samples that result when an original sample has been split into two for identical analyses -- the percent difference (%D) between the two samples may be used to estimate precision.

$$\%D = \frac{D_1 - D_2}{(D_1 + D_2)/2} \times 100$$

where: %D = percent difference
D_1 = first sample value
D_2 = second sample value (duplicate)

14.3.2 Accuracy

The determination of accuracy of a measurement requires a knowledge of the true or accepted value for the analyte being measured. Accuracy may be calculated in terms of bias as follows:

$$Bias = \bar{X} - T$$

$$\%Bias = 100(X - T)/T$$

where: $\bar{X}$ = average observed value of measurement

T = "true" value

Accuracy may also be calculated in terms of the recovery of spiked samples:

$$\%\text{Recovery} = 100 \frac{X}{T}$$

14.3.3 Analytical Completeness

Determining whether a data base is complete or incomplete may be quite difficult. To be considered complete, the data set must contain all QC check analyses verifying precision and accuracy for the analytical protocol. Less obvious is whether the data are sufficient to achieve the goals of the project. All data are reviewed in terms of goals in order to determine if the data base is sufficient.

Where possible, the percent completeness for each set of samples is calculated as follows:

$$\%\ \text{completeness} = \frac{\text{valid data obtained}}{\text{total data needed}} \times 100$$

14.3.4 Objectives

Objectives in terms of completeness, precision, and accuracy can be found in Section 5.

14.3.5 Reporting

Precision and accuracy data will be calculated at the time of the analysis from the laboratory perspective. If the control limits have been exceeded, the sample will be reanalyzed according to CLP reanalysis protocols. All

laboratory precision and accuracy results (standards: %RSD, %D; samples: surrogate recoveries, matrix spike recoveries, RPD's on duplicates) will be presented in tabular form in the report.

14.4 ERM's Laboratory Data Assessment

14.4.1 ERM Data Validation

All analytical data generated during the investigation will undergo a rigorous ERM data review. This review will be performed in accordance with the "Functional Guidelines for the Evaluation of Organic and Inorganic Analysis." (U.S. EPA 1988) and a draft EPA document for the "Evaluation of Dioxin and Furan Analysis."

A preliminary review will be performed to verify all necessary paperwork (chain-of-custodies, analytical reports, laboratory personnel signatures) and deliverables (as stated in EPA CLP SOW 8/87) for both organic and inorganic analyses are present.

A detailed quality assurance review will be performed by the Project QA/QC Manager to verify the qualitative and quantitative reliability of the data as it is presented. This review will include a detailed review and interpretation of all data generated by Enseco and CAL Analytical. The primary tools which will be used by experienced data review chemists are to be guidance documents, established (contractual) criteria, and professional judgment. Table 14-1 presents the items examined during the quality assurance review.

Based upon the review of the analytical data, an organic and inorganic quality assurance report will be prepared which

QAPP TABLE 14-1

ITEMS REVIEWED DURING THE ERM DATA VALIDATION

Areas Examined	Applicability (organic, inorganic, both)
ERM and Laboratory Chain of Custodies (Field Notes, Etc)	Both
Laboratory Narrative and QC Summaries	
Holding Times	Both
Extraction/Digestion Logs	Both
Blanks-field and laboratory (accuracy)	Both
Instrument Tune	Organic
Standards	Both
Linearity	Both
Sensitivity/Stability	Both
Selectivity/Specificity	Both
EPA Criteria (SPCC & LCS)	Both
Variability of Technique (internal standards)	Organic
Analyte Breakdown	Organic
Analytical Sequence	Organic
ICP Interference	Inorganic
Control Standards	Inorganic
Serial Dilutions	**Inorganic**
Samples	
Detection Limits	Both
Instrument Printouts	Both
ICP data	Inorganic
AA data	Inorganic
GC data	Organic
GG/MS data	Organic
Autoanalyzer data	Inorganic
Qualitative Identification	Both
Mass spectra	
Pesticide/PCB results	
Tentatively identified compounds	
Quantitative Reliability	Both
Calculations/Equations	Both
Matrix spikes (accuracy)	Both
Bias	
Matrix spike duplicates	Organic
Bias	
Accuracy & Precision	
Surrogate Spikes	Organic
Bias	
Duplicates (field and laboratory)	Both
Precision	
Representativeness	
Post-Digestion Spikes (GFAA)	Inorganic
Matrix Effects	

will state in a technical yet "user friendly" fashion the qualitative and quantitative reliability of the analytical data. The report will consist of a general introduction section, followed by qualifying statements that should be taken into consideration such that the analytical results can be best utilized. Based upon the quality assurance review, qualifier codes will be placed next to specific sample results on the sample data tables. These defined qualifier codes will serve as an indication of the qualitative and quantitative reliability.

During the course of the data review, an organic and inorganic support documentation package will be prepared which will provide backup information that will accompany all qualifying statements presented in the quality assurance review.

Once the review has been completed, the Project QA/QC Manager will submit these data to the Project Manager. These approved data tables and quality assurance reviews will be signed and dated by the Project QA/QC Manager.

After the analytical data has been reviewed by ERM's Project QA/QC Manager, the data package will be forwarded to U.S. EPA Region 5 as part of U.S. EPA's review process.

14.5 Management Data Quality Assessment

The analytical data generated from the investigation are validated, qualified and submitted to the Project Manager. The quality of the data will be assessed from an overall management perspective by an evaluation of the analytical results with respect to the project and data quality objectives. Information that can be obtained includes determination of the adequacy of current data points and the

identification of missing data points. By examination of the collective data at the "back-end" of the process, data quality will be assessed with respect to all of the PSARCC objectives.

15.0 CORRECTIVE ACTION

15.1 ERM's Corrective Action

One of the critical roles in a QA/QC Program is the implementation of corrective actions in the event that problems arise. During any large investigation, many large or small problems may arise. The Project QA/QC Manager will notify the Project Manager who is ultimately responsible for taking appropriate corrective action. The U.S. EPA and OEPA Project Coordinators will be informed of the problem and the proposed course of action to be taken if it is not determined to be acceptable to them.

To assess the quality of the samples collected in the field and to determine if there is a need for corrective action, field procedures will be evaluated through various audits. Some of the areas examined in the audit processes are summarized below.

- Sampling procedures are in accordance with the SSSP and QAPP
- Indications of faulty sampling equipment or inappropriate bottleware and preservatives
- Fully completed field data sheets for all activities
- Strict chain-of-custody on all supplies collected

Any such problems identified during the investigation will be reported on a Corrective Action Form as presented in Figure 15-1. The aforementioned examples are tests for soundness that will be checked during data evaluation and

FIGURE QAPP 15-1

Corrective Action Form

Date: _____

Job Name: _____

Initiator's Name and Title: _____

Problem Description: _____

_____

_____

_____

_____

_____

_____

_____

_____

_____

_____

Reported To: _____

Corrective Action: _____

_____

_____

_____

_____

_____

_____

_____

Reviewed and Implemented By: _____

cc: Project Manager - _____
QA Manager - _____
Qa Officer - _____

interpretation. Any errors or problems will be corrected by an appropriate action which may include:

- Replacing or repairing a faulty measurement system
- Discarding erroneous data
- Collecting new data
- Accepting the data and acknowledging a level of uncertainty

15.2 Enseco's Corrective Action

Corrective actions for laboratory problems are specified in Enseco laboratory manuals and standard operating procedures. Specific QC procedures are designed to help analysts determine the need for corrective action. Often, personal experience is most valuable in alerting the analyst to suspicious data or malfunctioning equipment. Corrective action taken at this point helps to avoid collection of poor quality data.

Problems not immediately detected during the course of analysis may require more formalized, long-term corrective action. The essential steps in the corrective action system are:

1. Identify and define the problem.
2. Assign responsibility for investigating the problem.
3. Investigate and determine the cause of the problem.

4. Determine a corrective action to eliminate the problem.

5. Assign and accept responsibility for implementing the corrective action.

6. Establish effectiveness of the corrective action and implement it.

7. Verify that the corrective action has eliminated the problem.

This scheme is generally accomplished through the use of the Corrective Action Request Form (Figure 15-2) which is available to all Enseco personnel. Using this form, any laboratory analyst or project member may notify the QA Director of a problem. The QA Director initiates the corrective action scheme by relating the problem to the appropriate laboratory managers and/or project managers who investigate or assign responsibility for investigating the problem and its cause. Once determined, an appropriate corrective action is approved by the QA Director. Its implementation is later verified through a laboratory audit.

Information contained on a Corrective Action form is kept confidential within Enseco and is generally limited to the individuals involved. Severe problems and difficulties may warrant special reports to the Corporate Vice President of QA who will ensure that the appropriate corrective actions are taken.

Close scrutiny is paid to the quality and validity of the analytical data for any given analysis. Variations of more than two standard deviations (unless stricter controls are stipulated in the method used) will warrant corrective

FIGURE QAPP 15-2

ENSECO Incorporated

ERCO

QUALITY ASSURANCE CORRECTIVE ACTION REQUEST

Originator: _____ Date: _____

Laboratory: _____ Project: _____

Problem: _____

Action Planned: Implemented:

QA Director: _____ Date: _____

action procedures. The nature of such will be determined by the method employed. In most instances, a reanalysis (and possibly a recalibration) may be necessary to correct the problem.

16.0 QUALITY ASSURANCE REPORTS TO MANAGEMENT

16.1 ERM's Quality Assurance Reports

A quality assurance report will be prepared at the end of each pertinent task effort by the Project Manager in conjunction with the Project QA/QC Manager. This report will be forwarded to the appropriate project coordinators. The report will provide information on the performance of measurement systems and data quality and will include the following information:

- The status and coverage of various laboratory and field quality assurance project activities
- Data quality assurance reviews including assessment of: accuracy, precision, completeness, representativeness, and comparability
- Significant quality assurance problems discovered, corrective actions taken, progress and improvements, plans, and recommendations for further implementation or updating of the investigative QAPP
- Any significant field observations noted in the field notebook during the sampling procedure
- A summarization of any performance and/or system audits that took place

16.2 Enseco's Quality Assurance Reports

Enseco's QA Director is required to submit monthly QA reports to the President of ERCO and to the Corporate Vice President of QA. The reports will include detailed information on the following items:

1. Systems audit reports

2. Scores of and comments on performance evaluation samples

3. Number of quality control samples performed and results of such samples

4. Results of on-site visits and audits by regulatory agents and other clients

5. Status of major contracts, projects, and certifications

6. Problems and corrective actions

7. General comments

The Corporate Vice President of Enseco is required to submit monthly reports to each member of the Executive Committee, the Chairman of the Board of Directors, and each Divisional President. These reports should include a summary of the reports submitted by the laboratory QA Directors, reports of the Corporate Vice President's systems audits and site inspections at each laboratory, results of performance evaluation samples that are sent to the laboratories, recommendations, and comments.

In case of severe problems and difficulties, special reports will be prepared and submitted to the project management in a timely manner.

Volume 2: Attachments
Ruetgers-Nease Salem, Ohio Site

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Submitted to

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ATTACHMENT #1

Enseco's SOP for the Analysis of TCL, VOA, BNA and Pesticide/PCB's in Fish Tissue

[THIS DOCUMENT IS CONFIDENTIAL AND IS ON FILE WITH BOTH THE OHIO EPA AND THE U.S. EPA]

ATTACHMENT #2

Enseco's SOP for the Analysis of TCL, VOA, BNA and Pesticide/PCB's with Low Detection Limits in Drinking Water

[THIS DOCUMENT IS CONFIDENTIAL AND IS ON FILE WITH BOTH THE OHIO EPA AND THE U.S. EPA]

11. Sample Analysis

11.1 Sample Purging

11.1.1 Prior to analysis all samples are purged at room temperature with pure, dry air or nitrogen to remove water vapor. Purging is accomplished as described in 9.7 except that the gas flow is in the same direction as sample flow (i.e. marked end of cartridge is connected to the flow system).

11.1.2 The sample is purged at 500 mL/minute for 5 minutes. After purging the endcaps are immediately replaced. The cartridges are returned to the metal can or analyzed immediately.

11.1.3 If very humid air is being sampled the purge time may be increased to more efficiently remove water vapor. However, the sum of sample volume and purge volume must be less than 75% of the retention volume for the most volatile component of interest.

11.2 GC/MS Setup

11.2.1 Considerable variation from one laboratory to another is expected in terms of instrument configuration. Therefore, each laboratory must be responsible for verifying that their particular system yields satisfactory results. Section 14 discusses specific performance criteria which should be met.

11.2.2 A block diagram of the analytical system required for analysis of CMS cartridges is depicted in Figure 3. The thermal desorption system must be designed to accommodate the particular cartridge configuration. For the CMS cartridge design shown in Figure 1, the cartridge heating is accomplished as described in 9.8. The use of a desorption oven, in conjunction with a

simplier cartridge design is also acceptable. Exposure of the sample to metal surfaces should be minimized and only stainless steel or nickel should be employed. The volume of tubing leading from the cartridge to the GC column must be minimized and all areas must be well-swept by helium carrier gas.

11.2.3 The GC column oven must be capable of being cooled to -70°C and subsequently temperature programmed to 150°C.

11.2.4 The specific GC column and temperature program employed will be dependent on the compounds of interest. Appropriate conditions are described in the literature (2). In general, a nonpolar stationary phase (e.g. SE-30, OV-1) temperature programmed from -70 to 150°C at 8°/minute will be suitable. Fused silica, bonded-phase columns are preferable to glass columns since they are more rugged and can be inserted directly into the MS ion source, thereby eliminating the need for a GC/MS transfer line. Fused silica columns are also more readily connected to the GC injection valve (Figure 3). A drawback of fused silica, bonded-phase columns is the lower capacity compared to coated, glass capillary columns. In most cases the column capacity will be less than 1 microgram injected for fused silica columns.

11.2.5 Capillary column dimensions of 0.3 mm ID and 50 meters long are generally appropriate although shorter lengths may be sufficient in many cases.

11.2.6 Prior to instrument calibration or sample analysis the GC/MS system is assembled as shown in Figure 3. Helium purge flow (through the cartridge) and carrier flow are set at approximately 50 mL/minute and 2-3 mL/minute respectively. When a cartridge is not in place a union is placed in the helium purge line to ensure a continuous inert gas flow through the injection loop.

11.2.7 Once the column and other system components are assembled and the various flows established the column temperature is increased to 250°C for approximately four hours (or overnight if desired) to condition the column.

11.2.8 The MS and data system are set up according to the manufacturer's instructions. Electron impact ionization (70eV) and an electron multiplier gain of approximately 5×10^4 should be employed. Once the entire GC/MS system has been setup the system is calibrated as described in Section 11.3. The user should prepare a detailed standard operating procedure (SOP) describing this process for the particular instrument being used.

11.3 GC/MS Calibration

11.3.1 Tuning and mass standardization of the MS system is performed according to manufacturer's instructions and relevant user prepared SOPs. Bromofluorobenzene (BFB) will be employed for this purpose. The material is introduced directly into the ion source through a molecular leak. The instrumental parameters (e.g., lens voltages, resolution, etc.) should be adjusted to give the relative ion abundances shown in Table 2, as well as acceptable resolution and peak shape. If these approximate relative abundances cannot be achieved, the ion source may require cleaning according to manufacturer's instructions. In the event that the user's instrument cannot achieve these relative ion abundances, but is otherwise operating properly, the user may adopt another set of relative abundances as performance criteria. However, these alternate values must be repeatable on a day-to-day basis.

11.3.2 After the mass standardization and tuning process has been completed and the appropriate values entered into the data system, the user should then calibrate the entire GC/MS system by introducing known quantities of the components of interest into the system. Three alternate procedures may be employed for the calibration process including 1) direct injection of dilute vapor phase standards, prepared in a dilution bottle or compressed gas cylinder, onto the GC column, 2) injection of dilute vapor phase standards into a flowing inert gas stream directed onto a CMS cartridge, and 3) introduction of permeation or diffusion tube standards onto a CMS cartridge. Direct injection of a compressed gas cylinder (aluminum) standard containing trace levels of the compounds of interest has been found to be the most convenient practice since such standards are stable over a several month period. The standards preparation processes for the various approaches are described in Section 13. The following paragraphs describe the instrument calibration process for these approaches.

11.3.3 If the system is to be calibrated by direct injection of a vapor phase standard, the standard, in either a compressed gas cylinder or dilution flask, is obtained as described in Section 13. The MS and data system are setup for acquisition, but the ionizer filament is shut off. The GC column oven is cooled to -70°C, the injection valve is placed in the <u>load</u> mode, and the cryogenic loop is immersed in liquid nitrogen or liquid argon. Liquid argon is required for standards prepared in nitrogen or air, but not for standards prepared in helium. A known volume of the standard (10-1000 mL) is injected through the cryogenic loop at a rate of 10-100 mL/minute.

11.3.4 Immediately after loading the vapor phase standard, the injection valve is placed in the inject mode, the GC program and system clock are started, and the cryogenic loop is heated to 60°C by applying voltage (15-20 volts) to the thermocouple wire heater surrounding the loop. The voltage is adjusted to maintain a loop temperature of 60°C. An automatic temperature controller can be used in place of the manual control system. After elution of unretained components (~3 minutes after injection) the ionizer filament is turned on and data acquisition is initiated. The helium purge line (set at 50 mL/minute) is connected to the injection valve and the valve is returned to the load mode. The loop temperature is increased to 150 °C, with helium purge, and held at this temperature until the next sample is to be loaded.

11.3.5 After the last component of interest has eluted, acquisition is terminated and the data is processed as described in Section 11.3.8. The standard injection process is repeated using different standard concentrations and/or volumes to cover the analytical range of interest.

11.3.6 If the system is to be calibrated by analysis of standard CMS cartridges, a series of cartridges is prepared as described in Sections 13.2 or 13.3. Prior to analysis the cartridges are stored (no longer than 48 hours) as described in Section 9.10. For analysis the injection valve is placed in the load mode and the cryogenic loop is immersed in liquid nitrogen (or liquid argon if desired). The CMS cartridge is installed in the helium purge line (set at 50 mL/minute) so that the helium flow through the cartridge is opposite to the direction of sample flow and the purge gas is directed through the cryogenic loop and vented to the

atmosphere. The CMS cartridge is heated to 370-400°C and maintained at this temperature for 10 minutes (using the temperature control process described in Section 9.8). During the desorption period, the GC column oven is cooled to -70°C and the MS and data system are setup for acquisition, but the ionizer filament is turned off.

11.3.7 At the end of the 10 minute desorption period, the analytical process described in Sections 11.3.4 and 11.3.5 is conducted. During the GC/MS analysis heating of the CMS cartridge is discontinued. Helium flow is maintained through the CMS cartridge and cryogenic loop until the cartridge has cooled to room temperature. At that time, the cryogenic loop is allowed to cool to room temperature and the system is ready for further cartridge analysis. Helium flow is maintained through the cryogenic loop at all times, except during the installation or removal of a CMS cartridge, to minimize contamination of the loop.

11.3.8 Data processing for instrument calibration involves determining retention times, and integrated characteristic ion intensities for each of the compounds of interest. In addition, for at least one chromatographic run, the individual mass spectra should be inspected and compared to reference spectra to ensure proper instrumental performance. Since the steps involved in data processing are highly instrument specific, the user should prepare a SOP describing the process for individual use. Overall performance criteria for instrument calibration are provided in Section 14. If these criteria are not achieved, the user should refine the instrumental parameters and/or operating procedures to meet these criteria.

11.4 Sample Analysis

11.4.1 The sample analysis is identical to that described in Sections 11.3.6 and 11.3.7 for the analysis of standard CMS cartridges.

11.4.2 Data processing for sample data generally involves 1) qualitatively determining the presence or absence of each component of interest on the basis of a set of characteristic ions and the retention time using a reversed-search software routine, 2) quantification of each identified component by integrating the intensity of a characteristic ion and comparing the value to that of the calibration standard, and 3) tentative identification of other components observed using a forward (library) search software routine. As for other user specific processes, a SOP should be prepared describing the specific operations for each individual laboratory.

12. Calculations

12.1 Calibration Response Factors

12.1.1 Data from calibration standards is used to calculate a response factor for each component of interest. Ideally the process involves analysis of at least three calibration levels of each component during a given day and determination of the response factor (area/nanogram injected) from the linear least squares fit of a plot of nanograms injected versus area (for the characteristic ion). In general, quantities of components greater than 1,000 nanograms should not be injected because of column overloading and/or MS response nonlinearity.

12.1.2 In practice the daily routine may not always allow analysis of three such calibration standards. In this situation calibration data from consecutive days may be pooled to yield a response factor, provided that analysis of replicate standards of the same concentration are shown to agree within 20% on the consecutive days. In all cases one given standard

concentration, near the midpoint of the analytical range of interest, should be injected at least once each day to determine day-to-day precision of response factors.

12.1.3 Since substantial nonlinearity may be present in the calibration curve, a nonlinear least squares fit (e.g. quadratic) should be employed. This process involves fitting the data to the following equation:

$$Y = A + BX + CX^2$$

where

Y = peak area
X = quantity of component injected nanograms
A, B, and C are coefficients in the equation.

12.2 Analyte Concentrations

12.2.1 Analyte quantities on a sample cartridge are calculated from the following equation:

$$Y_A = A + BX_A + CX^2$$

where Y_A is the area of the analyte characteristic ion for the sample cartridge.
X_A is the calculated quantity of analyte on the sample cartridge, in nanograms.
A, B, and C are the coefficients calculated from the calibration curve described in Section 12.1.3.

12.2.2 If instrumental response is essentially linear over the concentration range of interest, a linear equation (C=0 in the equation above) can be employed.

12.2.3 Concentration of analyte in the original air sample is calculated from the following equation:

$$C_A = \frac{X_A}{V_s}$$

where

C_A is the calculated concentration of analyte in ng/L.

V_s and X_A are as previously defined in Section 10.2.11 and 12.2.1, respectively.

13. Standard Preparation

13.1 Standards for Direct Injection

13.1.1 Standards for direct injection can be prepared in compressed gas cylinders or in dilution vessels. The dilution flask protocol has been described in detail in another method and is not repeated here (6). For the CMS method where only volatile compounds (boiling point <120°C) are of concern, the preparation of dilute standards in 15 liter aluminum compressed gas cylinders has been found to be most convenient. These standards are generally stable over at least a 3-4 month period and in some cases can be purchased from commercial suppliers on a custom prepared basis.

13.1.2 Preparation of compressed gas cylinders requires working with high pressure tubing and fittings, thus requiring a user prepared SOP which ensures that adequate safety precautions are taken. Basically, the preparation process involves injecting a predetermined amount of neat liquid or gas into an empty high pressure cylinder of known volume, using gas flow into the cylinder to complete the transfer.

The cylinder is then pressurized to a given value (500-1000 psi). The final cylinder pressure must be determined using a high precision gauge after the cylinder has thermally equilibrated for a 1-2 hour period after filling.

13.1.2 The concentration of components in the cylinder standard should be determined by comparison with National Bureau of Standards reference standards (e.g. SRM 1805-benzene in nitrogen) when available.

13.1.3 The theoretical concentration (at 25°C and 760 mm pressure) for preparation of cylinder standards can be calculated using the following equation:

$$C_T = \frac{V_I \times d}{V_c} \times \frac{14.7}{P_c + 14.7} \times 24.4 \times 1000$$

where C_T is the component concentration, in ng/mL at 25°C and 760 mm Hg pressure.

V_I is the volume of neat liquid component injected, in µL.

V_c is the internal volume of the cylinder, in L.

d is the density of the neat liquid component, in g/mL.

P_c is the final pressure of the cylinder standards, in pounds per square inch gauge (psig).

13.2 Preparation of Spiked Traps by Vapor Phase Injection

This process involves preparation of a dilution flask or compressed gas cylinder containing the desired concentrations of the compound(s) of interest and injecting the desired volume of vapor into a flowing gas stream which is directed onto a clean CMS cartridge. The procedure is described in detail in another method within the Compendium (6) and will not be repeated here.

13.3 Preparation of Spiked Traps Using Permeation or Diffusion Tubes

13.3.1 A flowing stream of inert gas containing known amounts of each compound of interest is generated according to ASTM Method D3609 (4). Note that a method of accurately maintaining temperature within ± 0.1°C is required and the system generally must be equilibrated for at least 48 hours before use.

13.3.2 An accurately known volume of the standard gas stream (usually 0.1-1 liter) is drawn through a clean CMS cartridge using the sampling system described in Section 10.2.1, or a similar system. However, if mass flow controllers are employed, they must be calibrated for the carrier gas used in Section 13.3.1 (usually nitrogen). Use of air as the carrier gas for permeation systems is not recommended, unless the compounds of interest are known to be highly stable in air.

13.3.3 The spiked traps are then stored or immediately analyzed as in Sections 11.3.6 and 11.3.7.

14. Performance Criteria and Quality Assurance

This section summarizes the quality assurance (QA) measures and provides guidance concerning performance criteria which should be achieved within each laboratory. In many cases the specific QA procedures have been described within the appropriate section describing the particular activity (e.g. parallel sampling).

14.1 Standard Operating Procedures (SOPs)

14.1.1 Each user should generate SOPs describing the following activities as accomplished in their laboratory: 1) assembly, calibration and operation of the sampling system, 2) preparation, handling and storage of CMS cartridges, 3) assembly and operation of GC/MS system including the thermal desorption apparatus and data system, and 4) all aspects of data recording and processing.

14.1.2 SOPs should provide specific stepwise instructions and should be readily available to, and understood by the laboratory personnel conducting the work.

14.2 CMS Cartridge Preparation

14.2.1 Each batch of CMS cartridges, prepared as described in Section 9, should be checked for contamination by analyzing one cartridge, immediately after preparation. While analysis can be accomplished by GC/MS, many laboratories may chose to use GC/FID due to logistical and cost considerations.

14.2.2 Analysis by GC/FID is accomplished as described for GC/MS (Section 11) except for use of FID detection.

14.2.3 While acceptance criteria can vary depending on the components of interest, at a minimum the clean cartridge should be demonstrated to contain less than one-fourth of the minimum level of interest for each component. For most compounds the blank level should be less than 10 nanograms per cartridge in order to be acceptable. More rigid criteria may be adopted, if necessary, within a specific laboratory. If a cartridge does not meet these acceptance criteria, the entire lot should be rejected.

14.3 Sample Collection

14.3.1 During each sampling event at least one clean cartridge will accompany the samples to the field and back to the laboratory, having been placed in the sampler but without sampling air, to serve as a field blank. The average amount of material found on the field blank cartridges may be subtracted from the amount found on the actual samples. However, if the blank level is greater than

25% of the sample amount, data for that component must be identified as suspect.

14.3.2 During each sampling event at least one set of parallel samples (two or more samples collected simultaneously) should be collected, preferably at different flow rates as described in Section 10.1.4. If agreement between parallel samples is not generally within ±25% the user should collect parallel samples on a much more frequent basis (perhaps for all sampling points). If a trend of lower apparent concentrations with increasing flow rate is observed for a set of parallel samples one should consider using a reduced sampling rate and longer sampling interval, if possible. If this practice does not improve the reproducibility further evaluation of the method performance for the compound of interest might be required.

14.3.3 Backup cartridges (two cartridges in series) should be collected with each sampling event. Backup cartridges should contain less than 10% of the amount of components of interest found in the front cartridges, or be equivalent to the blank cartridge level, whichever is greater.

14.4 GC/MS Analysis

14.4.1 Performance criteria for MS tuning and mass standardization have been discussed in Section 11.2 and Table 2. Additional criteria can be used by the laboratory, if desired. The following sections provide performance guidance and suggested criteria for determining the acceptability of the GC/MS system.

14.4.2 Chromatographic efficiency should be evaluated daily by the injection of calibration standards. A reference compound(s) should be chosen from the calibration standard and plotted on an expanded time scale so that its width at 10% of the peak height can be calculated, as shown in Figure 6. The width of the peak at 10% height should not exceed 10 seconds. More stringent criteria may be required for certain applications. The asymmetry factor (see Figure 6) should be between 0.8 and 2.0. The user should also evaluate chromatographic performance for any polar or reactive compounds of interest, using the process described above. If peaks are observed that exceed the peak width or asymmetry factor criteria above, one should inspect the entire system to determine if unswept zones or cold spots are present in any of the fittings or tubing and/or if replacement of the GC column is required. Some laboratories may chose to evaluate column performance separately by direct injection of a test mixture onto the GC column. Suitable schemes for column evaluation have been reported in the literature (7).

14.4.3 The detection limit for each component is calculated from the data obtained for calibration standards. The detection limit is defined as

$$DL = A + 3.3S$$

where

DL is the calculated detection limit in nanograms injected.

A is the intercept calculated in Section 12.1.3.

S is the standard deviation of replicate determinations of the lowest level standard (at least three such determinations are required). The lowest

level standard should yield a signal to noise ratio (from the total ion current response) of approximately 5.

14.4.4 The relative standard deviation for replicate analyses of cartridges spiked at approximately 10 times the detection limit should be 20% or less. Day to day relative standard deviation for replicate cartridges should be 25% or less.

14.4.5 A useful performance evaluation step is the use of an internal standard to track system performance. This is accomplished by spiking each cartridge, including blank, sample, and calibration cartridges with approximately 100 nanograms of a compound not generally present is ambient air (e.g. perfluorotoluene). Spiking is readily accomplished using the procedure outlined in Section 13.2, using a compressed gas standard. The integrated ion intensity for this compound helps to identify problems with a specific sample. In general the user should calculate the standard deviation of the internal standard response for a given set of samples analyzed under identical tuning and calibration conditions. Any sample giving a value greater than $\pm$ 2 standard deviations from the mean (calculated excluding that particular sample) should be identified as suspect. Any marked change in internal standard response may indicate a need for instrument recalibration.

14.5 Method Precision and Recovery

14.5.1 Recovery and precision data for selected volatile organic compounds are presented in Table 1. These data were obtained using ambient air, spiked with known amounts of the compounds in a dynamic mixing system (2).

14.5.2 The data in Table 1 indicate that in general recoveries better than 75% and precision (relative standard deviations) of 15-20% can be obtained. However, selected compounds (e.g. carbon tetrachloride and

benzene) will have poorer precision and/or recovery. The user must check recovery and precision for any compounds for which quantitative data are needed.

References

1. Kebbekus, B. B. and J. W. Bozzelli. Collection and Analysis of Selected Volatile Organic Compounds in Ambient Air. Proceedings of Air Pollution Control Association, Paper No. 82-65.2, Air Pollution Control Association, Pittsburgh, Pennsylvania, 1982.

2. Riggin R. M. and L. E. Slivon. Determination of Volatile Organic Compounds in Ambient Air Using Carbon Molecular Sieve Adsorbants, Special Report on Contract 68-02-3745 (WA-7), U.S. Environmental Protection Agency, Research Triangle Park, North Carolina, September, 1983.

3. Riggin, R. M., "Technical Assistance Document for Sampling and Analysis of Toxic Organic Compounds in Ambient Air", EPA-600/4-83-027, U.S. Environmental Protection Agency, Research Triangle Park, North Carolina, 1983.

4. Annual Book of ASTM Standards, Part 11.03, "Atmospheric Analysis: Occupational Health and Safety", American Society for Testing and Materials, 1983.

5. Walling, J. F., Berkley, R. E., Swanson, D. H., and Toth, F. J. "Sampling Air for Gaseous Organic Chemical-Applications to Tenax", EPA-600/7-54-82-059, U.S. Environmental Protection Agency, Research Triangle Park, North Carolina, 1982.

6. This Methods Compendium - Tenax Method (TO 1).

7. Grob, K., Jr., Grob, G., and Grob, K., "Comprehensive Standardized Quality Test for Glass Capillary Columns", J. Chromatog., 156 1-20, 1978.

TABLE 1. VOLATILE ORGANIC COMPOUNDS FOR WHICH THE CMS ADSORPTION METHOD HAS BEEN EVALUATED

Compound	Retention Time, Minutes[a]	Characteristic Mass Fragment Used For Quantification	Method Performance Data[b]		
			Concentration, ng/L	Percent Recovery	Standard Deviation
Vinyl Chloride	6.3	62	17	74	19
Acrylonitrile	10.8	53	20	85	18
Vinylidene Chloride	10.9	96	36	94	19
Methylene Chloride	11.3	84	28	93	16
Allyl Chloride	11.4	76	32	72	19
Chloroform	13.8	83	89	91	12
1,2-Dichloroethane	14.5	62	37	85	11
1,1,1-Trichloroethane	14.7	97	100	75	9.1
Benzene	15.4	78	15	140	37
Carbon Tetrachloride	15.5	117	86	55	2.9
Toluene	18.0	91	4.1	98	5.4

a) GC conditions as follows:

Column - Hewlett Packard, crosslinked methyl silicone, 0.32 mm ID x 50 mm long, thick film, fused silica.

Temperature Program - 70°C for 2 minutes then increased at 8°C/minute to 120°C.

b) From Reference 2. For spiked ambient air.

TABLE 2. SUGGESTED PERFORMANCE CRITERIA FOR RELATIVE ION ABUNDANCES FROM FC-43 MASS CALIBRATION

M/E	% Relative Abundance
51	1.8 ± 0.5
69	100
100	12.0 ± 1.5
119	12.0 ± 1.5
131	35.0 ± 3.5
169	3.0 ± 0.4
219	24.0 ± 2.5
264	3.7 ± 0.4
314	0.25 ± 0.1

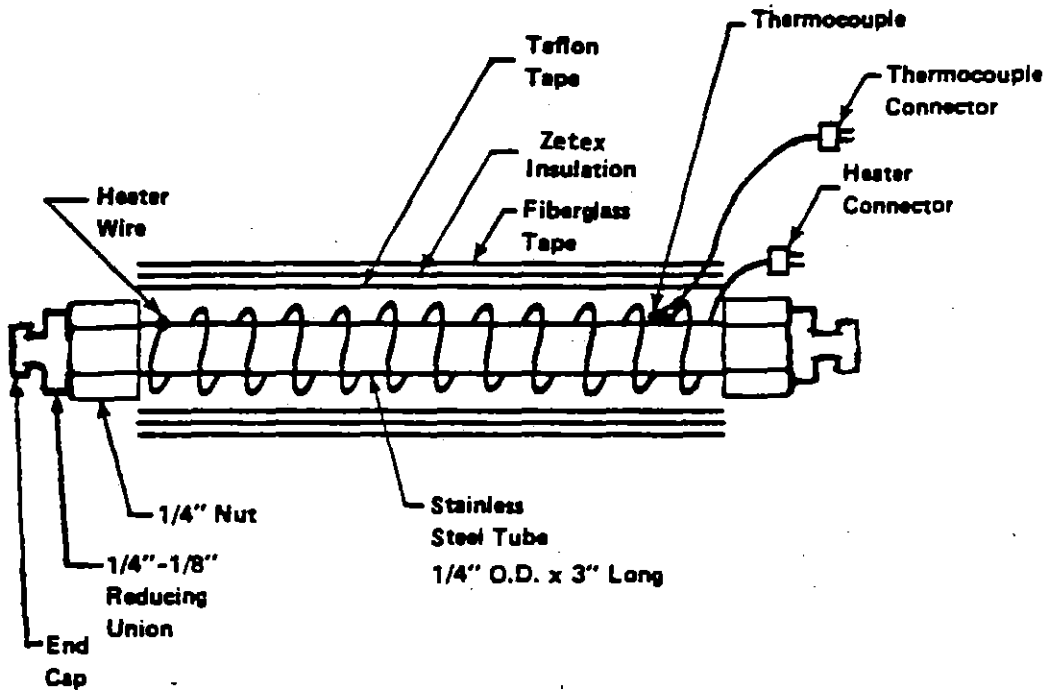
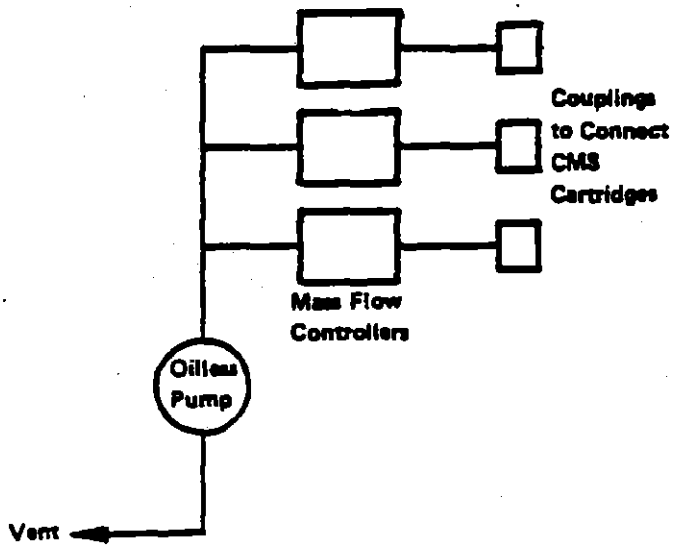
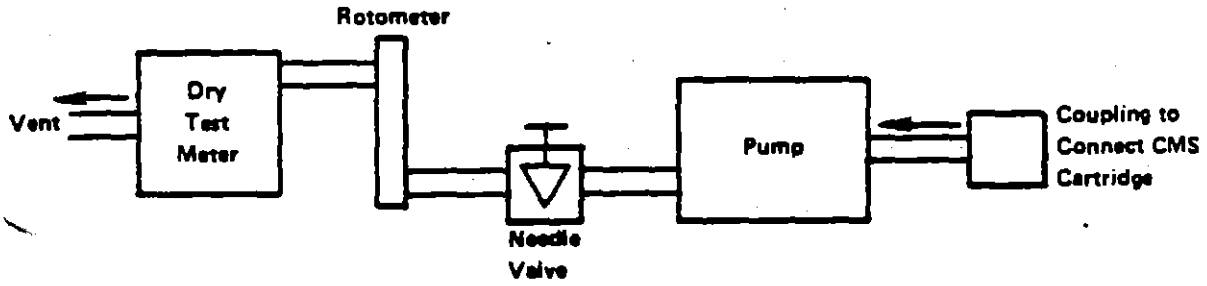


FIGURE 1. DIAGRAM SHOWING CARBON MOLECULAR SIEVE TRAP (CMS) CONSTRUCTION



(a) Mass Flow Control



(b) Needle Valve Control

FIGURE 2. TYPICAL SAMPLING SYSTEM CONFIGURATIONS

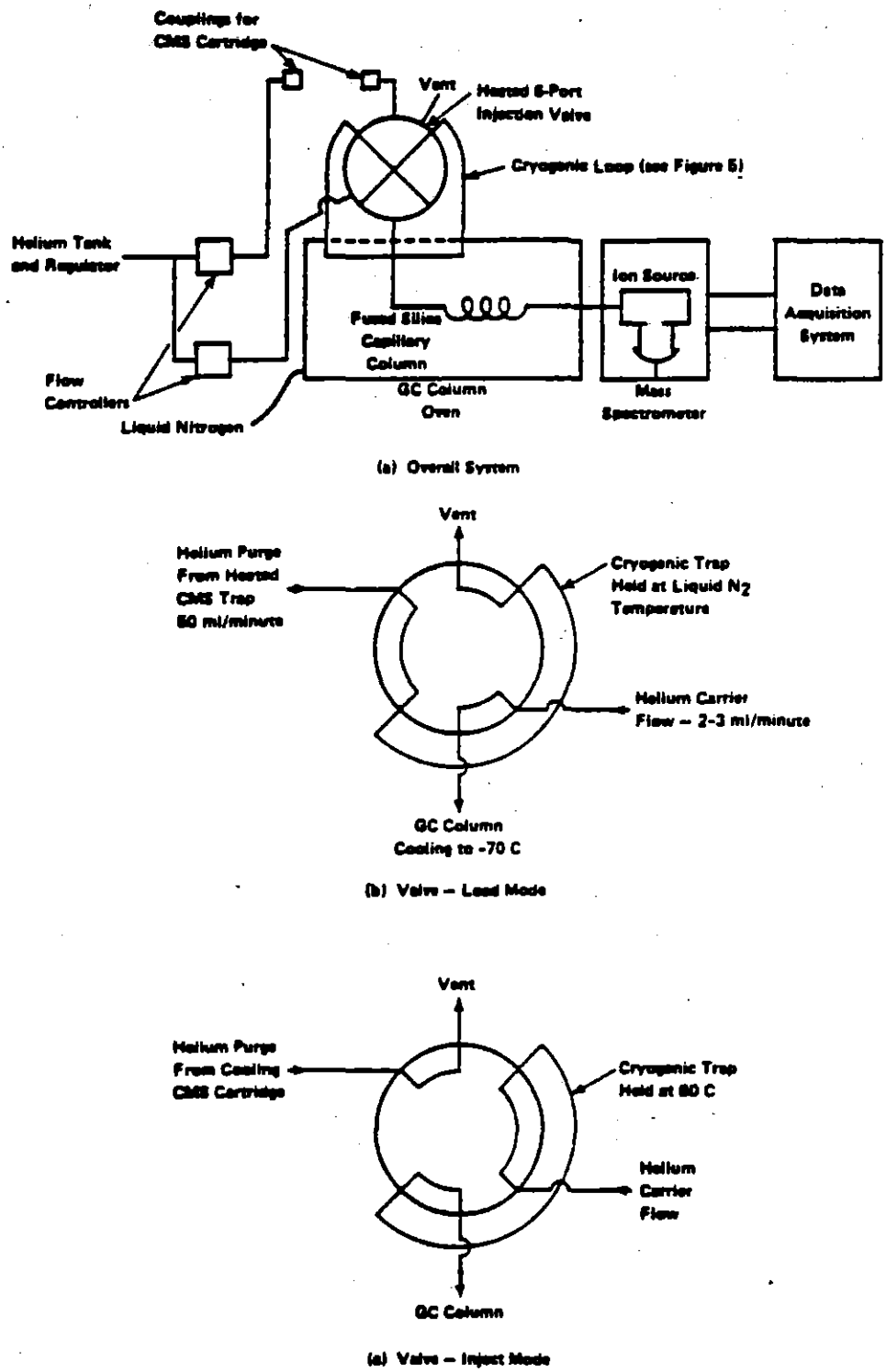


FIGURE 3. GC/MS ANALYSIS SYSTEM FOR CMS CARTRIDGES

SAMPLING DATA SHEET
(One Sample Per Data Sheet)

PROJECT: _____ DATE(S) SAMPLED: _____

SITE: _____ TIME PERIOD SAMPLED: _____

LOCATION: _____ OPERATOR: _____

INSTRUMENT MODEL NO: _____ CALIBRATED BY: _____

PUMP SERIAL NO: _____

SAMPLING DATA

Sample Number: _____

Start Time: _____ Stop Time: _____

	Time	Dry Gas Meter Reading	Rotameter Reading	Flow Rate,*Q ml/Min	Ambient Temperature °C	Barometric Pressure, mmHg	Relative Humidity, %	Comments
2.								
3.								
4.								
N.								

Total Volume Data**

V_m = (Final - Initial) Dry Gas Meter Reading, or = _____ Liters

$$\frac{Q_1 + Q_2 + Q_3 \ldots Q_N}{N} \times \frac{1}{1000 \times (\text{Sampling Time in Minutes})} = \_\_\_\_\_ \text{ Liters}$$

* Flowrate from rotameter or soap bubble calibrator (specify which).
** Use data from dry gas meter if available.

FIGURE 4. EXAMPLE SAMPLING DATA SHEET

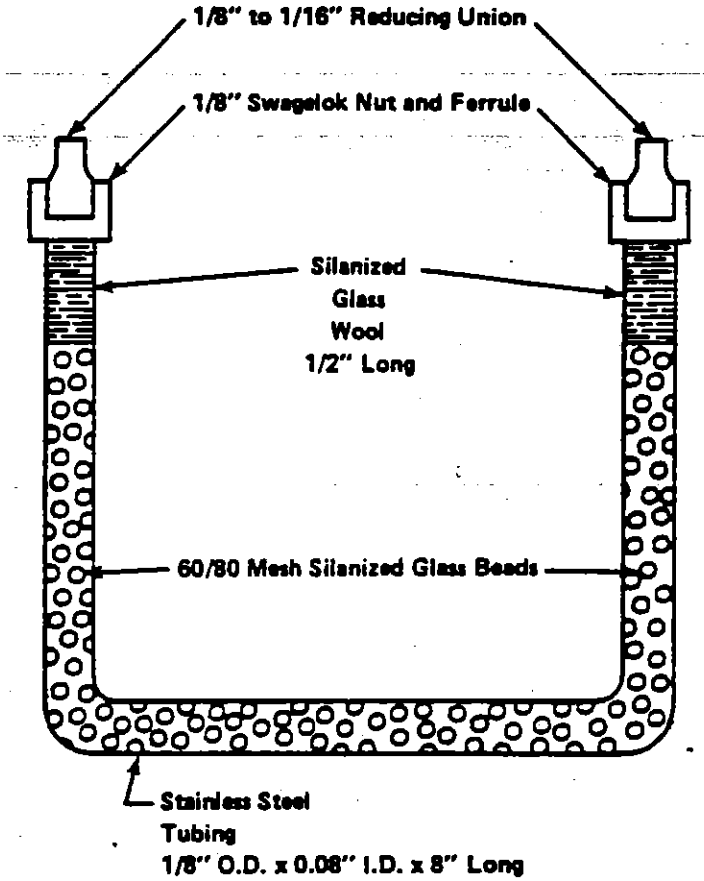
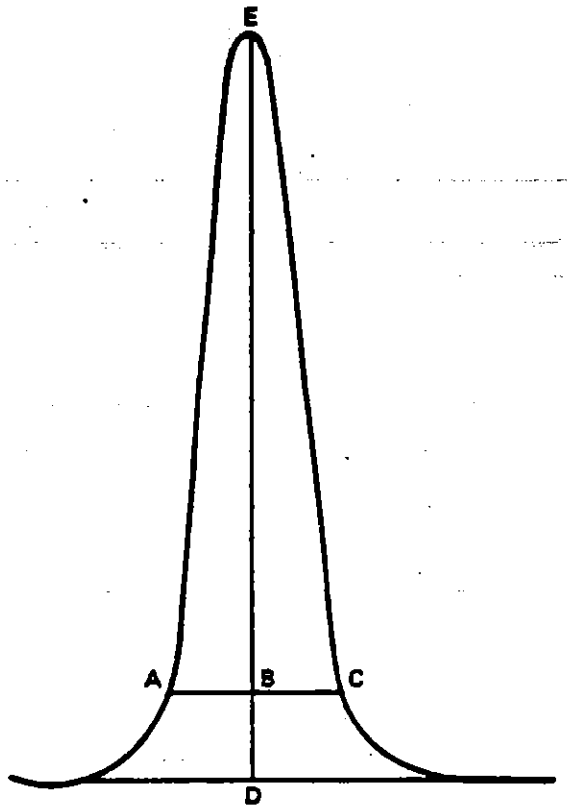


FIGURE 5. CRYOGENIC TRAP DESIGN



$$\text{Asymmetry Factor} = \frac{BC}{AB}$$

Example Calculation:

Peak Height = DE = 100 mm
10% Peak Height = BD = 10 mm
Peak Width at 10% Peak Height = AC = 23 mm
AB = 11 mm
BC = 12 mm

Therefore: Asymmetry Factor $= \frac{12}{11} = 1.1$

FIGURE 6. PEAK ASYMMETRY CALCULATION

METHOD FOR THE DETERMINATION OF ORGANOCHLORINE PESTICIDES AND POLYCHLORINATED BIPHENYLS IN AMBIENT AIR

1. Scope

1.1 This document describes a method for determination of a variety of organochlorine pesticides and polychlorinated biphenyls (PCBs) in ambient air. Generally, detection limits of >1 ng/m^3 are achievable using a 24-hour sampling period.

1.2 Specific compounds for which the method has been employed are listed in Table 1. Several references are available which provide further details on the development and application of the method. The sample cleanup and analysis methods are identical to those described in U. S. EPA Method 608. That method is included as Appendix A of this methods compendium.

2. Applicable Documents

2.1 ASTM Standards

D1356 Definition of Terms Related to Atmospheric Sampling and Analysis (7).

2.2 Other Documents

Ambient Air Studies (1-3)

U. S. EPA Technical Assistance Document (4).

U. S. EPA Method 608 (5). See Appendix A of methods compendium.

3. Summary of Method

3.1 A modified high volume sampler consisting of a glass fiber filter with a polyurethane foam (PUF) backup absorbent cartridge is used to sample ambient air at a rate of ∿200-280 L/minute.

3.2 The filter and PUF cartridge are placed in clean, sealed containers and returned to the laboratory for analysis. The PCBs and pesticides are recovered by Soxhlet extraction with 5% ether in hexane.

3.3 The extracts are reduced in volume using Kuderna-Danish (K-D) concentration techniques and subjected to column chromatographic cleanup.

3.4 The extracts are analyzed for pesticides and PCBs using gas chromatography with electron capture detection (GC-ECD), as described in U. S. EPA Method 608 (5).

4. Significance

4.1 Pesticides, particularly organochlorine pesticides, are widely used in both rural and urban areas for a variety of applications. PCBs are less widely used, due to extensive restrictions placed on their manufacture. However, human exposure to PCBs continues to be a problem because of their presence in various electrical products.

4.2 Many pesticides and PCBs exhibit bioaccumulative, chronic health effects and hence monitoring ambient air for such compounds is of great importance.

4.3 The relatively low levels of such compounds in the environment requires the use of high volume sampling techniques to acquire sufficient sample for analysis. However, the volatility of these compounds prevents efficient collection on filter media. Consequently, this method utilizes both a filter and a PUF backup cartridge which provides for efficient collection of most organochlorine pesticides, PCBs, and many other organics within the same volatility range.

5. Definitions

Definitions used in this document and any user-prepared SOPs should be consistent with ASTM D1356 (7). All abbreviations

and symbols are defined within this document at the point of use.

6. Interferences

6.1 The use of column chromatographic cleanup and selective GC detection (GC-ECD) minimizes the risk of interference from extraneous organic compounds. However, the fact that PCBs as well as certain organochlorine pesticides (e.g. toxaphene and chlordane) are complex mixtures of individual compounds can cause difficulty in accurately quantifying a particular formulation in a multiple component mixture.

6.2 Contamination of glassware and sampling apparatus with traces of pesticides or PCBs can be a major source of error in the method, particularly when sampling near high level sources (e.g. dumpsites, waste processing plants, etc.) careful attention to cleaning and handling procedures is required in all steps of the sampling and analysis to minimize this source of error.

7. Apparatus

7.1 Hi-Vol Sampler with PUF cartridge - available from General Metal Works (Model PS-1). See Figure 1.

7.2 Sampling Head to contain glass cartridge with PUF plug - available from General Metal Works. See Figure 2.

7.3 Calibration orifice - available from General Metal Works.

7.4 Manometer - to use with calibration orifice.

7.5 Soxhlet extraction system - including Soxhlet extractors (500 and 250 mL), heating mantels, variable voltage transformers, and cooling water source - for extraction of PUF cartridges before and after sampling. Also for extraction of filter samples.

7.6 Vacuum oven connected to water aspirator - for drying extracted PUF cartridges.

7.7 Gas chromatograph with electron capture detector - (consult U. S. EPA Method 608 for specifications).

7.8 Forceps - to handle quartz fiber filter samples.

7.9 Die - to cut PUF plugs.

7.10 Various items for extract preparation, cleanup, and analysis - consult U. S. EPA Method 608 for detailed listing.

7.11 Chromatography column - 2 mm I.D. x 15 cm long - for alumina cleanup.

8. Reagent and Materials

8.1 Polyurethane foam - 3 inch thick sheet stock, polyether type used in furniture upholstering. Density 0.022 g/cm^3.

8.2 Polyester gloves - for handling PUF cartridges and filters

8.3 Filters, quartz fiber - Pallflex 2500 QAST , or equivalent.

8.4 Wool felt filter - 4.9 mg/cm^2 and 0.6 mm thick. To fit sample head for collection efficiency studies. Pre-extracted with 5% diethyl ether in hexane.

8.5 Hexane - Pesticide or distilled in glass grade.

8.6 Diethyl ether - preserved with 2% ethanol - distilled in glass grade, or equivalent.

8.7 Acetone - Pesticide or distilled in glass grade.

8.8 Glass container for PUF cartridges.

8.9 Glass petri dish - for shipment of filters to and from the laboratory.

8.10 Ice chest - to store samples at ~0°C after collection.

8.11 Various materials needed for extract preparation, cleanup, and analysis - consult U. S. EPA Method 608 for details (Appendix A of this compendium).

8.12 Alumina - activity grade IV. 100/200 mesh

9. Assembly and Calibration of Sampling Apparatus

9.1 Description of Sampling Apparatus

9.1.1 The entire sampling system is diagrammed in Figure 1. This sampler was developed by Syracuse University

Research Corporation (SURC) under a U. S. EPA contract (6) and further modified by Southwest Research Institute and the U. S. EPA. A unit specifically designed for this method is now commercially available (Model PS-1 - General Metal Works, Inc., Village of Cleves, Ohio). The method writeup assumes the use of the commercial device, although the earlier modified device is also considered acceptable.

9.1.2 The sampling module (Figure 2) consists of a glass sampling cartridge and an air-tight metal cartridge holder. The PUF plug is retained in the glass sampling cartridge.

9.2 Calibration of Sampling System

9.2.1 The airflow through the sampling system is monitored by a venturi/Manehelic assembly, as shown in Figure 1. A multipoint calibration of the venturi/magnehelic assembly must be conducted every six months using an audit calibration orifice, as described in the U. S. EPA High Volume Sampling Method (8). A single point calibration must be performed before and after each sample collection, using the procedure described below.

9.2.2 Prior to calibration a "dummy" PUF cartridge and filter are placed in the sampling head and the sampling motor is activated. The flow control valve is fully opened and the voltage variator is adjusted so that a sample flow rate corresponding to ∿110% of the desired flow rate is indicated on the magnehelic (based on the previously obtained multipoint calibration curve). The motor is allowed to warmup for ∿10 minutes and then the flow control valve is adjusted to achieve the desired flow rate. The ambient temperature and barometric pressure should

be recorded on an appropriate data sheet (e.g. Figure 3).

9.2.3 The calibration orifice is then placed on the sampling head and a manometer is attached to the tap on the calibration orifice. The sampler is momentarily turned off to set the zero level of the manometer. The sampler is then switched on and the manometer reading is recorded, once a stable reading is achieved. The sampler is then shut off.

9.2.4 The calibration curve for the orifice is used to calculate sample flow from the data obtained in 9.2.3, and the calibration curve for the venturi/magnehelic assembly is used to calculate sample flow from the data obtained in 9.2.2. The calibration data should be recorded on an appropriate data sheet (e.g. Figure 3). If the two values do not agree within 10% the sampler should be inspected for damage, flow blockage, etc. If no obvious problems are found the sampler should be recalibrated (multipoint) according to the U. S. EPA High Volume Sampling procedure (8).

9.2.5 A multipoint calibration of the calibration orifice, against a primary standard, should be obtained annually.

10. Preparation of Sampling (PUF) Cartridges

10.1 The PUF adsorbent is a polyether-type polyurethane foam (density No. 3014 or 0.0225 g/cm^3). This type of foam is used for furniture upholstery. It is white and yellows on exposure to light.

10.2 The PUF inserts are 6.0 cm diameter cylindrical plugs cut from 3 inch sheet stock and should fit with slight compression in the glass cartridge, supported by the wire

screen. See Figure 2. During cutting the die is rotated at high speed (e.g. in a drill press) and continuously lubricated with water.

10.3 For initial cleanup the PUF plug is placed in a Soxhlet extractor and extracted with acetone for 14-24 hours at approximately 4 cycles per hour. When cartridges are reused, 5% diethyl ether in n-hexane can be used as the cleanup solvent.

10.4 The extracted PUF is placed in a vacuum oven connected to a water aspirator and dried at room temperature for approximately 2-4 hours (until no solvent odor is detected).

10.5 The PUF is placed into the glass sampling cartridge using polyester gloves. The module is wrapped with hexane rinsed aluminum foil, placed in a labeled container and tightly sealed.

10.6 Other adsorbents may be suitable for this method as indicated in the various references (1-3). If such materials are employed the user must define appropriate preparation procedures based on the information contained in these references.

10.7 At least one assembled cartridge from each batch must be analyzed, as a laboratory blank, using the procedures described in Section 12, before the batch is considered acceptable for field use. A blank level of <10 ng/plug for single compounds is considered to be acceptable. For multiple component mixtures (e.g. Arochlors) the blank level should be <100 ng/plug.

11. Sampling

11.1 After the sampling system has been assembled and calibrated as described in Section 9 it can be used to collect air samples as described below.

11.2 The samples should be located in an unobstructed area, at least two meters from any obstacle to air flow. The exhaust hose should be stretched out in the downwind

direction to prevent recycling of air.

11.3 A clean sampling cartridge and quartz fiber filter are removed from sealed transport containers and placed in the sampling head using forceps and gloved hands. The head is tightly sealed into the sampling system. The aluminum foil wrapping is placed back in the sealed container for later use.

11.4 The zero reading of the Magnehelic is checked. Ambient temperature, barometric pressure, elapsed time meter setting, sampler serial number, filter number and PUF cartridge number are recorded. A suitable data sheet is shown in Figure 4.

11.5 The voltage variator and flow control valve are placed at the settings used in 9.2.3 and the power switch is turned on. The elapsed time meter is activated and the start time recorded. The flow (Magnehelic setting) is adjusted, if necessary using the flow control valve.

11.6 The Magnehelic reading is recorded every six hours during the sampling period. The calibration curve (Section 9.2.7) is used to calculate the flow rate. Ambient temperature and barometric pressure are recorded at the beginning and end of the sampling period.

11.7 At the end of the desired sampling period the power is turned off and the filter and PUF cartridges are wrapped with the original aluminum foil and placed in sealed, labeled containers for transport back to the laboratory.

11.8 The Magnehelic calibration is checked using the calibration orifice as described in Section 9.2.4. If the calibration deviates by more than 10% from the initial reading the flow data for that sample must be marked as suspect and the sampler should be inspected and/or removed from service.

11.9 At least one field blank will be returned to the laboratory with each group of samples. A field blank is treated exactly as a sample except that no air is drawn through the cartridge.

11.10 Samples are stored at ∿20°C in an ice chest until receipt at the analytical laboratory, at which time they are stored refrigerated at 4°C.

12. Sample Preparation and Analysis

12.1 Sample Preparation

12.1.1 All samples should be extracted within 1 week after collection.

12.1.2 PUF cartridges are removed from the sealed con-container using gloved hands, the aluminum foil wrapping is removed, and the cartridges are placed into a 500-mL Soxhlet extraction. The cartridges are extracted for 14-24 hours at ∿4 cycles/hour with 5% diethyl ether in hexane. Extracted cartridges can be dried and reused following the handling procedures in Section 10. The quartz filter can be placed in the extractor with the PUF cartridges. However, if separate analysis is desired then one can proceed with 12.1.3.

12.1.3 If separate analysis is desired, quartz filters are placed in a 250-mL Soxhlet extractor and extracted for 14-24 hours with 5% diethyl ether in hexane.

12.1.4 The extracts are concentrated to 10 mL final volume using 500-mL Kuderna-Danish concentrators as described in EPA Method 608 (5), using a hot water bath. The concentrated extracts are stored refrigerated in sealed 4-dram vials having teflon-lined screw-caps until analyzed or subjected to cleanup.

12.2 Sample Cleanup

12.2.1 If only organochlorine pesticides and PCBs are sought, an alumina cleanup procedure reported in the literature is appropriate (1). Prior to cleanup the sample

extract is carefully reduced to 1 mL using a gentle steam of clean nitrogen.

12.2.2 A glass chromatographic column (2 mm ID x 15 cm long) is packed with alumina, activity grade IV and rinsed with ∿20 mL of n-hexane. The concentrated sample extract (from 12.2.1) is placed on the column and eluted with 10 mL of n-hexane at a rate of 0.5 mL/minute. The eluate volume is adjusted to exactly 10 mL and analyzed as described in 12.3.

12.2.3 If other pesticides are sought, alternate cleanup procedures (e.g. Florisil) may be required. Method 608 (5) identifies appropriate cleanup procedures.

12.3 Sample Analysis

12.3.1 Sample analysis is performed using GC/ECD as described in EPA Method 608 (5). The user must consult this method for detailed analytical procedures.

12.3.2 GC retention times and conditions are identified in Table 1 for the compounds of interest.

13. GC Calibration

Appropriate calibration procedures are identified in EPA Method 608 (5).

14. Calculations

14.1 The total sample volume (V_m) is calculated from the periodic flow readings (Magnehelic) taken in Section 11.6 using the following equation.

$$V_m = \frac{Q_1 + Q_2 \ldots Q_N}{N} \times \frac{T}{1000}$$

where

V_m = Total sample volume (m^3).

$Q_1, Q_2 \ldots Q_N$ = Flow rates determined at the beginning, end, and intermediate points during sampling (L/minute).

N = Number of data points averaged.

T = Elapsed sampling time (minutes).

14.2 The volume of air sampled can be converted to standard conditions (760 mm Hg pressure and 25°C) using the following equation:

$$V_s = V_m \times \frac{P_A}{760} \times \frac{298}{273+t_A}$$

where

V_s = Total sample volume at 25°C and 760 mm Hg pressure (m^3)

V_m = Total sample flow under ambient conditions (m^3)

P_A = Ambient pressure (mm Hg)

t_A = Ambient temperature (°C)

14.3 The concentration of compound in the sample is calculated using the following equation:

$$C_A = \frac{A \times V_E}{V_i \times V_s}$$

where

C_A = Concentration of analyte in the sample, $\mu g/m^3$

A = Calculated amount of material injected onto the chromatograph based on calibration curve for injected standards (nanograms)

V_i = Volume of extract injected (µL).

V_E = Final volume of extract (mL).
V_s = Total volume of air samples corrected to standard conditions (m^3).

14. Performance Criteria and Quality Assurance

This section summarizes the quality assurance (QA) measures and provides guidance concerning performance criteria which should be achieved within each laboratory.

14.1 Standard Operating Procedures (SOPs)

14.1.1 Users should generate SOPs describing the following activities as accomplished in their laboratory: 1) assembly, calibration and operation of the sampling system, 2) preparation, purification, storage and handling of sampling cartridges, 3) assembly, calibration and operation of the GC/ECD system, and 4) all aspects of data recording and processing.

14.1.2 SOPs should provide specific stepwise instructions and should be readily available to, and understood by, the laboratory personnel conducting the work.

14.2 Process, Field, and Solvent Blanks

14.2.1 One PUF cartridge and filter from each batch of approximately twenty should be analyzed, without shipment to the field, for the compounds of interest to serve as a process blank.

14.2.2 During each sampling episode at least one PUF cartridge and filter should be shipped to the field and returned, without drawing air through the sampler, to serve as a field blank.

14.2.3 During the analysis of each batch of samples at least one solvent process blank (all steps conducted but no PUF cartridge or filter included) should be

carried through the procedure and analyzed.

14.2.4 Blank levels should not exceed ∿10 ng/sample for single components or ∿100 ng/sample for multiple component mixtures (e.g. PCBs).

14.3 Collection Efficiency and Spike Recovery

14.3.1 Before using the method for sample analysis each laboratory must determine their collection efficiency for the components of interest.

14.3.2 The glass fiber filter in the sampler is replaced with a hexane-extracted wool felt filter (weight 14.9 mg/cm^2, 0.6 mm thick). The filter is spiked with microgram amounts of the compounds of interest by dropwise addition of hexane solutions of the compounds. The solvent is allowed to evaporate and filter is placed into the sampling system for immediate use.

14.3.3 The sampling system, including a clean PUF cartridge, is activated and set at the desired sampling flow rate. The sample flow is monitored for 24 hours.

14.3.4 The filter and PUF cartridge are then removed and analyzed as described in Section 12.

14.3.5 A second sample, unspiked is collected over the same time period to account for any background levels of components in the ambient air matrix.

14.3.6 A third PUF cartridge is spiked with the same amounts of the compounds used in 14.3.2 and extracted to determine analytical recovery.

14.3.7 In general analytical recoveries and collection efficiencies of 75% are considered to be acceptable method performance.

14.3.8 Replicate (at least triplicate) determinations of collection efficiency should be made. Relative standard deviations for these replicate determinations of ± 15% or less is considered acceptable performance.

14.3.9 Blind spiked samples should be included with sample sets periodically, as a check on analytical performance.

14.4 Method Precision and Accuracy

Typical method recovery data are shown in Table 1. Recoveries for the various chlorobiphenyls illustrate the fact that all components of an Arochlor mixture will not be retained to the same extent. Recoveries for tetrachlorobiphenyls and above are generally greater than 85% but di- and trichloro homologs may not be recovered quantitatively.

REFERENCES

1. Lewis, R. G., Brown, A. R., and Jackson, M. D., "Evaluation of Polyurethane Foam for Sampling of Pesticides, Polychlorinated Biphenyls, and Polychlorinated Naphthalenes in Ambient Air", Anal. Chem. 49, 1668-1672, 1977.

2. Lewis, R. G. and Jackson, M. D., "Modification and Evaluation of a High-Volume Air Sampler for Pesticides and Semivolatile Industrial Organic Chemicals", Anal. Chem. 54, 592-594, 1982.

3. Lewis, R. G., Jackson, M. D., and MacLeod, K. E., "Protocol for Assessment of Human Exposure to Airborne Pesticides", EPA-600/2-80-180, U.S. Environmental Protection Agency, Research Triangle Park, NC, 1980.

4. Riggin, R. M., "Technical Assistance Document for Sampling and Analysis of Toxic Organic Compounds in Ambient Air", EPA-600/4-83-027., U. S. Environmental Protection Agency, Research Triangle Park, NC, 1983.

5. Longbottom, J. E. and Lichtenberg, J. J., "Methods for Organic Chemical Analysis of Municipal and Industrial Wastewater", EPA-600/4-82-057, U. S. Environmental Protection Agency, Cincinnati, OH, 1982.

6. Bjorkland, J., Compton, B., and Zweig, G., "Development of Methods for Collection and Analysis of Airborne Pesticides." Report for Contract No. CPA 70-15, National Air Pollution Control Association, Durham, NC, 1970.

7. Annual Book of ASTM Standards, Part 11.03, "Atmospheric Analysis", American Society for Testing and Materials, Philadelphia, PA, 1983.

8. Reference Method for the Determination of Suspended Particulates in the Atmosphere (High Volume Method). Federal Register, Sept. 14, 1972 or 40CFR50 Appendix B.

TABLE 1. SELECTED COMPONENTS DETERMINED USING HI-VOL/PUF SAMPLING PROCEDURE

Compound	GC Retention Time, Minutes[a]	24-Hour Sampling Efficiency[b]	
		Air Concentration ng/m^3	% Recovery
Aldrin	2.4	0.3-3.0	28
4,4'-DDE	5.1	0.6-6.0	89
4,4'-DDT	9.4	1.8-18	83
Chlordane	(c)	15-150	73
Chlorobiphenyls			
4,4' Di-	--	2.0-20	62
2,4,5 Tri-	---	0.2-2.0	36
2,4',5 Tri-	--	0.2-2.0	86
2,2',5,5' Tetra-	--	0.2-2.0	94
2,2',4,5,5' Penta-	--	0.2-2.0	92
2,2',4,4',5,5' Hexa	--	0.2-2.0	86

(a) Data from U.S. EPA Method 608. Conditions are as follows:

Stationary Phase - 1.5% SP2250/1.95% SP-2401 on Supelcoport (100/120 mesh) packed in 1.8 mm long x 4 mm ID glass column.

Carrier - 5/95 methane/Argon at 60 mL/Minute

Column Temperature - 160°C except for PCBs which are determined at 200°C.

(b) From Reference 2.

(c) Multiple component formulation. See U.S. EPA Method 608.

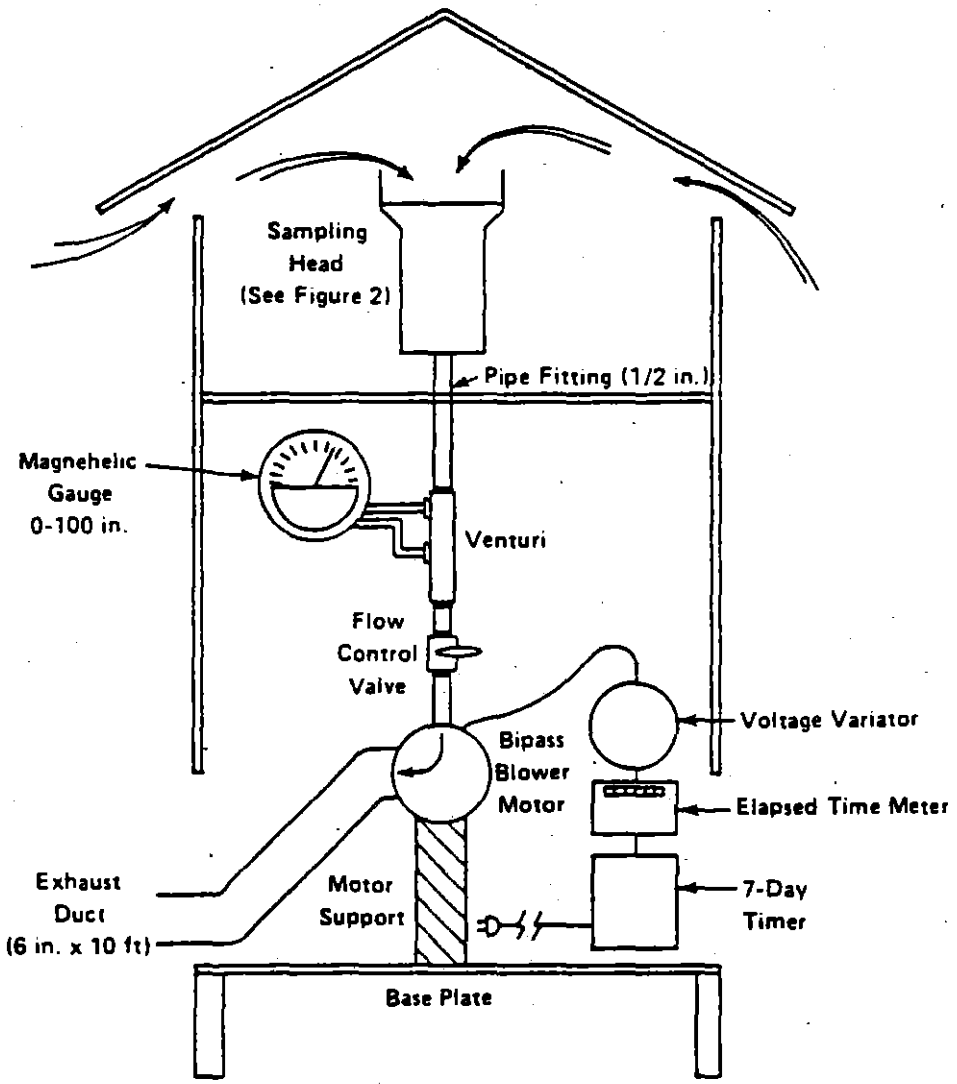


FIGURE 1. HIGH VOLUME AIR SAMPLER. AVAILABLE FROM GENERAL METAL WORKS (MODEL PS-1)

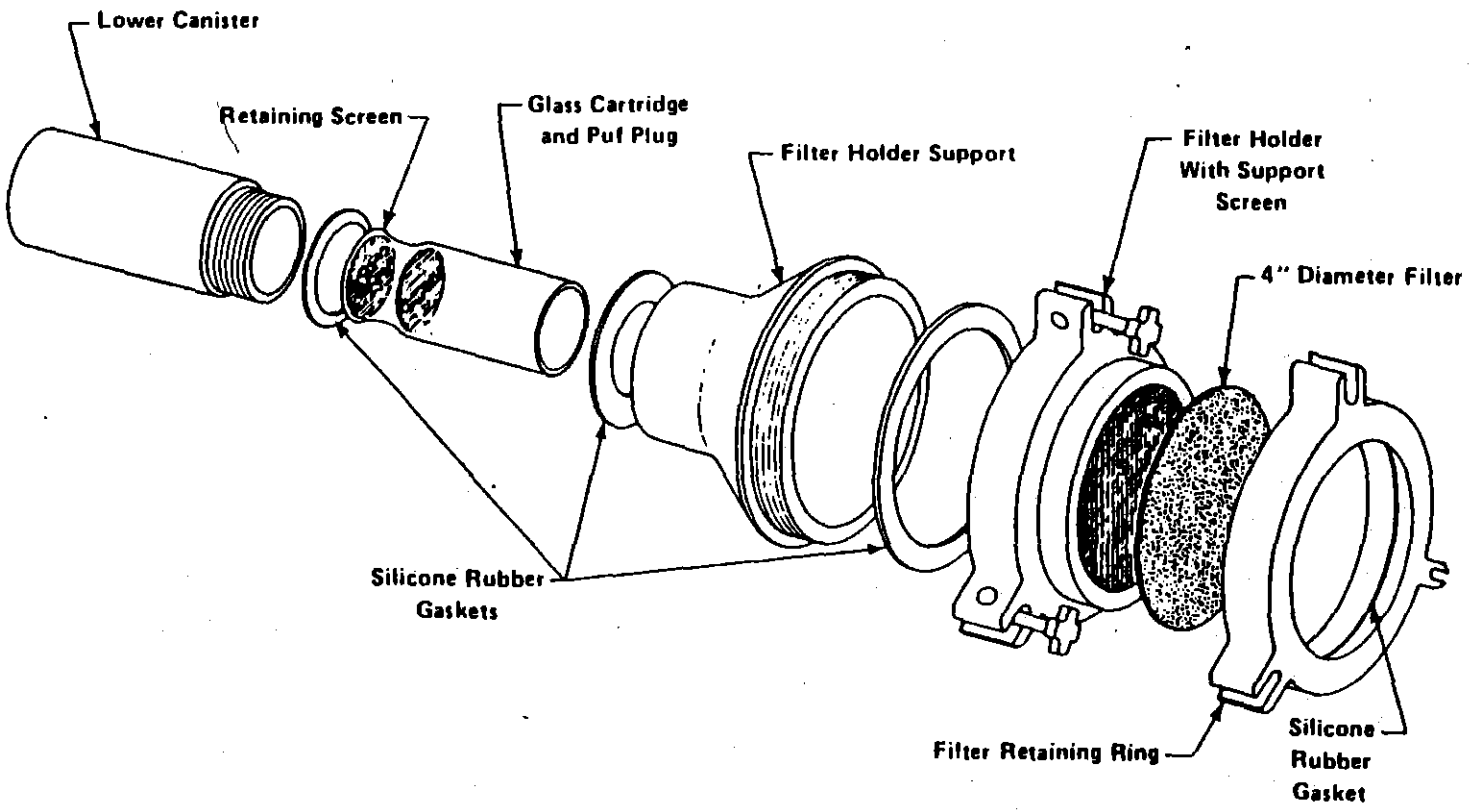


FIGURE 2. SAMPLING HEAD

Performed by_____ Calibration Orifice S/N_____ Ambient Temperature_____°C

Date/Time_____ Manometer S/N _____ Bar.Press. _____ mm Hg

Sampler S/N	Variac Setting V	Timer OK? Yes/No	Calibration Orifice Data		Sampler Venturi Data		% Difference Between Calibration and Sample Venturi Flow Rates	Comments
			Manometer, in. H_2O	Flow Rate, scm /min[a]	Magnehelic, in. H_2O	Flow Rate scm/min [b]		

(a) From Calibration Tables for Calibration Orifice or Venturi Tube

(b) From Calibration Tables for Venturi Tube in each Hi-Vol unit. Date check by_____ Date_____

FIGURE 3. TYPICAL CALIBRATION SHEET FOR HIGH VOLUME SAMPLER

Site _____ Date _____ Performed by _____

Sampler S/N	Sampling Location I.D.	New Filter (a)	PUF Cart. No.	Vernier Setting	Clock Time			Sampler Timer			Venturi Readings Time/Magnehelic in. H_2O				Ambient Temperature °C		Barometric Pressure mm Hg		Comments
					Start hr CDT	Stop hr CDT	Min Elapsed	Start min	Stop min	Min Elapsed	1	2	3	4	Start	Stop	Start	Stop	

(a) Record any evidence of tampering with sampler and/or abnormalities in sampler operation, PUF cartridge condition or handling etc.

Data Checked By _____ Date _____

FIGURE 4. TYPICAL SAMPLING DATA FORM FOR HIGH VOLUME PESTICIDE/PCB SAMPLER

11.0 REFERENCE METHOD*

APPENDIX B—REFERENCE METHOD FOR THE DETERMINATION OF SUSPENDED PARTICULATES IN THE ATMOSPHERE (HIGH VOLUME METHOD)

1. *Principle and Applicability.*

1.1 Air is drawn into a covered housing and through a filter by means of a high-flow-rate blower at a flow rate (1.13 to 1.70 m.³/min.; 40 to 60 ft.³/min.) that allows suspended particles having diameters of less than 100 μm. (Stokes equivalent diameter) to pass to the filter surface. (*1*) Particles within the size range of 100 to 0.1μm. diameter are ordinarily collected on glass fiber filters. The mass concentration of suspended particulates in the ambient air (μg./m.³) is computed by measuring the mass of collected particulates and the volume of air sampled.

1.2 This method is applicable to measurement of the mass concentration of suspended particulates in ambient air. The size of the sample collected is usually adequate for other analyses.

2. *Range and Sensitivity.*

2.1 When the sampler is operated at an average flow rate of 1.70 m.³/min. (60 ft.³/min.) for 24 hours, an adequate sample will be obtained even in an atmosphere having concentrations of suspended particulates as low as 1 μg./m.³. If particulate levels are unusually high, a satisfactory sample may be obtained in 6 to 8 hours or less. For determination of average concentrations of suspended particulates in ambient air, a standard sampling period of 24 hours is recommended.

2.2 Weights are determined to the nearest milligram, airflow rates are determined to the nearest 0.03 m.³/min. (1.0 ft.³/min.), times are determined to the nearest 2 minutes, and mass concentrations are reported to the nearest microgram per cubic meter.

3. *Interferences.*

3.1 Particulate matter that is oily, such as photochemical smog or wood smoke, may block the filter and cause a rapid drop in airflow at a nonuniform rate. Dense fog or high humidity can cause the filter to become too wet and severely reduce the airflow through the filter.

3.2 Glass-fiber filters are comparatively insensitive to changes in relative humidity, but collected particulates can be hygroscopic. (*2*)

4. *Precision, Accuracy, and Stability.*

4.1 Based upon collaborative testing, the relative standard deviation (coefficient of variation) for single analyst variation (repeatability of the method) is 3.0 percent. The corresponding value for multilaboratory variation (reproducibility of the method) is 3.7 percent. (*3*)

4.2 The accuracy with which the sampler measures the true average concentration depends upon the constancy of the airflow rate through the sampler. The airflow rate is affected by the concentration and the nature of the dust in the atmosphere. Under these conditions the error in the measured average concentration may be in excess of ±50 percent of the true average concentration, depending on the amount of reduction of airflow rate and on the variation of the mass concentration of dust with time during the 24-hour sampling period. (*4*)

5. *Apparatus.*

5.1 *Sampling.*

5.1.1 *Sampler.* The sampler consists of three units: (1) the faceplate and gasket, (2) the filter adapter assembly, and (3) the motor unit. Figure B1 shows an exploded view of these parts, their relationship to each other, and how they are assembled. The sampler must be capable of passing environmental air through a 406.5 cm.² (63 in.²) portion of a clean 20.3 by 25.4 cm. (8- by 10-in.) glass-fiber filter at a rate of at least 1.70 m.³/min. (60 ft.³/min.). The motor must be capable of continuous operation for 24-hour periods with input voltages ranging from 110 to 120 volts, 50–60 cycles alternating current and must have third-wire safety ground. The housing for the motor unit may be of any convenient construction so long as the unit remains airtight and leak-free. The life of the sampler motor can be extended by lowering the voltage by about 10 percent with a small "buck or boost" transformer between the sampler and power outlet.

5.1.2 *Sampler Shelter.* It is important that the sampler be properly installed in a suitable shelter. The shelter is subjected to extremes of temperature, humidity, and all types of air pollutants. For these reasons the materials of the shelter must be chosen carefully. Properly painted exterior plywood or heavy gauge aluminum serve well. The sampler must be mounted vertically in the shelter so that the glass-fiber filter is parallel with the ground. The shelter must be provided with a roof so that the filter is protected from precipitation and debris. The internal arrangement and configuration of a suitable shelter with a gable roof are shown in Figure B2. The clearance area between the main housing and the roof at its closest point should be 580.5±193.5 cm.² (90±30 in.²). The main housing should be rectangular, with dimensions of about 29 by 36 cm. (11½ by 14 in.).

5.1.3 *Rotameter.* Marked in arbitrary units, frequently 0 to 70, and capable of being calibrated. Other devices of at least comparable accuracy may be used.

5.1.4 *Orifice Calibration Unit.* Consisting of a metal tube 7.6 cm. (3 in.) ID and 15.9 cm. (6¼ in.) long with a static pressure tap 5.1 cm. (2 in.) from one end. See Figure B3. The tube end nearest the pressure tap is flanged to about 10.8 cm. (4¼ in.) OD with a male thread of the same size as the inlet end of the high-volume air sampler. A single metal plate 9.2 cm. (3⅝ in.) in diameter and 0.24 cm. (3/32 in.) thick with a central orifice 2.9 cm. (1⅛ in.) in diameter is held in place at the air inlet end with a female threaded ring. The other end of the tube is flanged to

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*Reproduced from Code of Federal Regulation 40, Part 50.11, Appendix B, July 1, 1975, Pages 12-16.

hold a loose female threaded coupling, which screws onto the inlet of the sampler. An 18-hole metal plate, an integral part of the unit, is positioned between the orifice and sampler to simulate the resistance of a clean glass-fiber filter. An orifice calibration unit is shown in Figure B3.

5.1.5 *Differential Manometer.* Capable of measuring to at least 40 cm. (16 in.) of water.

5.1.6 *Positive Displacement Meter.* Calibrated in cubic meters or cubic feet, to be used as a primary standard.

5.1.7 *Barometer.* Capable of measuring atmospheric pressure to the nearest mm.

5.2 *Analysis.*

5.2.1 *Filter Conditioning Environment.* Balance room or desiccator maintained at 15° to 35°C. and less than 50 percent relative humidity.

5.2.2 *Analytical Balance.* Equipped with a weighing chamber designed to handle unfolded 20.3 by 25.4 cm. (8- by 10-in.) filters and having a sensitivity of 0.1 mg.

5.2.3 *Light Source.* Frequently a table of the type used to view X-ray films.

5.2.4 *Numbering Device.* Capable of printing identification numbers on the filters.

6. *Reagents.*

6.1 *Filter Media.* Glass-fiber filters having a collection efficiency of at least 99 percent for particles of 0.3 μm. diameter, as measured by the DOP test, are suitable for the quantitative measurement of concentrations of suspended particulates, (*5*) although some other medium, such as paper, may be desirable for some analyses. If a more detailed analysis is contemplated, care must be exercised to use filters that contain low background concentrations of the pollutant being investigated. Careful quality control is required to determine background values of these pollutants.

7. *Procedure.*

7.1 *Sampling.*

7.1.1 *Filter Preparation.* Expose each filter to the light source and inspect for pinholes, particles, or other imperfections. Filters with visible imperfections should not be used. A small brush is useful for removing particles. Equilibrate the filters in the filter conditioning environment for 24 hours. Weigh the filters to the nearest milligram; record tare weight and filter identification number. Do not bend or fold the filter before collection of the sample.

7.1.2 *Sample Collection.* Open the shelter, loosen the wing nuts, and remove the faceplate from the filter holder. Install a numbered, preweighed, glass-fiber filter in position (rough side up), replace the faceplate without disturbing the filter, and fasten securely. Undertightening will allow air leakage, overtightening will damage the sponge-rubber faceplate gasket. A very light application of talcum powder may be used on the sponge-rubber faceplate gasket to prevent the filter from sticking. During inclement weather the sampler may be removed to a protected area for filter change. Close the roof of the shelter, run the sampler for about 5 minutes, connect the rotameter to the nipple on the back of the sampler, and read the rotameter ball with rotameter in a vertical position. Estimate to the nearest whole number. If the ball is fluctuating rapidly, tip the rotameter and slowly straighten it until the ball gives a constant reading. Disconnect the rotameter from the nipple; record the initial rotameter reading and the starting time and date on the filter folder. (The rotameter should never be connected to the sampler except when the flow is being measured.) Sample for 24 hours from midnight to midnight and take a final rotameter reading. Record the final rotameter reading and ending time and date on the filter folder. Remove the faceplate as described above and carefully remove the filter from the holder, touching only the outer edges. Fold the filter lengthwise so that only surfaces with collected particulates are in contact, and place in a manila folder. Record on the folder the filter number, location, and any other factors, such as meteorological conditions or razing of nearby buildings, that might affect the results. If the sample is defective, void it at this time. In order to obtain a valid sample, the high-volume sampler must be operated with the same rotameter and tubing that were used during its calibration.

7.2 *Analysis.* Equilibrate the exposed filters for 24 hours in the filter conditioning environment, then reweigh. After they are weighed, the filters may be saved for detailed chemical analysis.

7.3 *Maintenance.*

7.3.1 *Sampler Motor.* Replace brushes before they are worn to the point where motor damage can occur.

7.3.2 *Faceplate Gasket.* Replace when the margins of samples are no longer sharp. The gasket may be sealed to the faceplate with rubber cement or double-sided adhesive tape.

7.3.3 *Rotameter.* Clean as required, using alcohol.

8. *Calibration.*

8.1 *Purpose.* Since only a small portion of the total air sampled passes through the rotameter during measurement, the rotameter must be calibrated against actual airflow with the orifice calibration unit. Before the orifice calibration unit can be used to calibrate the rotameter, the orifice calibration unit itself must be calibrated against the positive displacement primary standard.

8.1.1 *Orifice Calibration Unit.* Attach the orifice calibration unit to the intake end of the positive displacement primary standard and attach a high-volume motor blower unit to the exhaust end of the primary standard. Connect one end of a differential manometer to the differential pressure tap of the orifice calibration unit and leave the other end open to the atmosphere. Operate the high-volume motor blower unit so that a series of different, but constant, airflows (usually six) are obtained for definite time periods. Record the reading on the differential manometer at each airflow. The different constant airflows are obtained by placing a

series of loadplates, one at a time, between the calibration unit and the primary standard. Placing the orifice before the inlet reduces the pressure at the inlet of the primary standard below atmospheric; therefore, a correction must be made for the increase in volume caused by this decreased inlet pressure. Attach one end of a second differential manometer to an inlet pressure tap of the primary standard and leave the other open to the atmosphere. During each of the constant airflow measurements made above, measure the true inlet pressure of the primary standard with this second differential manometer. Measure atmospheric pressure and temperature. Correct the measured air volume to true air volume as directed in 9.1.1, then obtain true airflow rate, Q, as directed in 9.1.3. Plot the differential manometer readings of the orifice unit versus Q.

8.1.2 *High-Volume Sampler.* Assemble a high-volume sampler with a clean filter in place and run for at least 5 minutes. Attach a rotameter, read the ball, adjust so that the ball reads 65, and seal the adjusting mechanism so that it cannot be changed easily. Shut off motor, remove the filter, and attach the orifice calibration unit in its place. Operate the high-volume sampler at a series of different, but constant, airflows (usually six). Record the reading of the differential manometer on the orifice calibration unit, and record the readings of the rotameter at each flow. Measure atmospheric pressure and temperature. Convert the differential manometer reading to m.³/min., Q, then plot rotameter reading versus Q.

8.1.3 *Correction for Differences in Pressure or Temperature.* See Addendum B.

9. *Calculations.*

9.1 *Calibration of Orifice.*

9.1.1 *True Air Volume.* Calculate the air volume measured by the positive displacement primary standard.

$$V_a = \frac{(P_a - P_m)}{P_a}(V_M)$$

V_a = True air volume at atmospheric pressure, m.³
P_a = Barometric pressure, mm. Hg.
P_m = Pressure drop at inlet of primary standard, mm. Hg.
V_M = Volume measured by primary standard, m.³

9.1.2 *Conversion Factors.*
Inches Hg. × 25.4 = mm. Hg.
Inches water × 73.48 × 10^{-4} = inches Hg.
Cubic feet air × 0.0284 = cubic meters air.

9.1.3 *True Airflow Rate.*

$$Q = \frac{V_a}{T}$$

Q = Flow rate, m.³/min.
T = Time of flow, min.

9.2 *Sample Volume.*

9.2.1 *Volume Conversion.* Convert the initial and final rotameter readings to true airflow rate, Q, using calibration curve of 8.1.2.

9.2.2 *Calculate volume of air sampled*

$$* \quad V = \frac{Q_i Q_f}{2} \times T$$

V = Air volume sampled, m.³
Q_i = Initial airflow rate, m.³/min.
Q_f = Final airflow rate, m.³/min.
T = Sampling time, min.

9.3 *Calculate mass concentration of suspended particulates*

$$S.P. = \frac{(W_f - W_i) \times 10^6}{V}$$

S.P. = Mass concentration of suspended particulates, μg/m.³
W_i = Initial weight of filter, g.
W_f = Final weight of filter, g.
V = Air volume sampled, m.³
10^6 = Conversion of g. to μg.

10. *References.*

(1) Robson, C. D., and Foster, K. E., "Evaluation of Air Particulate Sampling Equipment", *Am. Ind. Hyg. Assoc. J. 24*, 404 (1962).

(2) Tierney, G. P., and Conner, W. D., "Hygroscopic Effects on Weight Determinations of Particulates Collected on Glass-Fiber Filters", *Am. Ind. Hyg. Assoc. J. 28*, 363 (1967).

(3) Unpublished data based on a collaborative test involving 12 participants, conducted under the direction of the Methods Standardization Services Section of the National Air Pollution Control Administration. October, 1970.

(4) Harrison, W. K., Nader, J. S., and Fugman, F. S., "Constant Flow Regulators for High-Volume Air Sampler", *Am. Ind. Hyg. Assoc. J. 21*, 114–120 (1960).

(5) Pate, J. B., and Tabor, E. C., "Analytical Aspects of the Use of Glass-Fiber Filters for the Collection and Analysis of Atmospheric Particulate Matter", *Am. Ind. Hyg. Assoc. J. 23*, 144–150 (1962).

ADDENDA

A. *Alternative Equipment.*

A modification of the high-volume sampler incorporating a method for recording the actual airflow over the entire sampling period has been described, and is acceptable for measuring the concentration of suspended particulates (Henderson, J. S., Eighth Conference on Methods in Air Pollution and Industrial Hygiene Studies, 1967, Oakland,

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*This equation should read $V = \frac{(Q_i + Q_f)}{2} \times T$

Calif.). This modification consists of an exhaust orifice meter assembly connected through a transducer to a system for continuously recording airflow on a circular chart. The volume of air sampled is calculated by the following equation:

$$V = Q \times T.$$

Q = Average sampling rate, $m.^3$/min.
T = Sampling time, minutes.

The average sampling rate, Q, is determined from the recorder chart by estimation if the flow rate does not vary more than 0.11 $m.^3$/min. (4 $ft.^3$/min.) during the sampling period. If the flow rate does vary more than 0.11 $m.^3$ (4 $ft.^3$/min.) during the sampling period, read the flow rate from the chart at 2-hour intervals and take the average.

B. *Pressure and Temperature Corrections.*

If the pressure or temperature during high-volume sampler calibration is substantially different from the pressure or temperature during orifice calibration, a correction of the flow rate, Q, may be required. If the pressures differ by no more than 15 percent and the temperatures differ by no more than 100 percent (°C), the error in the uncorrected flow rate will be no more than 15 percent. If necessary, obtain the corrected flow rate as directed below. This correction applies only to orifice meters having a constant orifice coefficient. The coefficient for the calibrating orifice described in 5.1.4 has been shown experimentally to be constant over the normal operating range of the high-volume sampler (0.6 to 2.2 $m.^3$/min.; 20 to 78 $ft.^3$/min.). Calculate corrected flow rate:

$$Q_2 = Q_1 \left[\frac{T_2 P_1}{T_1 P_2}\right]^{1/2}$$

Q_2 = Corrected flow rate, $m.^3$/min.
Q_1 = Flow rate during high-volume sampler calibration (Section 8.1.2), $m.^3$/min.
T_1 = Absolute temperature during orifice unit calibration (Section 8.1.1), °K or °R.
P_1 = Barometric pressure during orifice unit calibration (Section 8.1.1), mm. Hg.
T_2 = Absolute temperature during high-volume sampler calibration (Section 8.1.2), °K or °R.
P_2 = Barometric pressure during high-volume sampler calibration (Section 8.1.2), mm. Hg.

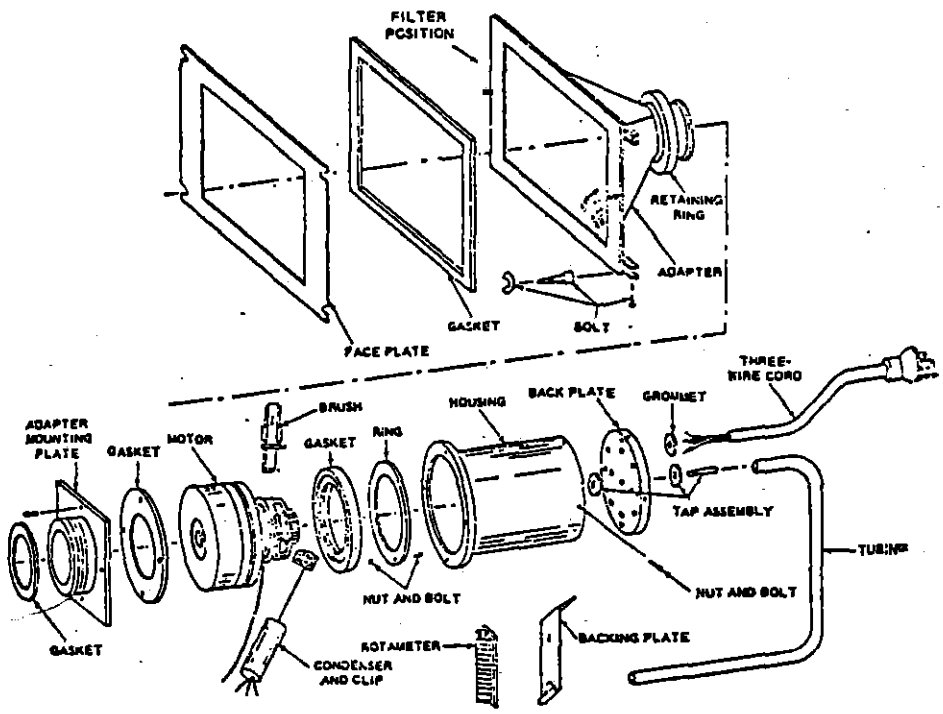


Figure B1. Exploded view of typical high-volume air sampler parts.

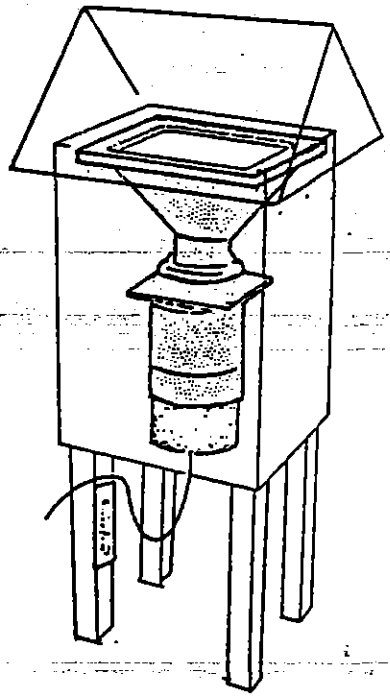


Figure B2. Assembled sampler and shelter.

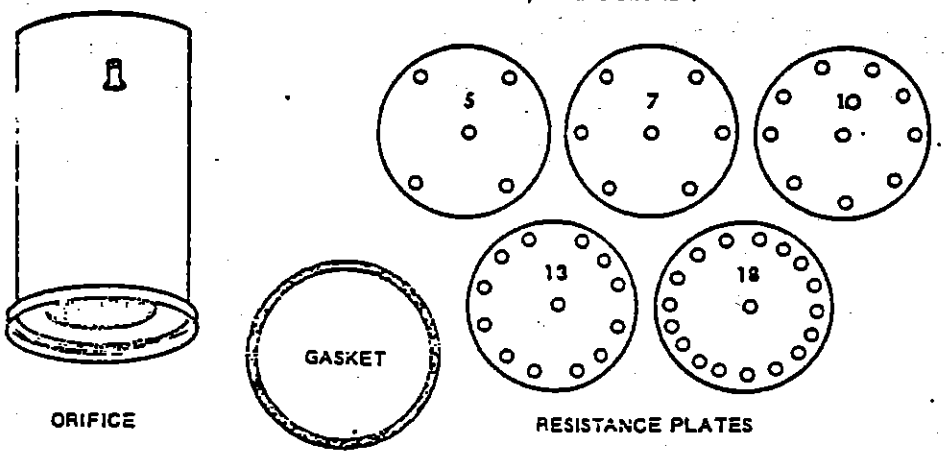


Figure B3. Orifice calibration unit.

METHOD TO1

Revision 1.0
April, 1984

ME ON OF VOLATILE ORGANIC COMPOUNDS NG TENAX® ADSORPTION AND MASS SPECTROMETRY (GC/MS)

1. Scope

1.1 The document describes a generalized protocol for collection and determination of certain volatile organic compounds which can be captured on Tenax® GC (poly(2,6-Diphenyl phenylene oxide)) and determined by thermal desorption GC/MS techniques. Specific approaches using these techniques are described in the literature (1-3).

1.2 This protocol is designed to allow some flexibility in order to accommodate procedures currently in use. However, such flexibility also results in placement of considerable responsibility with the user to document that such procedures give acceptable results (i.e. documentation of method performance within each laboratory situation is required). Types of documentation required are described elsewhere in this method.

1.3 Compounds which can be determined by this method are nonpolar organics having boiling points in the range of approximately 80° - 200°C. However, not all compounds falling into this category can be determined. Table 1 gives a listing of compounds for which the method has been used. Other compounds may yield satisfactory results but validation by the individual user is required.

2. Applicable Documents

2.1 ASTM Standards:

D1356 Definitions of Terms Related to Atmospheric Sampling and Analysis.

E355 Recommended Practice for Gas Chromatography Terms and Relationships.

2.3 Other documents:

Existing procedures (1-3).
U.S. EPA Technical Assistance Document (4).

3. Summary of Protocol

3.1 Ambient air is drawn through a cartridge containing ∿1-2 grams of Tenax and certain volatile organic compounds are trapped on the resin while highly volatile organic compounds and most inorganic atmospheric constituents pass through the cartridge. The cartridge is then transferred to the laboratory and analyzed.

3.2 For analysis the cartridge is placed in a heated chamber and purged with an inert gas. The inert gas transfers the volatile organic compounds from the cartridge onto a cold trap and subsequently onto the front of the GC column which is held at low temperature (e.g. - 70°C). The GC column temperature is then increased (temperature programmed) and the components eluting from the column are identified and quantified by mass spectrometry. Component identification is normally accomplished, using a library search routine, on the basis of the GC retention time and mass spectral characteristics. Less sophistacated detectors (e.g. electron capture or flame ionization) may be used for certain applications but their suitability for a given application must be verified by the user.

3.3 Due to the complexity of ambient air samples only high resolution (i.e. capillary) GC techniques are considered to be acceptable in this protocol.

4. Significance

4.1 Volatile organic compounds are emitted into the atmosphere from a variety of sources including industrial and commercial facilities, hazardous waste storage facilities, etc. Many of these compounds are toxic; hence knowledge of the levels of

such materials in the ambient atmosphere is required in order to determine human health impacts.

4.2 Conventional air monitoring methods (e.g. for workspace monitoring) have relied on carbon adsorption approaches with subsequent solvent desorption. Such techniques allow subsequent injection of only a small portion, typically 1-5% of the sample onto the GC system. However, typical ambient air concentrations of these compounds require a more sensitive approach. The thermal desorption process, wherein the entire sample is introduced into the analytical (GC/MS) system fulfills this need for enhanced sensitivity.

5. Definitions

Definitions used in this document and any user prepared SOPs should be consistent with ASTM D1356(6). All abbreviations and symbols are defined with this document at the point of use.

6. INTERFERENCES

6.1 Only compounds having a similar mass spectrum and GC retention time compared to the compound of interest will interfere in the method. The most commonly encountered interferences are structural isomers.

6.2 Contamination of the Tenax cartridge with the compound(s) of interest is a commonly encountered problem in the method. The user must be extremely careful in the preparation, storage, and handling of the cartridges throughout the entire sampling and analysis process to minimize this problem.

7. Apparatus

7.1 Gas Chromatograph/Mass Spectrometry system - should be capable of subambient temperature programming. Unit mass resolution or better up to 800 amu. Capable of scanning 30-440 amu region every 0.5-1 second. Equipped with data system for instrument control as well as data acquisition, processing and storage.

7.2 Thermal Desorption Unit - Designed to accommodate Tenax cartridges in use. See Figure 2a or b.

7.3 Sampling System - Capable of accurately and precisely drawing an air flow of 10-500 ml/minute through the Tenax cartridge. (See Figure 3a or b.)

7.4 Vacuum oven - connected to water aspirator vacuum supply.

7.5 Stopwatch

7.6 Pyrex disks - for drying Tenax.

7.7 Glass jar - Capped with Teflon-lined screw cap. For storage of purified Tenax.

7.8 Powder funnel - for delivery of Tenax into cartridges.

7.9 Culture tubes - to hold individual glass Tenax cartridges.

7.10 Friction top can (paint can) - to hold clean Tenax cartridges.

7.11 Filter holder - stainless steel or aluminum (to accommodate 1 inch diameter filter). Other sizes may be used if desired. (optional)

7.12 Thermometer - to record ambient temperature.

7.13 Barometer (optional).

7.14 Dilution bottle - Two-liter with septum cap for standards preparation.

7.15 Teflon stirbar - 1 inch long.

7.16 Gas-tight glass syringes with stainless steel needles - 10-500 µl for standard injection onto GC/MS system.

7.17 Liquid microliter syringes - 5,50 µL for injecting neat liquid standards into dilution bottle.

7.18 Oven - 60 ± 5°C for equilibrating dilution flasks.

7.19 Magnetic stirrer.

7.20 Heating mantel.

7.21 Variac

7.22 Soxhlet extraction apparatus and glass thimbles - for purifying Tenax.

7.23 Infrared lamp - for drying Tenax.

7.24 GC column - SE-30 or alternative coating, glass capillary or fused silica.

7.25 Psychrometer - to determine ambient relative humidity. (optional).

8. Reagents and Materials

8.1 Empty Tenax cartridges - glass or stainless steel (See Figure 1a or b).
8.2 Tenax 60/80 mesh (2,6-diphenylphenylene oxide polymer).
8.3 Glasswool - silanized.
8.4 Acetone - Pesticide quality or equivalent.
8.5 Methanol - Pesticide quality, or equivalent.
8.6 Pentane - Pesticide quality or equivalent.
8.7 Helium - Ultra pure, compressed gas. (99.9999%)
8.8 Nitrogen - Ultra pure, compressed gas. (99.9999%)
8.9 Liquid nitrogen.
8.10 Polyester gloves - for handling glass Tenax cartridges.
8.11 Glass Fiber Filter - one inch diameter, to fit in filter holder. (optional)
8.12 Perfluorotributylamine (FC-43).
8.13 Chemical Standards - Neat compounds of interest. Highest purity available.
8.14 Granular activated charcoal - for preventing contamination of Tenax cartridges during storage.

9. Cartridge Construction and Preparation

9.1 Cartridge Design

9.1.1 Several cartridge designs have been reported in the literature (1-3). The most common (1) is shown in Figure 1a. This design minimizes contact of the sample with metal surfaces, which can lead to decomposition in certain cases. However, a disadvantage of this design is the need to rigorously avoid contamination of the <u>outside</u> portion of the cartridge since the entire surface is subjected to the purge gas stream during the desorption porcess.

Clean polyester gloves must be worn at all times when handling such cartridges and exposure of the open cartridge to ambient air must be minimized.

9.1.2 A second common type of design (3) is shown in Figure 1b. While this design uses a metal (stainless steel) construction, it eliminates the need to avoid direct contact with the exterior surface since only the interior of the cartridge is purged.

9.1.3 The thermal desorption module and sampling system must be selected to be compatible with the particular cartridge design chosen. Typical module designs are shown in Figures 2a and b. These designs are suitable for the cartridge designs shown in Figures 1a and 1b, respectively.

9.2 Tenax Purification

9.2.1 Prior to use the Tenax resin is subjected to a series of solvent extraction and thermal treatment steps. The operation should be conducted in an area where levels of volatile organic compounds (other than the extraction solvents used) are minimized.

9.2.2 All glassware used in Tenax purification as well as cartridge materials should be thoroughly cleaned by water rinsing followed by an acetone rinse and dried in an oven at 250°C.

9.2.3 Bulk Tenax is placed in a glass extraction thimble and held in place with a plug of clean glasswool. The resin is then placed in the soxhlet extraction apparatus and extracted sequentially with methanol and then pentane for 16-24 hours (each solvent) at approximately 6 cycles/hour. Glasswool for cartidge preparation should be cleaned in the same manner as Tenax.

9.2.4 The extracted Tenax is immediately placed in an open glass dish and heated under an infrared lamp for two

hours in a hood. Care must be exercised to avoid over heating of the Tenax by the infrared lamp. The Tenax is then placed in a vacuum oven (evacuated using a water aspirator) without heating for one hour. An inert gas (helium or nitrogen) purge of 2-3 ml/minute is used to aid in the removal of solvent vapors. The oven temperature is then increased to 110°C, maintaining inert gas flow and held for one hour. The oven temperature control is then shut off and the oven is allowed to cool to room temperature. Prior to opening the oven, the oven is slightly pressurized with nitrogen to prevent contamination with ambient air. The Tenax is removed from the oven and sieved through a 40/60 mesh sieve (acetone rinsed and oven dried) into a clean glass vessel. If the Tenax is not to be used immediately for cartridge preparation it should be stored in a clean glass jar having a Teflon-lined screw cap and placed in a desiccator.

9.3 Cartridge Preparation and Pretreatment

9.3.1 All cartridge materials are pre-cleaned as described in Section 9.2.2. If the glass cartridge design shown in Figure 1a is employed all handling should be conducted wearing polyester gloves.

9.3.2 The cartridge is packed by placing a 0.5-1cm glass-wool plug in the base of the cartridge and then filling the cartridge to within approximately 1 cm of the top. A 0.5-1cm glasswool plug is placed in the top of the cartridge.

9.3.3 The cartridges are then thermally conditioned by heating for four hours at 270°C under an inert gas (helium) purge (100 - 200 ml/min).

9.3.4 After the four hour heating period the cartridges are allowed to cool. Cartridges of the type shown in Figure 1a are immediately placed (without cooling) in clean culture tubes having Teflon-lined screw caps with a glasswool cushion at both the top and the bottom. Each tube should be shaken to ensure that the cartridge is held firmly in place. Cartridges of the type shown in Figure 1b are allowed to cool to room temperature under inert gas purge and are then closed with stainless steel plugs.

9.3.5 The cartridges are labeled and placed in a tightly sealed metal can (e.g. paint can or similar friction top container). For cartridges of the type shown in Figure 1a the culture tube, not the cartridge,is labeled.

9.3.6 Cartridges should be used for sampling within 2 weeks after preparation and analyzed within two weeks after sampling. If possible the cartridges should be stored at -20°C in a clean freezer (i.e. no solvent extracts or other sources of volatile organics contained in the freezer).

10. Sampling

10.1 Flow rate and Total Volume Selection

10.1.1 Each compound has a characteristic retention volume (liters of air per gram of adsorbent) which must not be exceeded. Since the retention volume is a function of temperature, and possibly other sampling variables, one must include an adequate margin of safety to ensure good collection efficiency. Some considerations and guidance in this regard are provided in a recent report (5). Approximate breakthrough volumes at 38°C (100°F) in liters/gram of Tenax are provided in Table 1. These retention volume data are supplied only as rough guidance and are subject to considerable variability, depending on cartridge design as well as sampling parameters and atmospheric conditions.

10.1.2 To calculate the maximum total volume of air which can be sampled use the following equation:

$$V_{MAX} = \frac{V_b x W}{1.5}$$

where

V_{MAX} is the calculated maximum total volume in liters.

V_b is the breakthrough volume for the least retained compound of interest (Table 1) in liters per gram of Tenax.

W is the weight of Tenax in the cartridge, in grams.

1.5 is a dimensionless safety factor to allow for variability in atmospheric conditions. This factor is appropriate for temperatures in the range of 25-30°C. If higher temperatures are encountered the factor should be increased (i.e. maximum total volume decreased).

10.1.3 To calculate maximum flow rate use the following equation:

$$Q_{MAX} = \frac{V_{MAX}}{t} \times 1000$$

where

Q_{MAX} is the calculated maximum flow rate in millileters per minute.

t is the desired sampling time in minutes. Times greater than 24 hours (1440 minutes) generally are unsuitable because the flow rate required is too low to be accurately maintained.

10.1.4 The maximum flow rate Q_{MAX} should yield a linear flow velocity of 50-500 cm/minute. Calculate the linear velocity corresponding to the maximum flow rate using the following equation:

$$B = \frac{Q_{MAX}}{\pi r^2}$$

where

B is the calculated linear flow velocity in centimeters per minute.

r is the internal radius of the cartridge in centimeters.

If B is greater than 500 centimeters per minute either the total sample volume (V_{MAX}) should be reduced or the sample flow rate (Q_{MAX}) should be reduced by increasing the collection time. If B is less than 50 centimeters per minute the sampling rate (Q_{MAX}) should be increased by reducing the sampling time. The total sample value (V_{MAX}) cannot be increased due to component breakthrough.

10.1.4 The flow rate calculated as described above defines the maximum flow rate allowed. In general, one should collect additional samples in parallel, for the same time period but at lower flow rates. This practice yields a measure of quality control and is further discussed in the literature (5). In general, flow rates 2 to 4 fold lower than the maximum flow rate should be employed for the parallel samples. In all cases a constant flow rate should be achieved for each cartridge since accurate integration of the analyte concentration requires that the flow be constant over the sampling period.

10.2 Sample Collection

10.2.1 Collection of an accurately known volume of air is critical to the accuracy of the results. For this reason the use of mass flow controllers, rather than conventional needle valves or orifices is highly recommended, especially at low flow velocities (e.g. less than 100 milliliters/minute). Figure 3a illustrates a sampling system utilizing mass flow controllers. This system readily allows for collection of parallel samples. Figures 3b shows a commercially available system based on needle valve flow controllers.

10.2.2 Prior to sample collection insure that the sampling flow rate has been calibrated over a range including the rate to be used for sampling, with a "dummy" Tenax cartridge in place. Generally calibration is accomplished using a soap bubble flow meter or calibrated wet test meter. The flow calibration device is connected to the flow exit, assuming the entire flow system is sealed. ASTM Method D3686 describes an appropriate calibration scheme, not requiring a sealed flow system downstream of the pump.

10.2.3 The flow rate should be checked before and after each sample collection. If the sampling interval exceeds four hours the flow rate should be checked at an intermediate point during sampling as well. In general, a rotameter should be included, as showed in Figure 3b, to allow observation of the sampling flow rate without disrupting the sampling process.

10.2.4 To collect an air sample the cartridges are removed from the sealed container just prior to initiation of the collection process. If glass cartridges (Figure 1a) are employed they must be handled only with polyester gloves and should not contact any other surfaces.

10.2.5 A particulate filter and holder are placed on the inlet to the cartridges and the exit end of the cartridge is connected to the sampling apparatus. In many sampling situations the use of a filter is not necessary if only the total concentration of a component is desired. Glass cartridges of the type shown in Figure 1a are connected using teflon ferrules and Swagelok (stainless steel or teflon) fittings. Start the pump and record the following parameters on an appropriate data sheet (Figure 4): data, sampling location, time, ambient temperature, barometric

pressure, relative humidity, dry gas meter reading (if applicable) flow rate, rotameter reading (if applicable), cartridge number and dry gas meter serial number.

10.2.6 Allow the sampler to operate for the desired time, periodically recording the variables listed above. Check flow rate at the midpoint of the sampling interval if longer than four hours.
At the end of the sampling period record the parameters listed in 10.2.5 and check the flow rate and record the value. If the flows at the beginning and end of the sampling period differ by more than 10% the cartridge should be marked as suspect.

10.2.7 Remove the cartridges (one at a time) and place in the original container (use gloves for glass cartridges). Seal the cartridges or culture tubes in the friction-top can containing a layer of charcoal and package for immediate shipment to the laboratory for analysis. Store cartridges at reduced temperature (e.g. - 20°C) before analysis if possible to maximize storage stability.

10.2.8 Calculate and record the average sample rate for each cartridge according to the following equation:

$$Q_A = \frac{Q_1 + Q_2 + \ldots . Q_N}{N}$$

where

Q_A = Average flow rate in ml/minute.
$Q_1, Q_2, \ldots . Q_N$ = Flow rates determined at beginning, end, and immediate points during sampling.

N = Number of points averaged.

10.2.9 Calculate and record the total volumetric flow for each cartridge using the following equation:

$$V_m = \frac{T \times Q_A}{1000}$$

where

V_m = Total volume sampled in liters at measured temperature and pressure.

T_2 = Stop time.

T_1 = Start time.

T = Sampling time = $T_2 - T_1$, minutes

10.2.10 The total volume (V_s) at standard conditions, 25°C and 760 mmHg, is calculated from the following equation:

$$V_s = V_m \times \frac{P_A}{760} \times \frac{298}{273 + t_A}$$

where

P_A = Average barometric pressure, mmHg

t_A = Average ambient temperature, °C.

11. GC/MS Analysis

11.1 Instrument Set-up

11.1.1 Considerable variation from one laboratory to another is expected in terms of instrument configuration. Therefore each laboratory must be responsible for verifying that their particular system yields satisfactory results. Section 14 discusses specific performance criteria which should be met.

11.1.2 A block diagram of the typical GC/MS system required for analysis of Tenax cartridges is depicted in Figure 5. The operation of such devices is described in 11.2.4. The thermal desorption module must be designed to accommodate the particular cartridge configuration. Exposure of the sample to metal surfaces should be minimized and only stainless steel, or nickel metal surfaces should be employed.

The volume of tubing and fittings leading from the cartridge to the GC column must be minimized and all areas must be well-swept by helium carrier gas.

11.1.3 The GC column inlet should be capable of being cooled to -70°C and subsequently increased rapidly to approximately 30°C. This can be most readily accomplished using a GC equipped with subambient cooling capability (liquid nitrogen) although other approaches such as manually cooling the inlet of the column in liquid nitrogen may be acceptable.

11.1.4 The specific GC column and temperature program employed will be dependent on the specific compounds of interest. Appropriate conditions are described in the literature (1-3). In general a nonpolar stationary phase (e.g. SE-30, OV-1) temperature programmed from 30°C to 200°C at 8°/minute will be suitable. Fused silica bonded phase columns are preferable to glass columns since they are more rugged and can be inserted directly into the MS ion source, thereby eliminating the need for a GC/MS transfer line.

11.1.5 Capillary column dimensions of 0.3 mm ID and 50 meters long are generally appropriate although shorter lengths may be sufficient in many cases.

11.1.6 Prior to instrument calibration or sample analysis the GC/MS system is assembled as shown in Figure 5. Helium purge flows (through the cartridge) and carrier flow are set at approximately 10 ml/minute and 1-2 ml/minute respectively. If applicable, the injector sweep flow is set at 2-4 ml/minute.

11.1.7 Once the column and other system components are assembled and the various flows established the column temperature is increased to 250°C for approximately four hours (or overnight if desired) to condition the column.

11.1.8 The MS and data system are set according to the manufacturer's instructions. Electron impact ionization (70eV) and an electron multiplier gain of approximately 5×10^4 should be employed. Once the entire GC/MS system has been setup the system is calibrated as described in Section 11.2. The user should prepare a detailed standard operating procedure (SOP) describing this process for the particular instrument being used.

11.2 Instrument Calibration

11.2.1 Tuning and mass standarization of the MS system is performed according to manufacturer's instructions and relevant information from the user prepared SOP. Bromofluorobenzene (BFB) will be employed for this purpose. The material is introduced directly into the ion source through a molecular leak. The instrumental parameters (e.g. lens voltages, resolution, etc.) should be adjusted to give the relative ion abundances shown in Table 2 as well as acceptable resolution and peak shape. If these approximate relative abundances cannot be achieved, the ion source may require cleaning according to manufacturer's instructions. In the event that the user's instrument cannot achieve these relative ion abundances, but is otherwise operating properly, the user may adopt another set of relative abundances as performance criteria.

However, these alternate values must be repeatable on a day-to-day basis.

11.2.2 After the mass standarization and tuning process has been completed and the appropriate values entered into the data system the user should then calibrate the entire system by introducing known quantities of the standard components of interest into the system. Three alternate procedures may be employed for the calibration process including 1) direct syringe injection of dilute vapor phase standards, prepared in a dilution bottle, onto the GC column, 2) Injection of dilute vapor phase standards into a carrier gas stream directed through the Tenax cartridge, and 3) introduction of permeation or diffusion tube standards onto a Tenax cartridge. The standards preparation procedures for each of these approaches are described in Section 13. The following paragraphs describe the instrument calibration process for each of these approaches.

11.2.3 If the instrument is to be calibrated by direct injection of a gaseous standard, a standard is prepared in a dilution bottle as described in Section 13.1. The GC column is cooled to -70°C (or, alternately, a portion of the column inlet is manually cooled with liquid nitrogen). The MS and data system is set up for acquisition as described in the relevant user SOP. The ionization filament should be turned off during the initial 2-3 minutes of the run to allow oxygen and other highly volatile components to elute. An appropriate volume (less than 1 ml) of the gaseous standard is injected onto the GC system using an accurately calibrated gas tight syringe.

The system clock is started and the column is maintained at -70°C (or liquid nitrogen inlet cooling) for 2 minutes. The column temperature is rapidly increased to the desired initial temperature (e.g. 30°C). The temperature program is started at a consistent time (e.g. four minutes) after injection. Simultaneously the ionization filament is turned on and data acquisition is initiated. After the last component of interest has eluted acquisiton is terminated and the data is processed as described in Section 11.2.5. The standard injection process is repeated using different standard volumes as desired.

11.2.4 If the system is to be calibrated by analysis of spiked Tenax cartridges a set of cartridges is prepared as described in Sections 13.2 or 13.3. Prior to analysis the cartridges are stored as described in Section 9.3. If glass cartridges (Figure 1a) are employed care must be taken to avoid direct contact, as described earlier. The GC column is cooled to -70°C, the collection loop is immersed in liquid nitrogen and the desorption module is maintained at 250°C. The inlet valve is placed in the desorb mode and the standard cartridge is placed in the desorption module, making certain that no leakage of purge gas occurs. The cartridge is purged for 10 minutes and then the inlet valve is placed in the inject mode and the liquid nitrogen source removed from the collection trap. The GC column is maintained at -70°C for two minutes and subsequent steps are as described in 11.2.3. After the process is complete the cartridge is removed from the desorption module and stored for subsequent use as described in Section 9.3.

11.2.5 Data processing for instrument calibration involves determining retention times, and integrated characteristic ion intensities for each of the compounds of interest. In addition, for at least one chromatographic run, the individual mass spectra should be inspected and compared to reference spectra to ensure proper instrumental performance. Since the steps involved in data processing are highly instrument specific, the user should prepare a SOP describing the process for individual use. Overall performance criteria for instrument calibration are provided in Section 14. If these criteria are not achieved the user should refine the instrumental parameters and/or operating procedures to meet these criteria.

11.3 Sample Analysis

11.3.1 The sample analysis process is identical to that described in Section 11.2.4 for the analysis of standard Tenax cartridges.

11.3.2 Data processing for sample data generally involves 1) qualitatively determining the presence or absence of each component of interest on the basis of a set of characteristic ions and the retention time using a reverse-search software routine, 2) quantification of each identified component by integrating the intensity of a characteristic ion and comparing the value to that of the calibration standard, and 3) tentative identification of other components observed using a forward (library) search software routine. As for other user specific processes, a SOP should be prepared describing the specific operations for each individual laboratory.

12. Calculations

12.1 Calibration Response Factors

12.1.1 Data from calibration standards is used to calculate a response factor for each component of interest. Ideally the process involves analysis of at least three calibration levels of each component during a given day and determination of the response factor (area/nanogram injected) from the linear least squares fit of a plot of nanograms injected versus area (for the characteristic ion). In general quantities of component greater than 1000 nanograms should not be injected because of column overloading and/or MS response nonlinearity.

12.1.2 In practice the daily routine may not always allow analysis of three such calibration standards. In this situation calibration data from consecutive days may be pooled to yield a response factor, provided that analysis of replicate standards of the same concentration are shown to agree within 20% on the consecutive days. One standard concentration, near the midpoint of the analytical range of interest, should be chosen for injection every day to determine day-to-day response reproducibility.

12.1.3 If substantial nonlinearity is present in the calibration curve a nonlinear least squares fit (e.g. quadratic) should be employed. This process involves fitting the data to the following equation:

$$Y = A + BX + CX^2$$

where

Y = peak area

X = quantity of component, nanograms

A,B, and C are coefficients in the equation

12.2 Analyte Concentrations

12.2.1 Analyte quantities on a sample cartridge are calculated from the following equation:

$$Y_A = A + BX_A + CX_A$$

where

Y_A is the area of the analyte characteristic ion for the sample cartridge.

X_A is the calculated quantity of analyte on the sample cartridge, in nanograms.

A,B, and C are the coefficients calculated from the calibration curve described in Section 12.1.3.

12.2.2 If instrumental response is essentially linear over the concentration range of interest a linear equation (C=0 in the equation above) can be employed.

12.2.3 Concentration of analyte in the original air sample is calculated from the following equation:

$$C_A = \frac{X_A}{V_S}$$

where

C_A is the calculated concentration of analyte in nanograms per liter.

V_S and X_A are as previously defined in Section 10.2.10 and 12.2.1, respectively.

13. Standard Preparation

13.1 Direct Injection

13.1.1 This process involves preparation of a dilution bottle containing the desired concentrations of compounds of interest for direct injection onto the GC/MS system.

13.1.2 Fifteen three-millimeter diameter glass beads and a one-inch Teflon stirbar are placed in a clean two-liter glass septum capped bottle and the exact volume is determined by weighing the bottle before and after filling with deionized water. The bottle is then rinsed with acetone and dried at 200°C.

13.1.3 The amount of each standard to be injected into the vessel is calculated from the desired injection quantity and volume using the following equation:

$$W_T = \frac{W_I}{V_I} \times V_B$$

where

W_T is the total quantity of analyte to be injected into the bottle in milligrams

W_I is the desired weight of analyte to be injected onto the GC/MS system or spiked cartridge in nanograms

V_I is the desired GC/MS or cartridge injection volume (should not exceed 500) in microliters.

V_B is total volume of dilution bottle determined in 13.1.1, in liters.

13.1.4 The volume of the neat standard to be injected into the dilution bottle is determined using the following equation:

$$V_T = \frac{W_T}{d}$$

where

V_T is the total volume of neat liquid to be injected in microliters.

d is the density of the neat standard in grams per milliliter.

13.1.6 The bottle is placed in a 60°C oven for at least 30 minutes prior to removal of a vapor phase standard.

13.1.7 To withdraw a standard for GC/MS injection the bottle is removed from the oven and stirred for 10-15 seconds. A suitable gas-tight microber syring warmed to 60°C, is inserted through the septum cap and pumped three times slowly. The appropriate volume of sample (approximately 25% larger than the desired injection volume) is drawn into the syringe and the volume is adjusted to the exact value desired and then immediately injected over a 5-10 seconds period onto the GC/MS system as described in Section 11.2.3.

13.2 Preparation of Spiked Cartridges by Vapor Phase Injection

13.2.1 This process involves preparation of a dilution bottle containing the desired concentrations of the compound(s) of interest as described in 13.1 and injecting the desired volume of vapor into a flowing inert gas stream directed through a clean Tenax cartridge.

13.2.2 A helium purge system is assembled wherein the helium flow 20-30 mL/minute is passed through a stainless steel Tee fitted with a septum injector. The clean Tenax cartridge is connected downstream of the tee using appropriate Swagelok fittings. Once the cartridge is placed in the flowing gas stream the appropriate volume vapor standard, in the dilution bottle, is injected through the septum as described in 13.1.6. The syringe is flushed several times by alternately filling the syringe with carrier gas and displacing the contents into the flow stream, without removing the syringe from the septum. Carrier flow is maintain through the cartridge for approximately 5 minutes after injection.

13.3 Preparation of Spiked Traps Using Permeation or Diffusion tubes

13.3.1 A flowing stream of inert gas containing known amounts of each compound of interest is generated according to ASTM Method D3609(6). Note that a method of accuracy maintaining temperature within ± 0.1°C is required and the system generally must be equilibrated for at least 48 hours before use.

13.3.2 An accurately known volume of the standard gas stream (usually 0.1-1 liter) is drawn through a clean Tenax cartridge using the sampling system described in Section 10.2.1, or a similar system. However, if mass flow controllers are employed they must be calibrated for the carrier gas used in Section 13.3.1 (usually nitrogen). Use of air as the carrier gas for permeation systems is not recommended, unless the compounds of interest are known to be highly stable in air.

13.3.3 The spiked cartridges are then stored or immediately analyzed as in Section 11.2.4.

14. Performance Criteria and Quality Assurance

This section summarizes quality assurance (QA) measures and provides guidance concerning performance criteria which should be achieved within each laboratory. In many cases the specific QA procedures have been described within the appropriate section describing the particular activity (e.g. parallel sampling).

14.1 Standard Opreating Procedures (SOPs)

14.1.1 Each user should generate SOPs describing the following activities as they are performed in their laboratory:

1) assembly, calibration, and operation of the sampling system,
2) preparation, handling and storage of Tenax cartridges,
3) assembly and operation of GC/MS system including the thermal desorption apparatus and data system, and
4) all aspects of data recording and processing.

14.1.2 SOPs should provide specific stepwise instructions and should be readily available to, and understood by the laboratory personnel conducting the work.

14.2 Tenax Cartridge Preparation

14.2.1 Each batch of Tenax cartridges prepared (as described in Section 9) should be checked for contamination by analyzing one cartridge immediately after preparation. While analysis can be accomplished by GC/MS, many laboratories may chose to use GC/FID due to logistical and cost considerations.

14.2.2 Analysis by GC/FID is accomplished as described for GC/MS (Section 11) except for use of FID detection.

14.2.3 While acceptance criteria can vary depending on the components of interest, at a minimum the clean cartridge should be demonstrated to contain less than one fourth of the minimum level of interest for each component. For most compounds the blank level should be less than 10 nanograms per cartridge in order to be acceptable. More rigid criteria may be adopted, if necessary, within a specific laboratory. If a cartridge does not meet these acceptance criteria the entire lot should be rejected.

14.3 Sample Collection

14.3.1 During each sampling event at least one clean cartridge will accompany the samples to the field and back to the laboratory, without being used for sampling, to serve as a field blank. The average amount of material found on the field blank cartridge may be subtracted from the amount found on the actual samples. However, if the blank level is greater than 25% of the sample amount, data for that component must be identified as suspect.

14.3.2 During each sampling event at least one set of parallel samples (two or more samples collected simultaneously) will be collected, preferably at different flow rates as described in Section 10.1. If agreement between parallel samples is not generally within ± 25% the user should collect parallel samples on a much more frequent basis (perhaps for all sampling points). If a trend of lower apparent concentrations with increasing flow rate is observed for a set

of parallel samples one should consider using a reduced flow rate and longer sampling interval if possible. If this practice does not improve the reproducibility further evaluation of the method performance for the compound of interest may be required.

14.3.3 Backup cartridges (two cartridges in series) should be collected with each sampling event. Backup cartridges should contain less than 20% of the amount of components of interest found in the front cartridges, or be equivalent to the blank cartridge level, whichever is greater. The frequency of use of backup cartridges should be increased if increased flow rate is shown to yield reduced component levels for parallel sampling. This practice will help to identify problems arising from breakthrough of the component of interest during sampling.

14.4 GC/MS Analysis

14.4.1 Performance criteria for MS tuning and mass calibration have been discussed in Section 11.2 and Table 2. Additional criteria may be used by the laboratory if desired. The following sections provide performance guidance and suggested criteria for determining the acceptability of the GC/MS system.

14.4.2 Chromatographic efficiency should be evaluated using spiked Tenax cartridges since this practice tests the entire system. In general a reference compound such as perfluorotoluene should be spiked onto a cartridge at the 100 nanogram level as described in Section 13.2 or 13.3. The cartridge is then analyzed by GC/MS as

described in Section 11.4. The perfluorotoluene (or other reference compound) peak is then plotted on an expanded time scale so that its width at 10% of the peak can be calculated, as shown in Figure 6. The width of the peak at 10% height should not exceed 10 seconds. More stringent criteria may be required for certain applications. The assymmetry factor (See Figure 6) should be between 0.8 and 2.0. The assymmetry factor for any polar or reactive compounds should be determined using the process described above. If peaks are observed that exceed the peak width or assymmetry factor criteria above, one should inspect the entire system to determine if unswept zones or cold spots are present in any of the fittings and is necessary. Some laboratories may chose to evaluate column performance separately by direct injection of a test mixture onto the GC column. Suitable schemes for column evaluation have been reported in the literature (7). Such schemes cannot be conducted by placing the substances onto Tenax because many of the compounds (e.g. acids, bases, alcohols) contained in the test mix are not retained, or degrade, on Tenax.

14.4.3 The system detection limit for each component is calculated from the data obtained for calibration standards. The detection limit is defined as

$$DL = A + 3.3S$$

where

DL is the calculated detection limit in nanograms injected.

A is the intercept calculated in Section 12.1.1 or 12.1.3.

S is the standard deviation of replicate determinations of the lowest level standard (at least three such determinations are required.

In general the detection limit should be 20 nanograms or less and for many applications detection limits of 1-5 nanograms may be required. The lowest level standard should yield a signal to noise ratio, from the total ion current response, of approximately 5.

14.4.4 The relative standard deviation for replicate analyses of cartridges spiked at approximately 10 times the detection limit should be 20% or less. Day to day relative standard deviation should be 25% or less.

14.4.5 A useful performance evaluation step is the use of an internal standard to track system performance. This is accomplished by spiking each cartridge, including blank, sample, and calibration cartridges with approximately 100 nanograms of a compound not generally present in ambient air (e.g. perfluorotoluene). The integrated ion intensity for this compound helps to identify problems with a specific sample. In general the user should calculate the standard deviation of the internal standard response for a given set of samples analyzed under identical tuning and calibration conditions. Any sample giving a value greater than $\pm$ 2 standard deviations from the mean (calculated

excluding that particular sample) should be identified as suspect. Any marked change in internal standard response may indicate a need for instrument recalibration.

REFERENCES

1. Krost, K. J., Pellizzari, E. D., Walburn, S. G., and Hubbard, S. A., "Collection and Analysis of Hazardous Organic Emissions", Analytical Chemistry, 54, 810-817, 1982.

2. Pellizzari, E. O. and Bunch, J. E., "Ambient Air Carcinogenic Vapors-Improved Sampling and Analytical Techniques and Field Studies", EPA-600/2-79-081, U.S. Environmental Protection Agency, Research Triangle Park, North Carolina, 1979.

3. Kebbekus, B. B. and Bozzelli, J. W., "Collection and Analysis of Selected Volatile Organic Compounds in Ambient Air", Proc. Air Pollution Control Assoc., Paper No. 82-65.2. Air Poll. Control Assoc., Pittsburgh, Pennsylvania, 1982.

4. Riggin, R. M., "Technical Assistance Document for Sampling and Analysis of Toxic Organic Compounds in Ambient Air", EPA-600/4-83-027, U.S. Environmental Protection Agency, Research Triangle Park, North Carolina, 1983.

5. Walling, J. F., Berkley, R. E., Swanson, D. H., and Toth, F. J. "Sampling Air for Gaseous Organic Chemical-Applications to Tenax", EPA-600/7-54-82-059, U.S. Environmental Protection Agency, Research Triangle Park, North Carolina, 1982.

6. Annual Book of ASTM Standards, Part 11.03, "Atmospheric Analysis", American Society for Testing and Material, Philadelphia, Pennsylvania.

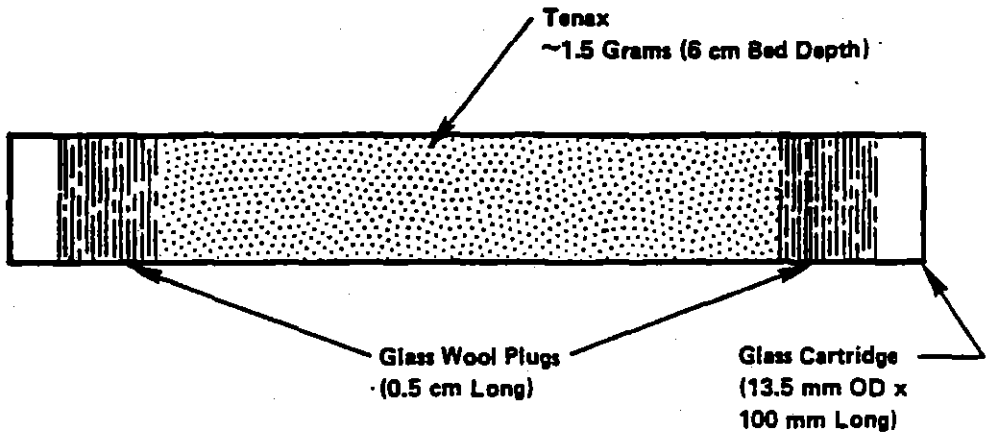
7. Grob, K., Jr., Grob, G., and Grob, K., "Comprehensive Standardized Quality Test for Glass Capillary Columns", J. Chromatog., 156, 1-20, 1978.

TABLE 1. RETENTION VOLUME ESTIMATES FOR COMPOUNDS ON TENAX

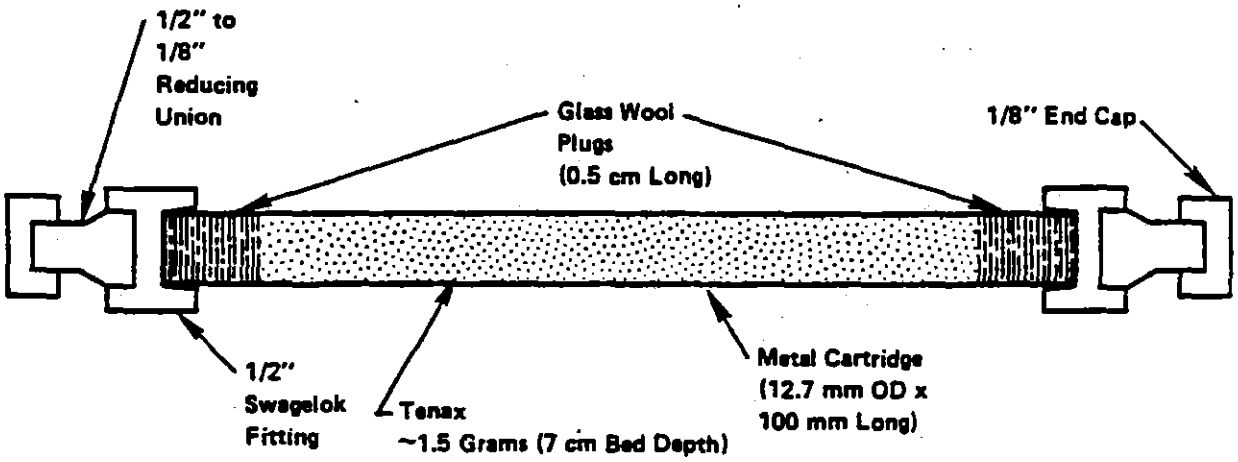
COMPOUND	ESTIMATED RETENTION VOLUME AT 100°F (38°C)-LITERS/GRAM
Benzene	19
Toluene	97
Ethyl Benzene	200
Xylene(s)	∿ 200
Cumene	440
n-Heptane	20
1-Heptene	40
Chloroform	8
Carbon Tetrachloride	8
1,2-Dichloroethane	10
1,1,1-Trichloroethane	6
Tetrachloroethylene	80
Trichloroethylene	20
1,2-Dichloropropane	30
1,3-Dichloropropane	90
Chlorobenzene	150
Bromoform	100
Ethylene Dibromide	60
Bromobenzene	300

TABLE 2. SUGGESTED PERFORMANCE CRITERIA FOR RELATIVE ION ABUNDANCES FROM FC-43 MASS CALIBRATION

M/E	% RELATIVE ABUNDANCE
51	1.8 ± 0.5
69	100
100	12.0 ± 1.5
119	12.0 ± 1.5
131	35.0 ± 3.5
169	3.0 ± 0.4
219	24.0 ± 2.5
264	3.7 ± 0.4
314	0.25 ± 0.1

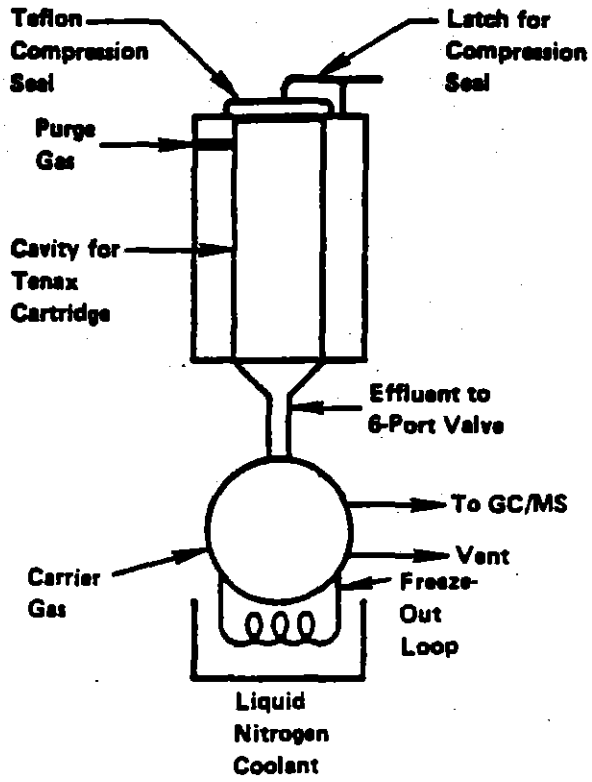


(a) Glass Cartridge

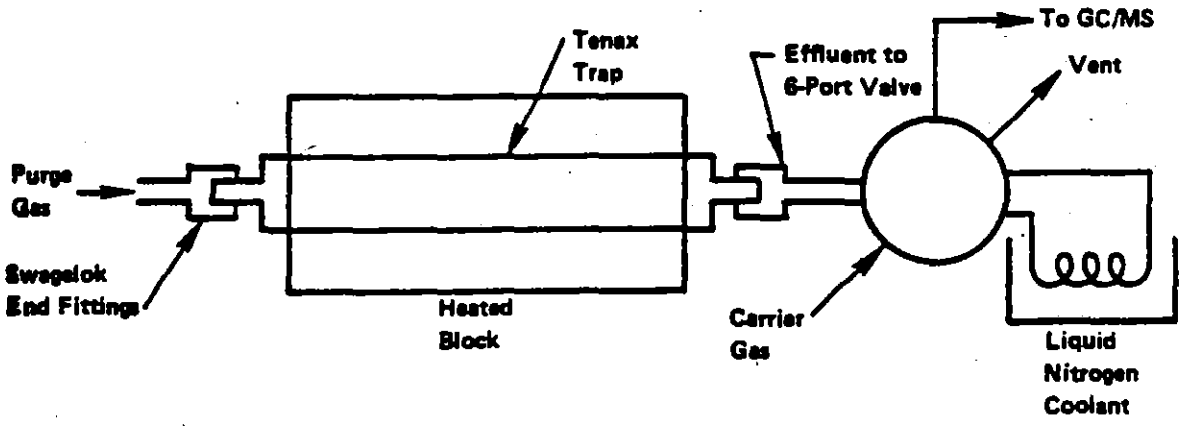


(b) Metal Cartridge

FIGURE 1. TENAX CARTRIDGE DESIGNS

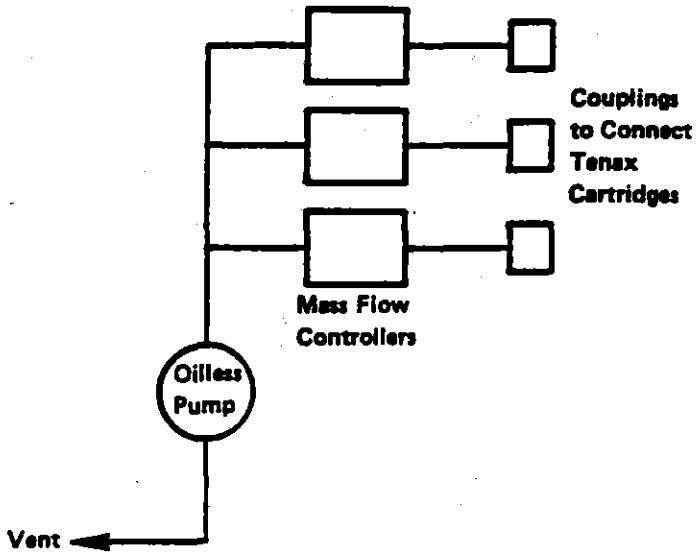


(a) Glass Cartridges (Compression Fit)

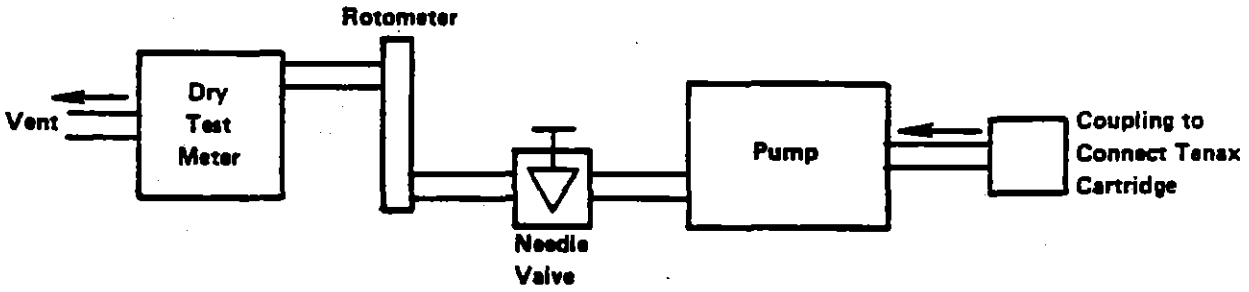


(b) Metal Cartridges (Swagelok Fittings)

FIGURE 2. TENAX CARTRIDGE DESORPTION MODULES



(a) Mass Flow Control



(b) Needle Valve Control

FIGURE 3. TYPICAL SAMPLING SYSTEM CONFIGURATIONS

SAMPLING DATA SHEET
(One Sample Per Data Sheet)

PROJECT: _____ DATE(S) SAMPLED: _____

SITE: _____ TIME PERIOD SAMPLED: _____

LOCATION: _____ OPERATOR: _____

INSTRUMENT MODEL NO: _____ CALIBRATED BY: _____

PUMP SERIAL NO: _____

SAMPLING DATA

Sample Number: _____

Start Time: _____ Stop Time: _____

Time	Dry Gas Meter Reading	Rotameter Reading	Flow Rate,*Q ml/Min	Ambient Temperature °C	Barometric Pressure, mmHg	Relative Humidity, %	Comments
1.							
3.							
4.							
N.							

Total Volume Data**

V_m = (Final - Initial) Dry Gas Meter Reading, or = _____ Liters

$$= \frac{Q_1 + Q_2 + Q_3 \ldots Q_N}{N} \times \frac{1}{1000 \times (\text{Sampling Time in Minutes})} = \_\_\_\_\_ \text{ Liters}$$

* Flowrate from rotameter or soap bubble calibrator (specify which).

** Use data from dry gas meter if available.

FIGURE 4. EXAMPLE SAMPLING DATA SHEET

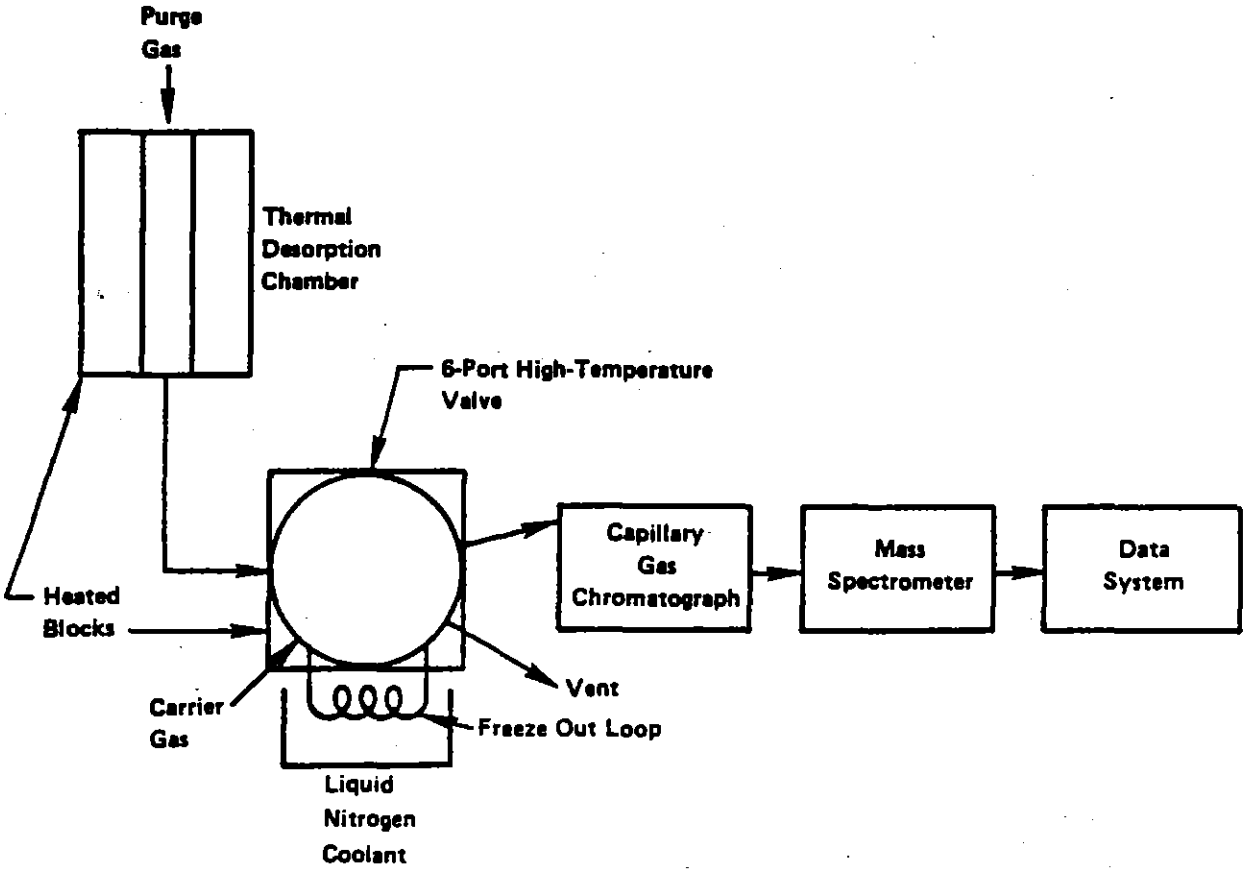
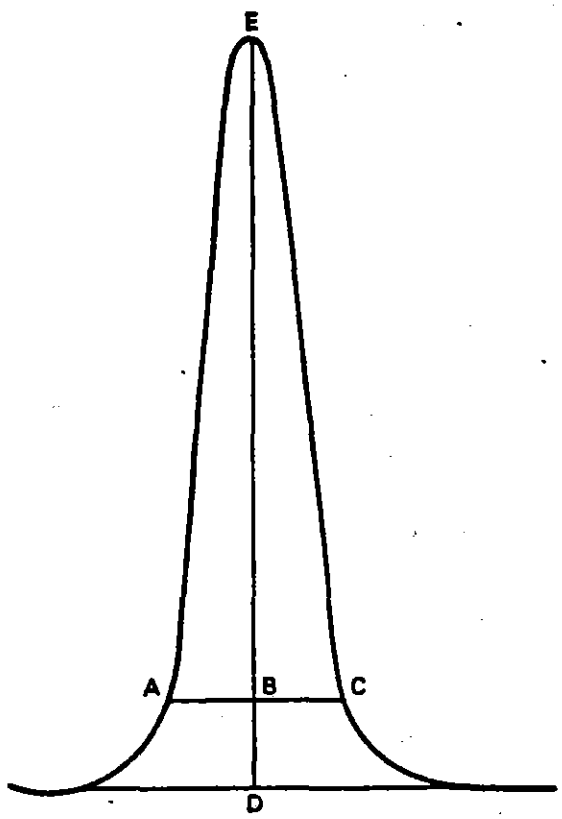


FIGURE 5. BLOCK DIAGRAM OF ANALYTICAL SYSTEM



$$\text{Asymmetry Factor} = \frac{BC}{AB}$$

Example Calculation:

Peak Height = DE = 100 mm
10% Peak Height = BD = 10 mm
Peak Width at 10% Peak Height = AC = 23 mm
AB = 11 mm
BC = 12 mm

Therefore: Asymmetry Factor = $\frac{12}{11}$ = 1.1

FIGURE 6. PEAK ASYMMETRY CALCULATION

METHOD TO2

Revision 1.0
April, 1984

METHOD FOR THE DETERMINATION OF VOLATILE ORGANIC COMPOUNDS IN AMBIENT AIR BY CARBON MOLECULAR SIEVE ADSORPTION AND GAS CHROMATOGRAPHY/MASS SPECTROMETRY (GC/MS)

1. Scope

1.1 This document describes a procedure for collection and determination of selected volatile organic compounds which can be captured on carbon molecular sieve (CMS) adsorbents and determined by thermal desorption GC/MS techniques.

1.2 Compounds which can be determined by this method are nonpolar and nonreactive organics having boiling points in the range -15 to +120°C. However, not all compounds meeting these criteria can be determined. Compounds for which the performance of the method has been documented are listed in Table 1. The method may be extended to other compounds but additional validation by the user is required. This method has been extensively used in a single laboratory. Consequently, its general applicability has not been thoroughly documented.

2. Applicable Documents

2.1 ASTM Standards

D 1356 Definitions of Terms Related to Atmospheric Sampling and Analysis.

E 355 Recommended Practice for Gas Chromatography Terms and Relationships.

2.2 Other Documents

Ambient Air Studies (1,2).

U.S. EPA Technical Assistance Document (3).

3. Summary of Method

3.1 Ambient air is drawn through a cartridge containing ~0.4 of a carbon molecular sieve (CMS) adsorbent. Volatile organic compounds are captured on the adsorbent while major inorganic atmospheric constituents pass through (or are only partially retained). After sampling, the cartridge is returned to the laboratory for analysis.

3.2 Prior to analysis the cartridge is purged with 2-3 liters of pure, dry air (in the same direction as sample flow) to remove adsorbed moisture.

3.3 For analysis the cartridge is heated to 350°-400°C, under helium purge and the desorbed organic compounds are collected in a specially designed cryogenic trap. The collected organics are then flash evaporated onto a capillary column GC/MS system (held at -70°C). The individual components are identified and quantified during a temperature programmed chromatographic run.

3.4 Due to the complexity of ambient air samples, only high resolution (capillary column) GC techniques are acceptable for most applications of the method.

4. Significance

4.1 Volatile organic compounds are emitted into the atmosphere from a variety of sources including industrial and commercial facilities, hazardous waste storage and treatment facilities, etc. Many of these compounds are toxic; hence knowledge of the concentration of such materials in the ambient atmosphere is required in order to determine human health impacts.

4.2 Traditionally air monitoring methods for volatile organic compounds have relied on carbon adsorption followed by solvent desorption and GC analysis. Unfortunately, such methods are not sufficiently sensitive for ambient air monitoring, in most cases, because only a small portion of

the sample is injected onto the GC system. Recently on-line thermal desorption methods, using organic polymeric adsorbents such as Tenax® GC, have been used for ambient air monitoring. The current method uses CMS adsorbents (e.g. Spherocarb®) to capture highly volatile organics (e.g. vinyl chloride) which are not collected on Tenax®. The use of on-line thermal desorption GC/MS yields a sensitive, specific analysis procedure.

5. Definitions

Definitions used in this document and any user prepared SOPs should be consistent with ASTM D1356 (4). All abbreviations and symbols are defined with this document at the point of use.

6. Interferences

6.1 Only compounds having a mass spectrum and GC retention time similar to the compound of interest will interfere in the method. The most commonly encountered interferences are structural isomers.

6.2 Contamination of the CMS cartridge with the compound(s) of interest can be a problem in the method. The user must be careful in the preparation, storage, and handling of the cartridges through the entire process to minimize contamination.

7. Apparatus

7.1 Gas Chromatograph/Mass Spectrometry system - must be capable of subambient temperature programming. Unit mass resolution to 800 amu. Capable of scanning 30-300 amu region every 0.5-0.8 seconds. Equipped with data system for instrument control as well as data acquisition, processing and storage.

7.2 Thermal Desorption Injection Unit - Designed to accommodate CMS cartridges in use (See Figure 3) and including cryogenic trap (Figure 5) and injection valve (Carle Model 5621 or equivalent).

7.3 Sampling System - Capable of accurately and precisely drawing an air flow of 10-500 ml/minute through the CMS cartridge. (See Figure 2a or b.)

7.4 Dewar flasks - 500 mL and 5 liter.

7.5 Stopwatches.

7.6 Various pressure regulators and valves - for connecting compressed gas cylinders to GC/MS system.

7.7 Calibration gas - In aluminum cylinder. Prepared by user or vendor. For GC/MS calibration.

7.8 High pressure apparatus for preparing calibration gas cylinders (if conducted by user). Alternatively, custom prepared gas mixtures can be purchased from gas supply vendors.

7.9 Friction top can (e.g. one-gallon paint can) - With layer of activated charcoal to hold clean CMS cartridges.

7.10 Thermometer - to record ambient temperature.

7.11 Barometer (optional).

7.12 Dilution bottle - Two-liter with septum cap for standard preparation.

7.13 Teflon stirbar - 1 inch long

7.14 Gas tight syringes - 10-500 µl for standard injection onto GC/MS system and CMS cartridges.

7.15 Liquid microliter syringes - 5-50 µL for injecting neat liquid standards into dilution bottle.

7.16 Oven - 60 ± 5°C for equilibrating dilution bottle.

7.17 Magnetic stirrer.

7.18 Variable voltage transformers - (120 V and 1000 VA) and electrical connectors (or temperature controllers) to heat cartridge and cryogenic loop.

7.19 Digital pyrometer - 30 to 500°C range.

7.20 Soap bubble flow meter - 1, 10 and 100 mL calibration points.

7.21 Copper tubing (1/8 inch) and fittings for gas inlet lines.

7.22 GC column - SE-30 or alternative coating, glass capillary or fused silica.

7.23 Psychrometer (optional).

7.24 Filter holder - stainless steel or aluminum (to accommodate 1 inch diameter filter). Other sizes may be used if desired. (optional)

8. Reagents and Materials

8.1 Empty CMS cartridges - Nickel or stainless steel (See Figure 1).

8.2 CMS Adsorbent, 60/80 mesh- Spherocarb® from Analabs Inc., or equivalent.

8.3 Glasswool - silanized.

8.4 Methylene chloride - pesticide quality, or equivalent.

8.5 Gas purifier cartridge for purge and GC carrier gas containing charcoal, molecular sieves, and a drying agent. Available from various chromatography supply houses.

8.6 Helium - Ultra pure, (99.9999%) compressed gas.

8.7 Nitrogen - Ultra pure, (99.9999%) compressed gas.

8.8 Liquid nitrogen or argon (50 liter dewar).

8.9 Compressed air, if required - for operation of GC oven door.

8.10 Perfluorotributylamine (FC-43) for GC/MS calibration.

8.11 Chemical Standards - Neat compounds of interest. Highest purity available.

9. Cartridge Construction and Preparation

9.1 A suitable cartridge design in shown in Figure 1. Alternate designs have been reported (1) and are acceptable, provided the user documents their performance. The design shown in Figure 1 has a built-in heater assembly. Many users may choose to replace this heater design with a suitable separate heating block or oven to simplify the cartridge design.

9.2 The cartridge is assembled as shown in Figure 1 using standard 0.25 inch O.D. tubing (stainless steel or nickel), 1/4 inch to 1/8 inch reducing unions, 1/8 inch nuts, ferrules, and endcaps. These parts are rinsed with methylene chloride and heated at 250°C for 1 hour prior to assembly.

9.3 The thermocouple bead is fixed to the cartridge body, and insulated with a layer of Teflon tape. The heater wire (constructed from a length of thermocouple wire) is wound around the length of the cartridge and wrapped with Teflon tape to secure the wire in place. The cartridge is then wrapped with woven silica fiber insulation (Zetex or equivalent). Finally the entire assembly is wrapped with fiber glass tape.

9.4 After assembly one end of the cartridge is marked with a serial number to designate the cartridge inlet during sample collection.

9.5 The cartridges are then packed with ~0.4 grams of CMS adsorbent. Glasswool plugs (~0.5 inches long) are placed at each end of the cartridge to hold the adsorbent firmly in place. Care must be taken to insure that no strands of glasswool extend outside the tubing, thus causing leakage in the compression endfittings. After loading the endfittings (reducing unions and end caps) are tightened onto the cartridge.

9.6 The cartridges are conditioned for initial use by heating at 400°C overnight (at least 16 hours) with a 100 mL/minute purge of pure nitrogen. Reused cartridges need only to be heated for 4 hours and should be reanalyzed before use to ensure complete desorption of impurities.

9.7 For cartridge conditioning ultra-pure nitrogen gas is passed through a gas purifier to remove oxygen,moisture and organic contaminants. The nitrogen supply is connected to the unmarked end of the cartridge and the flow adjusted to ∿50 mL/minute using a needle valve. The gas flow from the inlet (marked) end of the cartridge is vented to the atmosphere.

9.8 The cartridge thermocouple lead is connected to a pyrometer and the heater lead is connected to a variable voltage transformer (Variac) set at 0 V. The voltage on the Variac is increased to ∿15 V and adjusted over a 3-4 minute period to stabilize the cartridge temperature at 380-400°C.

9.9 After 10-16 hours of heating (for new cartridges) the Variac is turned off and the cartridge is allowed to cool to ∿30°C, under continuing nitrogen flow.

9.10 The exit end of the cartridge is capped and then the entire cartridge is removed from the flow line and the other endcap immediately installed. The cartridges are then placed in a metal friction top (paint) can containing ∿2 inches of granulated activated charcoal (to prevent contamination of the cartridges during storage) in the bottom, beneath a retaining screen. Clean paper tissues (e.g. Kimwipes) are placed in can to avoid damage to the cartridges during shipment.

9.11 Cartridges are stored in the metal can at all times except when in use. Adhesives initially present in the cartridge insulating materials are "burnt off" during initial conditioning. Therefore, unconditioned cartridges should not be placed in the metal can since they may contaminate the other cartridges.

9.12 Cartridges are conditioned within two weeks of use. A blank from each set of cartridges is analyzed prior to use in field

sampling. If an acceptable blank level is achieved, that batch of cartridges (including the cartridge serving as the blank) can be used for field sampling.

10. Sampling

10.1 Flow Rate and Total Volume Selection

10.1.1 Each compound has a characteristic retention volume (liters of air per unit weight of adsorbent). However, all of the compounds listed in Table 1 have retention volumes (at 37°C) in excess of 100 liters/cartridge (0.4 gram CMS cartridge) except vinyl chloride for which the value is ∿30 liters/cartridge. Consequently, if vinyl chloride or similarly volatile compounds are of concern the maximum allowable sampling volume is approximately 20 liters. If such highly volatile compounds are not of concern, samples as large as 100 liters can be collected.

10.1.2 To calculate the maximum allowable sampling flow rate the following equation can be used:

$$Q_{Max} = \frac{V_{Max}}{t} \times 1000$$

where

Q_{Max} is the calculated maximum sampling rate in mL/minute.

t is the desired sampling time in minutes.

V_{Max} is the maximum allowable total volume based on the discussion in 10.1.1.

10.1.3 For the cartridge design shown in Figure 1 Q_{Max} should be between 20 and 500 mL/minute. If Q_{Max} lies outside this range the sampling time or total sampling volume must be adjusted so that this criterion is achieved.

10.1.4 The flow rate calculated in 10.1.3 defines the <u>maximum</u> allowable flow rate. In general, the user should collect additional samples in parallel, at successive 2- to 4-fold lower flow rates. This practice serves as a quality control procedure to check on component breakthrough and related sampling and adsorption problems, and is further discussed in the literature (5).

10.2 Sample Collection

10.2.1 Collection of an accurately known volume of air is critical to the accuracy of the results. For this reason the use of mass flow controllers, rather than conventional needle valves or orifices is highly recommended, especially at low flow rates (e.g. less than 100 milliliters/minute). Figure 2a illustrates a sampling system based on mass flow controllers which readily allows for collection of parallel samples. Figure 2b shows a commercially available sampling system based on needle valve flow controllers.

10.2.2 Prior to sample collection the sampling flow rate is calibrated near the value used for sampling, with a "dummy" CMS cartridge in place. Generally calibration is accomplished using a soap bubble flow meter or calibrated wet test meter connected to the flow exit, assuming the entire flow system is sealed. ASTM Method D 3686 (4) describes an appropriate calibration scheme, not requiring a sealed flow system downstream of the pump.

10.2.3 The flow rate should be checked before and after each sample collection. Ideally, a rotemeter or mass flow meter should be included in the sampling system to allow periodic observation of the flow rate without disrupting the sampling process.

10.2.4 To collect an air sample the cartridges are removed from the sealed container just prior to initiation of the collection process.

10.2.5 The exit (unmarked) end of the cartridge is connected to the sampling apparatus. The endcap is left on the sample inlet and the entire system is leak checked by activating the sampling pump and observing that no flow is obtained over a 1 minute period. The sampling pump is then shut off.

10.2.6 The endcap is removed from the cartridge, a particulate filter and holder are placed on the inlet end of the cartridge, and the sampling pump is started. In many situations a particulate filter is not necessary since the compounds of interest are in the vapor state. However, if, large amounts of particulate matter are encountered, the filter may be useful to prevent contamination of the cartridge. The following parameters are recorded on an appropriate data sheet (Figure 4): date, sampling location, time, ambient temperature, barometric pressure, relative humidity, dry gas meter reading (if applicable), flow rate, rotometer reading (if applicable), cartridge number, pump, and dry gas meter serial number.

10.2.7 The samples are collected for the desired time, periodically recording the variables listed above. At the end of the sampling period the parameters listed in 10.2.6 are recorded and the flow rate is checked. If the flows at the beginning and end of the sampling period differ by more than 10%, the cartridge should be marked as suspect.

10.2.8 The cartridges are removed (one at a time), the endcaps are replaced, and the cartridges are placed into the original container. The friction top can is sealed and packaged for immediate shipment to the analytical laboratory.

10.2.9 The average sample rate is calculated and recorded for each cartridge according to the following equation:

$$Q_A = \frac{Q_1 + Q_2 + \ldots Q_N}{N}$$

where

Q_A = Average flow rate in ml/minute.
$Q_1, Q_2, \ldots Q_N$ = Flow rates determined at beginning, end, and immediate points during sampling.

N = Number of points averaged.

10.2.10 The total volumetric flow is obtained directly from the dry gas meter or calculated and recorded for each cartridge using the following equation:

$$V_m = \frac{T \times Q_A}{1000}$$

where

V_m = Total volume sampled in liters at measured temperature and pressure.
T = Sampling time = $T_2 - T_1$, minutes.

10.2.11 The total volume sampled (V_s) at standard conditions, 760 mm Hg and 25°C, is calculated from the following equation:

$$V_s = V_m \times \frac{Pa}{760} \times \frac{298}{273 + ta}$$

where

Pa = Average barometric pressure, mm Hg
ta = Average ambient temperature, °C.