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FIG. II-15 - RI/FS SCHEDULE

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ENVIRON

REMEDIAL INVESTIGATION/FEASIBILITY STUDY WORK PLAN

REMEDIAL INVESTIGATION/FEASIBILITY STUDY EAGLE ZINC COMPANY SITE HILLSBORO, ILLINOIS

Submitted to:

U.S. Environmental Protection Agency, Region V
and
Illinois Environmental Protection Agency

Submitted by:

ENVIRON International Corporation
Deerfield, Illinois

On behalf of

Eagle Zinc Group

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I. INTRODUCTION

This Remedial Investigation/Feasibility Study (RI/FS) Work Plan details planned RI/FS activities for the Eagle Zinc Company site (the "Site"), located in Hillsboro, Illinois. This work plan has been prepared by ENVIRON International Corporation (ENVIRON) on behalf of the Eagle Zinc Group (the "Group") as part of the RI/FS for the Site. The RI/FS is being completed pursuant to the Statement of Work (SOW) within the Administrative Order on Consent (AOC) between the Group and the U.S. Environmental Protection Agency (USEPA) dated December 31, 2001.

II. REMEDIAL INVESTIGATION

As stated in the SOW, the overall purpose of the RI is to investigate the Site's physical characteristics, identify sources of contamination, and determine the nature and extent of contamination at the Site. The purpose of the FS is to develop and evaluate remedial action alternatives based on the RI data and report.

In accordance with the SOW, the RI consists of six (6) component tasks:

- Task 1: Description of Current Situation and Investigative Support;
- Task 2: Site Investigations;
- Task 3: Site Investigation Analyses;
- Task 4: Bench/Pilot Testing Studies;
- Task 5: Reports; and
- Task 6: Community Relations Support.

In accordance with the AOC, USEPA will take the lead role in the development and implementation of a Community Relations program for the Site activities. Therefore, a Community Relations Plan has not been included as a component of the RI/FS Work Plan.

The results of the field sampling activities will provide input to the baseline risk assessment and the evaluation of potential response action alternatives in the Feasibility Study. The baseline risk assessment, to be completed as part of the RI, will quantify the potential risk to human health and the environment posed by contaminants potentially present in soil, sediment, surface water and ground water on site, as well as sediment and surface water in drainageways located off site. The evaluation of remedial alternatives for the Site will include an assessment of risk. The results of the RI will be sufficient to provide a basis for the development and evaluation of potential remedial response alternatives for the Site during the subsequent FS. The RI for the Eagle Zinc Site will be conducted in accordance with the *Guidance for Conducting Remedial Investigations and Feasibility Studies Under CERCLA* (USEPA, 1988b), as applicable.

A. Task 1: Description of Current Situation and Investigative Support

1. General

Background information and environmental conditions pertinent to the RI/FS were discussed in detail in the *Preliminary Site Evaluation Report* (the "PSE Report") for the Site, dated February 2002. Portions of the PSE Report are reiterated below, as this information satisfies the requirements of Task 1 of the RI, as outlined in the SOW. Nevertheless, the entire PSE Report should be considered included herein by reference.

2. Site Location and Description

The Site is located in the Township of Hillsboro, Illinois. Hillsboro is located in central Montgomery County, Illinois, approximately 50 miles northeast of St. Louis, Missouri and 35 miles south of Springfield, Illinois. The Site is approximately 132 acres in size and is defined as the parcels of land currently owned by Eagle Zinc Company. The Site is situated on two adjoining tracts of land in the Southeast quarter of Section 1 and the Northeast quarter of Section 12, Township 8 North, Range 4 West, as well as part of the Southwest quarter of Section 6, Township 8 North, Range 3 West of the 3rd Principal Meridian.

Figure II-1 presents a portion of the U.S. Geological Survey Hillsboro, Illinois 7.5-minute quadrangle, indicating the location of the Site property. Figure II-2 is a generalized Site layout map. The Site was surveyed in June 1998 by Hurst-Roche Engineers Inc. (HR Engineers), including a boundary survey and a topographic survey that covers a large portion of the Site. The topographic contour map will be completed to include the entire Site property and will be used as the base map for the project. As specified in the SOW, all sample location (grid) maps will be produced as overlays to the general base map.

The Site is located in a mixed commercial/industrial/residential area located in the northeastern part of Hillsboro. The Site extends from Smith Road south to an unnamed tributary to the Middle Fork of Shoal Creek. Industrial Drive extends north and south along much of the eastern property boundary. North of the Site is Smith Road, a small facility called Hayes Abrasives, a golf course, and farm fields.

Industrial Drive, an asphalt company, a railroad corridor, and the former Hillsboro Glass Company facility (now a steel warehouse) are located east of the Site. Some small commercial/industrial facilities (University of Illinois Extension office, Fuller Brothers Construction/Ready Mix, Illinois Wood Preservers, Hillsboro Rental, Vogel Plumbing) are located south of the Site. Some undeveloped land and a residential area containing single- and multi-family dwellings are located west of the Site. The nearest residential properties are located approximately 200 feet west of the Site in the southwestern and central portions of the Site property.

It is estimated that between 10 and 15 percent of the Site is covered by buildings. Approximately 23 buildings currently exist at the Site, some of which are currently unused or are used only for storage purposes. The types of buildings currently used for facility operations include the office/laboratory building, manufacturing/processing buildings, equipment/raw material/finished product storage buildings, baghouses, and maintenance facilities. Other Site features include railroad spurs, raw material and residual material stockpiles, two storm water retention ponds, a small pond in the southeast corner of the property, and several paved and unpaved roadways.

3. Environmental Setting

a. Climate

The following information on the climate of Hillsboro, Illinois was obtained from on-line sources of historical weather data. The climate of Montgomery County is considered continental and temperate. The summer months are hot and humid with an average temperature of 75° Fahrenheit (F) and an average daily high temperature is 87° F. The winter months are moderately cool with an average temperature of 31° F and an average daily high temperature of 40° F. Rainfall is well distributed throughout the year, with the highest average rainfall in May. Total annual precipitation for the area is approximately 41 inches. Approximately 57 percent, or 23 inches, of the total annual precipitation occurs as rain from April through September and

coincides with the growing season. The average total snowfall accumulation is approximately 18 inches.

The following information is for Springfield, Illinois, which is located approximately 35 miles north of Hillsboro. The average relative humidity is 83 percent in the morning and 63 percent in the afternoon. With the exception of January, the prevailing average wind direction throughout the year is from the south. In January, the average wind direction is from the west-northwest. The average wind speeds are greatest in January, March, April, and November, at 13 miles per hour (mph). The lowest average wind speeds are in July and August, at 8 mph.

b. Topography and Hydrology

The surface topography of the Site is relatively level, with surface elevations ranging from about 600 feet above mean sea level (msl) at the southwest retention pond to about 635 feet above msl in the central portion of the Site. The predominant topographic slope of the Site is southerly. Three surface water ponds exist at the Site: a southwestern storm water retention pond; an engineered storm water retention pond located near the eastern Site property boundary; and a small pond located in the southeastern part of the Site. The southwestern storm water pond receives a large proportion of the Site's storm water runoff. Storm water intermittently discharges westward from this pond to a drainage swale, which in turn discharges to an unnamed intermittent tributary of Middle Fork Shoal Creek. This outfall is permitted with the Illinois Environmental Protection Agency (IEPA) Division of Water Pollution Control as National Pollutant Discharge Elimination System (NPDES) Outfall 001. Middle Fork Shoal Creek flows southwestward and joins Shoal Creek approximately 6 miles southwest of the Site.

Storm water that originates in most of the manufacturing areas and the easternmost part of the Site enters an engineered storm water retention system located near the eastern property boundary. The storm water retention system includes a small concrete settlement structure and a two-cell, clay-lined

retention pond. This system is designed to provide adequate detention time to clarify the water prior to discharge. Storm water generally evaporates from the retention basins or is used as make-up water for the plant's non-contact cooling system. However, periodically, storm water would be discharged to a drainage swale (designated NPDES Outfall 002), which channels the storm water off the Site property to the east. The drainage swale extending from Outfall 002 discharges to Lake Hillsboro, approximately ½-mile east of the Site. Lake Hillsboro is a man-made reservoir, which discharges to Middle Fork Shoal Creek approximately one mile north of the Site.

The southeastern pond is located between two railroad spurs near the entrance to the plant. This pond does not appear to receive storm water runoff and has no inlet or outlet.

In addition to the drainage pathways noted above, storm water that collects in a limited area along the southern Site boundary discharges to a small stream located south of the Site. This stream joins the drainage swale that originates at Outfall 001 just west of the southwest Site property line.

According to the National Wetland Inventory (NWI) Map for Hillsboro, Illinois (U.S. Fish and Wildlife Service, 1988) the only mapped wetlands on the Site property include the southwest retention pond and the small pond located in the southeast part of the Site. These ponds are mapped as "intermittently exposed palustrine wetlands with unconsolidated materials in diked or impounded areas". According to the Federal Emergency Management Agency (FEMA) Flood Hazard Boundary Map for Montgomery County, Illinois (1991), no portions of the Site or the off-Site areas planned for investigation are located within either a 500-year or 100-year flood zone.

According to Illinois State Geological Survey (ISGS) publications, the Site is located within the Central Lowland Physiographic Province of Illinois. Within this province, the Site lies within the Springfield Plain Division of the Till Plains Section. This area is characterized by Pleistocene glacial till and outwash deposits derived from the Illinoian Stage glacial episode.

c. Soils

According to the *Soil Survey of Montgomery County,* the soils at the Site belong to five types, generally corresponding to their elevation and proximity to drainage channels. The soil types include the Hickory Series, the Blair Silt Loam, the Stoy Silt Loam, the Hosmer Silt Loam, and the Cowden-Piasa Complex. These are generally well-drained to somewhat poorly-drained silty soils that form on glacial till or loess (wind-blown, glacially derived deposits that generally consist of silt).

d. Geology

According to the map entitled *Thickness of Glacial Drift in Illinois* (ISGS, 1975), the Site is underlain by between 50 and 100 feet of Pleistocene-age unconsolidated glacial deposits. The surface deposits in the area of the Site consist of up to 5 feet of loess. According to the map entitled *Quaternary Deposits of Illinois* (ISGS, 1979), the site is underlain by the Vandalia Member of the Glasford Formation. This unit consists of hard, compact sandy or silty till. According to maps contained in the document entitled *Potential for Contamination of Shallow Aquifers in Illinois* (ISGS, 1984), the geologic materials underlying the Site are classified as Type E, which is described as "uniform, relatively impermeable silty or clayey till at least 50 feet thick, with no evidence of inter-bedded sand or gravel". This description is verified by monitoring well installation logs prepared by Goodwin & Broms, Inc. (GBI) as part of a ground water investigation conducted at the Site in November 1998. The soil borings for all of the monitoring wells except for G-107 terminated at a depth of 15 feet below grade. The logs indicate that, with the exception of the soil boring for well G-107, which encountered thick deposits of residue materials, clay, silty clay and sandy clay materials were encountered to a depth of 15 feet below ground surface (bgs) at locations throughout the Site.

According to the Geological Map of Illinois (ISGS, 1967), the glacial deposits are underlain by bedrock consisting of the Pennsylvanian-age Bond

Formation. This unit is between 100 and 300 feet thick and predominantly consists of limestone, with some layers of shale and sandstone.

e. Hydrogeology

Based on water level measurements made by GBI in December 1998, the shallow ground water flow direction generally varies across the Site – from southwestward in the southwest part of the Site, to southward and southeastward in the northern and central portions of the Site. A shallow groundwater contour map is presented as Figure II-3. Based on the ground surface elevations at the monitoring wells, the pattern of shallow ground water flow generally reflects the Site topography.

Site activities conducted by Philip Services Corporation (Philip) as part of an underground storage tank investigation completed at the Site in 2000[1] included the completion of four slug tests within monitoring wells installed in the southeastern portion of the Site. The slug tests indicated hydraulic conductivities in the shallow water-bearing zone that ranged from 1.11×10^{-4} centimeters per second (cm/sec) to 8.54×10^{-5} cm/sec. These measurements are within the ranges of hydraulic conductivity generally reported for both glacial till and loess.

Based on well searches conducted by ENVIRON and GBI, State well records indicate the potential existence of several shallow domestic wells in residential areas located east and west of the Site. However, local and county governmental officials indicated: (1) Hillsboro does not use wells to obtain its potable water supplies; and (2) all residents of Hillsboro are supplied with potable water obtained from Lake Hillsboro and Glen Shoals Lake.

In addition to information on nearby water supply wells, ENVIRON obtained analytical results for several rounds of surface water samples collected from Lake Hillsboro by IEPA's Division of Public Water Supply between April and October 2001. The samples were collected from the area

[1] As documented in a report entitled *Site Classification Completion Report*, dated September 13, 2000 ("Philip, 2000").

of the City's potable water intake, which is located near the dam for the reservoir, approximately one mile north of the Site. The samples were analyzed for metal, pesticides, and certain inorganic and physical parameters. The results indicate that no metal or pesticides have exceeded federal primary Maximum Contaminant Limits (MCLs). Manganese concentrations exceeded the secondary MCL for manganese in some of the samples and are likely representative of background concentrations.

4. Site History and Current Operations

The following information concerning the history of the Site is largely summarized from the report entitled *CERCLA Expanded Site Inspection Report* prepared by the IEPA in 1994, a September 5, 2000 letter prepared by Eagle-Picher responding to an information request received from IEPA, a report entitled *Environmental Risk Assessment* prepared by Risk Science International in 1982, historical information sources reviewed at the Hillsboro Public Library, and discussions with Eagle Zinc Company personnel. Additional details on the history of the Site were presented in the PSE Report, including a description of historical aerial photographs reviewed by ENVIRON.

Zinc processing operations began at the Site in 1912, at which time the facility operated as a zinc smelter under the name Lanyon Zinc Company. The Site was purchased by Eagle-Picher Industries in 1919. Eagle-Picher conducted zinc smelting and manufacture of sulfuric acid until approximately 1935. Subsequent to 1919 and most likely in the early 1920s,the manufacture of zinc oxide and leaded zinc oxide commenced at the Site. The leaded zinc oxide was manufactured by combining basic lead sulfate (obtained from off-Site sources) with zinc oxide. Additional details on the leaded zinc oxide are currently unavailable; however, these activities ceased around 1958. Eagle-Picher continued to manufacture zinc oxide at the Site until November 1980, at which time the Site was purchased by The Sherwin-Williams Company. According to Sherwin-Williams personnel, The Sherwin-Williams Company conducted zinc oxide and other manufacturing operations for a period of less than one year. In 1984, the facility was sold by The

Sherwin-Williams Company to Eagle Zinc Company, a division of T.L. Diamond & Company. Eagle Zinc has continued manufacturing zinc oxide using the American process employed by Sherwin-Williams and Eagle-Picher.

Zinc oxide has been manufactured at the Site using both direct and indirect processes. The indirect process involved the processing of zinc metal in a muffle furnace. The direct process, which continues to be used at the Site (the American process) involves the processing of zinc ores and stockpiled furnace residues in a rotary kiln furnace. While it is likely that Eagle-Picher, Sherwin-Williams and Eagle Zinc all used the direct process, only Eagle-Picher and Sherwin-Williams used the indirect process (muffle furnace). Residual materials historically generated by the manufacturing operations have included, among other things, rotary kiln residue, muffle dross, metallic zinc particles, and refractory bricks from the facility's furnaces. Zinc oxide is used in many applications, including the paint and ceramics industries, agricultural products, rubber products and cosmetics.

As discussed above, other products historically manufactured at the Site include leaded zinc oxide, metallic zinc, and sulfuric acid. Sulfuric acid was manufactured at the Site by roasting zinc sulfide to remove the sulfur, with the southwest pond used to provide non-contact cooling water. In addition, a fine-grained product rich in carbon was produced by screening stockpiled rotary residues using a rotary screen and other methods.

The pyrometallurgical process currently used by Eagle Zinc for zinc oxide manufacturing, and which had also been used by Eagle-Picher and Sherwin-Williams, is known as the American process. As currently implemented, this process involves mixing zinc-bearing feedstocks with sized anthracite coal at the mix room. The coal is delivered to the Site by railcar; the zinc ore is delivered to the Site by railcar and truck. The furnace mix is fed into a natural gas-fired rotary furnace, 8-foot diameter by 50 foot long, at the Block 2 Furnace Building. The natural gas provides the heat source and the anthracite coal provides a reducing atmosphere to reduce the zinc feedstocks to zinc vapor. The zinc vapor is drawn from the rotary furnace into a refractory brick combustion chamber and combusted to zinc oxide by the addition of ambient air. The zinc oxide, suspended in the vapor

stream (products of combustion and air), is drawn into a steel flue and a series of steel cooling loops to cool the zinc oxide and vapor stream before it is collected in a baghouse. The residue left in the rotary furnace is expelled from the rotary furnace into the discharge chamber, quenched in water and hauled to a pad for storage. The zinc oxide collected in the baghouse is conveyed to the refinery and stored in bins before refining. Based on the physical and chemical properties of the zinc oxide, bins of zinc oxide may be blended while being refined. The refining process involves conveying the zinc oxide through a natural gas fired rotary dryer in which the temperature of the zinc oxide is varied to achieve the desired product characteristics. The historic operation of the American process would have been similar.

Eagle Zinc currently produces two products: zinc oxide by the American process (described above), and a carbon-rich by-product by a screening process.

The facility screens stockpiled rotary residues using a double rotary screen with ¼-inch and 2-inch screen sizes to produce the fine-grained product that is rich in carbon. This operation is conducted on a concrete pad located immediately west of the zebra building. Large and medium oversize materials created by this process are currently stockpiled to the west of the concrete pad. Eagle Zinc is exploring options for beneficial reuse of this material and is consulting with IEPA on this issue.

Eagle Zinc has also produced zinc oxide using a Waelz Kiln in the Block 3 Building as part of a pilot project. The Waelz Kiln process feedstock is the furnace residue from Block 2 and stored residue onsite. The Waelz Kiln operates like the Block 2 process where the zinc oxide is collected in a baghouse. The product collected is used as a feed for Block 2. The Waelz Kiln has not been used since October 2000.

In addition, Eagle Zinc formerly conducted a metallic zinc granule process in the Zebra building, located in the northern part of the manufacturing plant. Crude zinc granules were conveyed to a Stedman Mill and then screened. The granule product was screened to a desired size fraction. The oversize material (metallic zinc) was collected and shipped off-Site to a different zinc processing facility. The

undersized fraction was zinc oxide, which was sold in bulk. This operation was last conducted in September 2001.

For the purpose of characterizing plant residual materials, residue piles have been categorized as the following types: RR1 (Rotary Residue Type 1); RR2 (Rotary Residue Type 2); RRO (Rotary Residue Oversize); RCO (Rotary Clean Out); CPH (Carbon Plant Hutch); and MP (Miscellaneous Piles). These residue types were described by Eagle Zinc personnel as follows:

RR1: Rotary Residue Type 1 originated from the passing of feedstock through a rotary furnace under the normal American Process. Rotary furnaces 1, 2 and 4 all may have produced this type of residue. The residue is a carbon-bearing material and the non-carbon material is typically smaller than the Rotary Clean Out residue. Much of this material has been processed by screening, resulting in carbon-rich material, which is sold and/or reused on Site in the process, and oversize materials, which are currently stockpiled pending approval for off-Site beneficial reuse.

RR2: This material came from the Block 3 rotary kiln, (now referred to as the Waelz Kiln) prior to 1979 and most likely much earlier. The feedstock that resulted in this rotary residue likely consisted of muffle dross; therefore, this material could differ chemically from RR1 residues.

RCO: Rotary Clean Out residue exhibits, for the most part, the same characteristics as Rotary Residue Type 1. It originates from the formation of a slag ring in a rotary furnace, which is removed with the use of an air hammer. Therefore, it tends to have a larger proportion of irregular shaped particles. Production of this type of residue could have occurred any time a rotary furnace was used. The frequency of furnace clean out has varied significantly.

RRO: Rotary Residue Oversize material consists of Rotary Residue Type 1 that is passed through a screening process. RRO currently generated by Eagle-Zinc consists of material that is between ¼-inch and 2 inches in size. However, RRO

materials historically generated at the Site ranged in size from ¼-inch to significantly larger than 2 inches. RRO material currently generated by Eagle Zinc is staged at a designated location in the northern part of the Site. This is the same location that was historically used to store RRO.

CPH: Carbon Plant Hutch residues were historically produced by a process that passed RR1 residues through a 1/8- or 1/4-inch screen. A majority of the carbon-containing material would pass through the screen and the large particles would be rejected. The carbon-rich fraction would then go through a carbon jig, which consisted of a series of two pans. Water was pumped upwards through the pans. The carbon floated at the top and the heavier material was carried along the bottom. It is the heavier material, called "hutch", which makes up the CPH residue piles.

MP: The materials referred to as "miscellaneous piles" for the purpose of sampling may have originated from the historic use of retort or Wetherill furnaces. These piles also appear to contain other debris, such as refractory brick, construction rubble, and pipe fragments.

5. Regulatory History

The following information concerning the regulatory history of the Site is largely summarized from the report entitled *CERCLA Expanded Site Inspection Report*. Key events in the operational and regulatory history of the Site are noted in the form of a timeline in Figure II-4. The facility was initially listed on the Comprehensive Environmental Response Compensation and Liability Information System (CERCLIS) on June 1, 1981 as a discovery action initiated during Sherwin-Williams' ownership of the Site. Sherwin-Williams filed U.S. EPA form 8900-1, Notification of Hazardous Waste Site, in accordance with Section 103(c) of CERCLA, which indicated that slag had been disposed on the Site property. A Preliminary Assessment (PA) of the Site was conducted in 1984 by the IEPA pursuant to CERCLA, which culminated in the submission of a PA Report to U.S. EPA Region V. Sampling of residual materials by IEPA in the early 1980s resulted

in a determination that the materials were not hazardous waste and the Site was not subject to RCRA permitting.

In addition to the CERCLA activities described above, several sets of surface water samples were collected by the IEPA from the southwest storm water discharge between 1980 and 1982 and analyzed for metals. Detected concentrations of zinc, iron, lead and copper in the surface runoff above applicable state surface water quality standards on one or more occasion resulted in a Notice of Violation (NOV) from the IEPA. This prompted Sherwin-Williams to remove approximately 18,000 tons of residue materials from 10 acres of the Site.

A CERCLA Expanded Site Inspection was conducted by IEPA on October 26 and 27, 1993, and included the collection of 28 environmental samples. The results of the Expanded Site Inspection are summarized in the following section. Based on information provided by IEPA and as reported in the Expanded Site Inspection Report, the U.S. EPA's Chief of Emergency Response for Illinois, Mr. Donald Bruce, determined that the Site did not require a time-critical or non time-critical removal action, and that the Site property did not pose an immediate threat to human health or the environment.

On May 22, 1998, Eagle Zinc entered into an Interim Consent Order with the Illinois Attorney General and IEPA, which contained an interim site plan for: (1) preparation and submittal of a Storm Water Pollution Prevention Plan (SWPPP), (2) sampling of on-site materials, (3) sampling of storm water discharges, (4) development and implementation of a ground water monitoring plan, and (5) disposal of construction and demolition debris.

Pursuant to the Interim Consent Order, a monitoring well installation and ground water sampling program was conducted at the Site by GBI, which included the installation and sampling of nine shallow monitoring wells. IEPA representatives collected split samples from the monitoring wells. This investigation culminated in the submission of the March 1999 report entitled *Monitoring Well Installation and Ground Water Sampling Interim Report* to the IEPA. Sampling of residual piles and underlying soils was also conducted by GBI pursuant to the Interim Consent Order. The results of this investigation, which also

included the collection of split samples by IEPA, were submitted to IEPA in a March 1999 report entitled *Interim Report of Residue Sampling and Analysis.*

Based on the Site's discharges of storm water from two point sources into "Waters of the State", and the occurrence of "regulated industrial activities" at the Site based on the facility's SIC code, the Site was determined by Eagle Zinc to be subject to NPDES storm water permitting requirements as per 40 CFR 122.26 (b)(14)(ii). An NPDES Notice of Intent (NOI) was prepared by Eagle Zinc and submitted to the IEPA. On June 20, 2000, IEPA issued NPDES Permit No. IL0074519. The NPDES permit requires: monthly monitoring of NPDES Outfall 002; preparation/implementation of a SWPPP; and submission of an annual inspection report to IEPA. A SWPPP was prepared for the Site in December 2000. The structural improvements and best management practices specified in the SWPPP included the construction of a new storm water retention system in the northeast area of the Site to allow for settling of runoff prior to discharge to Outfall 002. The storm water retention system, which consists of a two-cell retention basin, was completed in 2001.

The removal of a 500-gallon gasoline UST in April 1998 resulted in the reporting of a Leaking UST (LUST) incident to IEPA, because a limited amount of impacted soil was observed in the tank excavation and a pin-size hole was observed in the tank itself. No free-phase gasoline or ground water was observed in the tank excavation. No contaminated soil was excavated or transported off-Site. The former location of the UST is indicated on Figure II-2. The monitoring wells used for the UST investigation (MW-A, MW-B, MW-C/G-106, MW-D, and MW-E) are shown on Figure II-3.

To address the LUST incident, site classification and assessment activities were performed by GBI and Philip, including: (1) screening of soil samples collected from soil borings using a photoionization detector (PID)[2], (2) collection of a soil sample for laboratory analysis of benzene, toluene, ethylbenzene and xylenes (BTEX), (3) collection of soil samples for particle size analysis, (4) installation of

[2] The soil screening included the soil borings for wells MW-A through MW-E, as well as a soil boring completed to a depth of 5 feet below grade located 20 feet west of the former UST.

four new monitoring wells (one existing well, G-106, was included as part of the investigation and designated MW-C), (5) sampling of five monitoring wells for BTEX compounds, (6) completion of slug tests to estimate hydraulic conductivity, and (7) completion of a well search. Neither the soil sample, nor the any of the ground water samples collected from the monitoring wells contained detectable concentrations of BTEX compounds. Based on these results and discussions with IEPA, the LUST incident was classified as "low priority" and ground water in the former tank area is being monitored periodically[3] for three years, after which time a no further action proposal is expected to be made, barring any detections of contaminants above applicable ground water standards. The ground water monitoring continues to be conducted by Philip on behalf of Eagle Zinc Company and all reports are submitted to IEPA. The ground water monitoring program associated with the former UST is being completed independent from the RI/FS.

6. Evaluation of Existing Data

Several environmental investigations have been conducted on-Site and in adjacent off-Site areas since the early 1980s. These investigations are summarized in Table II-1. The data generated by the previous investigations are summarized below by medium, including soil, sediment, residues, surface/storm water, and ground water. Data summary tables are provided for each medium. The analytical data were compared with: (1) site-specific background data collected during the investigations; and (2) regional background values. Where site-specific background data were available, the "t-Test" was used to determine if a statistically significant difference exists between the media data and background. These comparisons were used in the preliminary identification of potential contaminants of concern (PCOCs) and potential areas of concern (PAOCs).

a. Soil

The 1982 *Environmental Risk Assessment* report prepared by Risk Science International (RSI) presents the results of soil samples collected at

[3] Quarterly during the first year, semi-annually during the second year, and annually during the third year.

various locations on the Site property in October 1980. RSI's report states that the soil samples did not contain concentrations of metals significantly above background soil samples collected in the Hillsboro area. Concerning the soil data noted in RSI's report, all of which were collected by others prior to RSI's risk assessment, the 1982 report concluded: "much of the lead, cadmium, copper, and zinc, although high in concentrations in the dross, kiln residues and ore spoils, appears to be relatively inert and fixed in these materials." An accurate location map for the soil samples was not available to ENVIRON for review. Therefore, the conclusions made by RSI are discussed herein for informational purposes only, and the soil data collected in 1980 have not been included in the preliminary evaluation of Site soil data discussed below.

The Expanded Site Inspection conducted by IEPA included the collection of 18 soil samples: a background sample and duplicate sample collected from a location in the nearby town of Butler, and 16 samples collected at various off-Site locations. All soil samples were collected from the ground surface (0-4 inches below grade) and analyzed for Target Compound List (TCL) inorganic compounds. The locations of the off-Site soil samples collected in 1993 are shown on Figure II-5.

In May 1998, 44 soil samples were collected by GBI at 25 on-Site boring locations. In addition, 6 split samples were collected by IEPA representatives. The boring locations were grouped within four Site areas, which were designated Areas 1 through 4. Between one and three samples were collected from each of the soil borings, which generally extended to the depth at which native clay was encountered. All soil samples were analyzed for lead and cadmium, with selected soil samples also analyzed for Toxicity Characteristic Leaching Procedure (TCLP) lead and TCLP cadmium. The locations of the soil samples collected by GBI in May 1998 are shown on Figure II-6.

The results of the soil samples collected off-Site and on-Site are presented in Tables II-2 and II-3, respectively. All non-leachate data, both on-

Site and off-Site, were compared with Metropolitan Statistical Area (MSA) and non-MSA background values presented in the Illinois Tiered Approach to Corrective Action Objectives (TACO), and off-Site soil data were statistically compared with the results of off-Site background samples collected by IEPA using the t-Test.

Based on these comparisons, no constituent concentrations were determined to be significantly different from site-specific background levels. While arsenic concentrations were determined to be different from site-specific background, the highest detected concentration was only marginally above the average regional background level, as reflected by the non-MSA background value[4]. In addition, arsenic is not known to have been used or released at the Site. As the off-Site soil samples were well distributed around the Site, the available data do not indicate any detectable impacts to off-Site soils through airborne emissions from the Site or other pathways.

For on-Site soil, the lead and cadmium concentrations in each soil area exceeded site area background levels determined by IEPA in 1993, as well as MSA and non-MSA background values. Based on these comparisons, Areas 1 through 4 have been identified as PAOCs for on-Site soil at this time, with lead and cadmium as the principal constituents. As the amount of on-Site soil data is limited, these metals will be considered PCOCs, pending collection of additional data.

b. Sediment

Eight sediment samples were collected by IEPA as part of its October 1993 Expanded Site Inspection. Three of the samples were collected on-Site: sample X-205 from the area of Outfall 001 (drainage point for southwest pond); sample X-206 from the upper reach of the storm water drainage swale that discharges to the southwest pond; and sample X-207 from the drainage swale in the northern part of the Site that discharges at Outfall 002. The remaining samples were collected at off-Site locations in the eastern

[4] Montgomery County is listed in TACO as being located within a non-MSA area.

drainageway (sample X-208) or within the western drainageway (samples X-203 and X-204). Background sample X-201 and its duplicate (X-202) were collected from an unnamed tributary to Middle Fork Shoal Creek, upgradient of the point at which NPDES Outfall 001 discharges to this tributary. All sediment samples were analyzed for the full TCL list, including both organic and inorganic compounds. The locations of the sediment samples collected by IEPA in October 1993 are shown on Figure II-7.

Table II-4 is a compilation of the sediment sampling data. The data were compared with relevant background values. Based on this evaluation, the two major drainageways that receive storm water discharges from the Site have been identified as PAOCs for sediments. The principal constituents identified for sediments at this time include: antimony, arsenic, beryllium, cadmium, lead, nickel, silver, thallium and zinc. As the amount of sediment data is limited, all of these metals will be considered PCOCs, pending collection of additional sediment data in the drainageways.

c. Residues

Two samples of residue piles, designated X-103 and X-105, were collected by IEPA as part of the 1993 Expanded Site Inspection and 68 samples of residue piles were collected by GBI in May 1998, with split samples collected by IEPA. The samples collected by IEPA in 1993 were analyzed for TCL inorganics; the samples collected by GBI in May 1998 were analyzed for lead and cadmium, with selected samples analyzed for TCLP lead and TCLP cadmium. The residue samples collected by GBI represented 15 discrete stockpiles that were categorized as the following types: RRO (Rotary Residue Oversize); RR1 (Rotary Residue Type 1); RR2 (Rotary Residue Type 2); RCO (Rotary Clean Out); CPH (Carbon Plant Hutch); and MP (Miscellaneous Piles).

The stockpiles and locations of all residue samples collected in 1993 and 1998 are shown on Figure II-8. The analytical results for the residue samples are compiled in Table II-5. The residue data were compared with RCRA

maximum concentrations for toxicity. No samples contained TCLP cadmium concentrations above the RCRA limit. In addition, no samples collected from RRO, RCO, or CPH stockpiles contained TCLP lead concentrations above the RCRA limit. At this time, the RR1, RR2 and MP stockpiles have been designated as PAOCs. The principal constituent appears to be TCLP lead. As the amount of data from the residues piles is limited, TCLP lead will be considered a PCOC, pending collection of additional residue data.

As discussed above, the soil data collected by IEPA do not indicate that off-Site soils have been impacted by historical particulate emissions from the Site. Furthermore, based on current and on-going observations made by Eagle Zinc personnel and ENVIRON, the existing residue piles do not appear to be a source of airborne dust emissions. These observations include the relatively large grain size of the materials exposed at the surface of the stockpiles, the consolidated/compacted nature of the older stockpiles, and no observed airborne dust in the areas of the piles during windy conditions. The potential for off-site migration of residues from the piles will be evaluated through visual inspections conducted at the time of sampling. Potential impacts resulting from historical airborne emissions will be investigated through the soil investigations proposed in the Section II of this Work Plan.

d. Surface Water

Storm water samples were collected from the outlet for the southwest pond (general area of current NPDES Outfall 001) for laboratory analysis of inorganic constituents on four occasions between 1980 and 1982. Data are available for two of these sampling rounds: November 19, 1981, and March 23, 1982.

On June 9, 1998, pursuant to the Interim Consent Order with the IEPA, first flush and composite samples were collected from Outfall 001 by GBI and analyzed for metals and other inorganic parameters, and on June 29/30, 1998, GBI collected first flush and composite samples from Outfall 002 and analyzed the samples for metals and other inorganic parameters. Since that

sampling episode, the facility installed a storm water retention basin to capture storm water prior to it being discharged. In addition, since July 18, 2000, the facility has sampled Outfall 002 on a monthly basis[5] as required under the NPDES permit, which regulates the Site's storm water discharges. The analytical parameters for the monthly sampling rounds are total suspended solids (TSS), sulfate, cadmium, and zinc.

The locations of the previous storm water samples, Outfall 001 and Outfall 002, are shown on Figure II-9. The analytical results are summarized in Table II-6. For screening purposes, the storm water data were compared with Illinois Water Quality Standards: 35 IAC 302 Subpart B (General Water Quality Standards), and 35 IAC 302 Subpart D (Secondary Contact and Indigenous Aquatic Life Standards). It should be noted that these criteria are statewide standards and are not effluent limitations specific to the facility's NPDES permit.

While historical storm water data collected in the vicinity Outfall 001 in 1981 and 1982 exceeded Subpart B standards for iron and zinc and Subpart D standards for iron, zinc and lead, the samples collected at Outfall 001 in June 1998 did not exceed either the Subpart B or Subpart D standards. The June 1998 results for Outfall 001 were subsequently used by IEPA as a basis for determining that storm water monitoring was not required at Outfall 001 as part of the facility's NPDES permit.

For storm water samples collected at Outfall 002, chromium, copper and zinc have exceeded the Subpart B standards; however, only zinc also exceeded the Subpart D standard. Based on these comparisons, surface water in the Site's two major drainageways has been identified as a PAOC. The principal constituents appear to be chromium, copper, lead, zinc, manganese, iron and sulfate. As the amount of surface water data is limited, all of these inorganic constituents will be considered PCOCs, pending collection of additional surface water data in the drainageways.

[5] No monthly samples are collected during periods when storm water is not discharging from Outfall 002.

e. Ground Water

In December 1998, GBI collected ground water samples from nine shallow on-Site monitoring wells. The samples were split with IEPA and analyzed for 35 IAC Part 620.410 inorganic and organic parameters. The locations of the monitoring wells are shown on Figure II-3.

No 35 IAC 620 organic constituents were detected in any of the ground water samples collected by GBI or the split samples retained by IEPA. Therefore, Table II-7 shows the results of the inorganic analyses. For screening purposes, the ground water data were compared to the Illinois Ground Water Protection Act (GPA) (35 IAC 620) standards. Metals, including lead, iron, manganese, cadmium, and zinc, as well as sulfate, exceeded GPA standards (Class I, Class II, or both). The exceedances were limited to downgradient wells G-107 and G-108, both of which are located in the southwestern part of the Site. Based on this evaluation, ground water in the area of wells G-107 and G-108 have been identified as a PAOC at this time. The principal constituents appear to be lead, iron, sulfate, manganese, cadmium and zinc. As the amount of ground water data is limited, all of these inorganic constituents will be considered PCOCs, pending collection of additional ground water data at the Site.

As discussed in Section V, four monitoring wells were installed by GBI in the area of a former 500-gallon gasoline UST that exhibited evidence of leakage. The sampling results, which indicated no detectable BTEX compounds, show that ground water has not been impacted. As these data were not collected to assess environmental conditions on the Site as a whole and were all non-detect, neither the data nor the on-going UST monitoring program is discussed further in this report. Based on their locations and relative spacing, it is not anticipated that the monitoring wells installed for the purpose of evaluating potential impacts from the tank (i.e., MW-A, MW-B, MW-D, and MW-E) will be used during the RI.

7. Site Inspection and Access

On January 16, 2002, ENVIRON conducted a preliminary inspection of the Site and neighboring properties and visited several local governmental offices to obtain information pertinent to the PSE. ENVIRON also interviewed Eagle Zinc personnel concerning information pertinent to the PSE. In addition to the information presented above, the following information was obtained:

- All Site monitoring wells were inspected and were observed to be in good condition.
- With the exception of some limited areas of steep or uneven terrain, ENVIRON does not anticipate significant on-Site access limitations.
- Through the use of tax maps and physical inspection, ENVIRON has identified the off-Site properties on which field activities are expected to be conducted during the RI.
- ENVIRON conducted an inspection of the undeveloped northern and western portions of the Site property. With the exception of installation and sampling of monitoring wells by GBI in 1998, these portions of the Site have not been subjected to previous phases of environmental investigation. The majority of the undeveloped area north of the manufacturing plant was historically used as farmland. Based on historical aerial photographs, this area does not appear to have been used by the facility for any purpose. Some limited areas in the vicinity of the eastern drainage ditch, which transects this area of the Site, contain surface deposits of plant residues. Several historical residue piles and surface residue deposits exist in the undeveloped western part of the Site. Using aerial photographs and field sketches, the areas covered by these residues are depicted on Figure II-8. Based on ENVIRON's visual observations, these materials appear to be of the rotary residue types described above. As indicated in the SOW, supplementary soil, residue and ground water sampling activities will be conducted in the undeveloped northern and western areas of the Site as part of the RI.

- A dedicated field office area, restroom facilities, and potable water for drinking and other purposes (e.g., equipment decontamination) will be provided by Eagle Zinc Company to ENVIRON during the course of the field investigations. In addition to a cellular telephone, which will be carried by ENVIRON's on-site field activities coordinator, ENVIRON's field office will have a telephone connected to the plant's main telephone line. A caller will have the ability to either leave a voice mail message or to leave a message with Eagle Zinc's receptionist, who will promptly deliver it to ENVIRON.

8. Site Conceptual Model

Based on the evaluation of previous site data presented in the PSE Report, the following affected media and PCOCs were identified:

On-Site Soil	**Sediment**	**Residues**	**Ground Water**	**Surface Water**
		Analytical Fractions		
TAL-Metals	**TAL-Metals**	**TCLP Metals**	**TAL-Metals**	**TAL-Metals**
Cadmium	Antimony	TCLP-Lead	Cadmium	Chromium
Lead	Arsenic		Lead	Copper
	Beryllium		Manganese	Lead
	Cadmium		Zinc	Manganese
	Lead		Iron	Zinc
	Silver			Iron
	Thallium			
	Zinc			
			Other Inorganics	**Other Inorganics**
			Sulfate	Sulfate

Based on the site investigations conducted to date, the PAOCs for on-Site soil, sediment, residues, surface water and ground water are depicted on Figures II-10 through II-14 and are summarized as follows:

On-site Soil	Sediment	Residues	Groundwater	Surface Water
Areas 1-4	Western Drainageway Eastern Drainageway	RR1 Stockpiles RR2 Stockpiles MP Stockpiles	SW Part of Site	Western Drainageway Eastern Drainageway

Based on a qualitative evaluation, the following potential on-Site and off-Site exposure routes have been identified:

	On-Site Soil	On-Site Ground Water	Off-Site Ground Water	Off-Site Sediments	Surface Water
Potentially Affected Population	Employee Construction Worker Trespasser Future Resident[6]	Construction Worker Future Resident[8]	Resident	Resident, Ecological Receptors	Resident, Ecological Receptors
Exposure Route(s)	Inhalation, Ingestion, Dermal	Inhalation, Ingestion, Dermal	Incidental Residential Exposure	Secondary Residential Exposure, Potential Ecological Impacts	Secondary Residential Exposure, Potential Ecological Impacts

The Site Conceptual Model will be modified and supplemented as necessary during the course of the RI/FS, as additional data are generated and evaluated.

B. Tasks 2 and 3: Site Investigation and Analyses

1. Work Plan Rationale

Consistent with the December 31, 2001 AOC for governing the RI/FS for the Eagle Zinc Site, the RI has been designed to both complement and complete the prior investigations conducted at, and in the vicinity of the Site. Specifically, this work plan is designed to build upon prior investigations of soil, sediments, residues,

[6] This scenario is hypothetical, as residential development of the site is not permitted under current zoning ordinances.

surface water and ground water on Site, as well as sediments and surface water off-Site. Using historical sampling data to the extent possible and through the collection of additional data, where needed, the primary focus of the RI will be to characterize the nature and extent of metals concentrations in these media, to assess potential migration pathways by which the contaminants could impact human or ecological receptors, and to evaluate potential risks to those receptors. The RI will include two phases of investigation: Phase 1 (Source Characterization), and Phase 2 (Migration Pathway Assessment). The investigation results of the RI will be compiled and interpreted as a basis for performing a baseline risk assessment to establish the need for future remedial response activities for the Site. The following sections present an analysis of the three major site characterization components of the RI (Phase 1, Phase 2 and the baseline risk assessment) as a basis for developing data quality objectives.

While the sampling program discussed below conforms with the specifications set forth in the SOW. The actual number and types of samples may be adjusted as necessary, based on evaluation of data developed as field work proceeds. If the initial sampling approach is not adequate to meet the SOW objectives or to support assessment of remedial alternatives, further sampling will be proposed and will be implemented as approved by EPA, in consultation with IEPA. As stated in the SOW, U.S. EPA agrees that it will request additional work under the AOC only when it determines based on available data (including information developed pursuant to the AOC) that hazardous substances released at or from the Site may present a credible threat to human health or the environment and further investigation of the extent of such hazardous substances is necessary.

a. Phase 1- Source Characterization

The first phase of investigation will be carried out to obtain data related to:

- The physical and chemical characteristics of residual materials generated by site manufacturing practices (e.g., type, quantity, chemical and physical properties, and concentrations).

- On-Site soils and on-Site/off-Site sediments. The physical and chemical concentration characteristics of on-Site soils and drainageway sediments will be evaluated.

This information will be obtained from a combination of existing site information, field inspections, and site sampling activities. The source characterization will culminate in the preparation and submittal of a technical memorandum for the Phase 1 investigation activities. This technical memorandum will summarize the findings of the source characterization activities outlined below, and may be used to refine the scope of the Phase 2 investigation.

i. Soil Investigation

As discussed in detail in the PSE Report, two historical investigations of soils have been conducted at the Site: (1) a 1993 investigation performed by the IEPA, primarily involving the collection of off-Site surface soil samples for analysis of Target Analyte List (TAL) inorganic compounds; and (2) a 1998 investigation conducted by GBI which included the collection of shallow soil samples in four broadly-defined on-Site areas for analysis of lead, cadmium, TCLP lead and TCLP cadmium. No constituents were identified as PCOCs in off-Site soils based on the analysis of the prior data presented in the PSE Report. As such, the soil investigations conducted as part of the Phase 1 RI will focus on on-Site soils.

Lead and cadmium were identified as PCOCs for on-Site soil in each of the four areas sampled; however, the sample analyses were limited to these two metals and their leachate concentrations. The

primary objectives of the soil investigation are to: (1) identify any additional PCOCs in soils; (2) determine the nature and extent of impacts to soils resulting from residual materials staged at the Site; and (3) collect data that provide the basis for the baseline risk assessment and evaluation of the need for remedial response alternatives.

To provide data of sufficient quality to achieve these objectives, the analytical data levels for the Phase 1 soil investigation are discussed below.

- Level I field screening of soils will be implemented throughout the field investigation activities using a photoionization detector (PID) and X-ray fluorescence (XRF) analyzer to provide support for health and safety requirements (PID) as well as to guide the decision-making process on which samples should be submitted for quantitative analysis (PID and XRF).
- A fixed-base laboratory will complete level III data analyses for soil samples. Based on field screening results, soil samples will be retained for analysis of Target Analyte List (TAL) metals and/or Target Compound List (TCL) organic compounds and polychlorinated biphenyls (PCBs).

ii. Sediment Investigation

As discussed in detail in the PSE Report, the 1993 investigation performed by the IEPA included the collection of 8 sediment samples. Three of the samples were collected on Site, three of the samples were collected off Site within storm water drainageways and streams downgradient of the Site, and one sample and a duplicate sample were collected at an off-Site background location. All of the sediment samples were analyzed for the full TCL/TAL list of constituents. As discussed in the PSE Report, several metals were identified as PCOCs,

and both major drainageways receiving storm water discharges from the Site were identified as PAOCs for sediments.

The primary objectives of the sediment investigation are to: (1) determine the distribution of metals at key locations in the drainageways, including immediately down stream of the Site outfalls, at major stream confluences, and at the discharge points to Lake Hillsboro and Middle Fork Shoal Creek; (2) determine the nature and extent of any metals impacts to sediments in a small stream that borders the southern property boundary and subsequently joins the western drainageway; and (3) to determine metals concentrations at locations that are upstream of drainageways receiving storm water discharges from the Site (i.e., background locations). To provide data of sufficient quality to achieve these objectives, the analytical data levels for the Phase 1 sediment investigation are as follows:

- Level I field screening of sediments will be implemented throughout the field investigation activities using a PID to provide support for health and safety requirements as well as to guide the decision-making process on which samples should be submitted for quantitative analysis for organic parameters.

- Level III data analyses for sediment samples will be completed by a fixed-base laboratory. All samples will be analyzed for TAL metals. Based on field screening of sediments using a PID, some of the samples will also be analyzed for TCL organic compounds and PCBs.

iii. Residue Investigation

As discussed in detail in the PSE Report, two historical investigations of stockpiled residues at the Site were conducted: (1) the 1993 investigation performed by the IEPA included the collection of 2

residue samples for analysis of TAL metals; and (2) a 1998 investigation conducted by GBI included the collection of residue samples representing each of the six residue types for analysis of lead, cadmium, TCLP lead and TCLP cadmium. TCLP lead was identified as a PCOC for residues of the RR1, RR2 and MP types.

The Phase 1 RI will include additional sampling of each residue type to further evaluate the leachability of the materials for the purpose of RCRA characterization, risk evaluation, and evaluation of potential remedial options.

To provide data of sufficient quality to achieve these objectives, the analytical data levels for the Phase 1 soil investigation are as follows:

- Level I field screening of soils will be implemented throughout the field investigation activities using a PID to provide support for health and safety requirements.

- Level III data analyses for residue samples will be completed by a fixed-base laboratory. The residue samples will be analyzed for TCLP metals and Synthetic Precipitation Leaching Procedure (SPLP) metals.

b. Phase 2- Migration Pathway Assessment

The second phase of investigation will consist of a migration pathway assessment. The potential migration pathways at the Site consist of groundwater and surface water. The migration pathway assessment will culminate in the preparation and submittal of a Phase 2 technical memorandum describing the findings of the Phase 2 investigations. The Phase 2 investigations are expected to including the following activities:

i. Surface Water Investigation

As discussed in the PSE Report, first flush and composite storm water samples were collected from the storm water discharge points referred to in the Site's NPDES permit as Outfalls 001 and 002 by GBI in June 1998 and analyzed for metals and other inorganic parameters. Since 1998, the facility has sampled Outfall 002 on a monthly basis[7] as required under the NPDES permit. For PSE screening purposes, the storm water data were compared with Illinois Water Quality Standards: 35 IAC 302 Subpart B (General Water Quality Standards), and 35 IAC 302 Subpart D (Secondary Contact and Indigenous Aquatic Life Standards). These criteria are statewide standards and are not effluent limitations specific to the facility's NPDES permit. Historical data collected in 1981 and 1982 in the area of Outfall 001 were also evaluated.

While historical storm water data collected in the vicinity of Outfall 001 in 1981 and 1982 exceeded Subpart B standards for iron and zinc and Subpart D standards for iron, zinc and lead, the samples collected at Outfall 001 in June 1998 did not exceed either the Subpart B or Subpart D standards. The June 1998 results for Outfall 001 were subsequently used by IEPA as a basis for determining that storm water monitoring was not required at Outfall 001 as part of the facility's NPDES permit.

For storm water samples collected at Outfall 002, chromium, copper and zinc have exceeded the Subpart B standards; however, only zinc also exceeded the Subpart D standard. As discussed in the PSE Report, surface water in the Site's two major drainageways has been identified as a PAOC, with PCOCs including chromium, copper, lead, zinc, manganese, iron and sulfate.

The primary objectives of the surface water investigation are to: (1) quantify any impacts to surface waters within the drainage swales

[7] No monthly samples are collected during periods when storm water is not discharging from Outfall 002.

and streams that receive storm water discharges from the Site; (2) determine the nature and extent of any metals impacts to surface water in a small stream that borders the southern property boundary and subsequently joins the western drainageway; and (3) to determine metals concentrations in surface waters at background locations that are upstream of the drainageways receiving storm water discharges from the Site. To provide data of sufficient quality to achieve these objectives, the analytical data levels for the Phase 1 sediment investigation are as follows:

- No Level I or Level II data analyses will be performed as part of the surface water investigation. Visual screening of surface waters will be performed prior to sampling to identify any visual impacts, such as sheens or discoloration, as well as the presence of any springs or tributaries. These field observations may be used to guide the decision-making process for identifying sample locations. In addition, the results of the sediment samples collected from the drainageways as part of the Phase 1 investigation will be considered in the selection of surface water sampling locations.

- Level III data analyses for surface water samples will be completed by a fixed-base laboratory. Based on the criteria noted above, surface water samples will be retained for analysis of TAL metals, TCL organic compounds, PCBs, or a combination of these constituent groups.

ii. Ground Water Investigation

In December 1998, GBI collected ground water samples from nine shallow on-Site monitoring wells. The samples were split with IEPA and analyzed for 35 IAC Part 620.410 inorganic and organic parameters.

No 35 IAC 620 organic constituents were detected in any of the ground water samples collected by GBI or the split samples retained by IEPA. For screening purposes, the ground water data were compared to the Illinois Ground Water Protection Act (GPA) (35 IAC 620) standards. Metals, including lead, iron, manganese, cadmium, and zinc, as well as sulfate, exceeded GPA standards (Class I, Class II, or both). The exceedances were limited to downgradient wells G-107 and G-108, both of which are located in the southwestern part of the Site. Based on this evaluation, ground water in the area of wells G-107 and G-108 have been identified as a PAOC at this time. The principal constituents appear to be lead, iron, sulfate, manganese, cadmium and zinc. As the amount of ground water data is limited, all of these inorganic constituents will be considered PCOCs, pending collection of additional ground water data at the Site.

The primary objectives of the ground water investigation are to: (1) provide additional monitoring well coverage and sampling data in the southwestern, western, northern and manufacturing areas of the Site; (2) better characterize the pattern of shallow ground water flow; and (3) better characterize the hydraulic relationship between the shallow ground water and the pond in the southwestern part of the Site. To provide data of sufficient quality to achieve these objectives, the analytical data levels for the Phase 1 sediment investigation are as follows:

- Level I field screening will be conducted during well installation and ground water sampling activities using a PID. Level I data analyses will be conducted during ground water

sampling, including the field parameters pH, specific conductance, temperature, and dissolved oxygen.

- Level II screening samples will be collected from selected temporary monitoring wells installed as part of the Phase 2 ground water investigation. These samples will be analyzed at a fixed-base laboratory for TAL metals, TCL organic compounds/ PCBs, or both of these groups of parameters. The screening results will be used to identify locations for installation of permanent monitoring wells.

- Level III data analyses for ground water samples collected from permanent monitoring wells will be completed by a fixed-base laboratory. The ground water samples will be analyzed for TAL metals, with a subset of the samples also analyzed for the TCL organic compounds and PCBs.

2. RI Tasks

The following sections present an overview of the investigations to be carried out during the RI. The Field Sampling Plan (FSP), included in this document as Appendix A, presents a detailed discussion of sampling procedures and methodologies, as well as criteria used to select sampling locations and samples retained for laboratory analysis. Project quality assurance/quality control (QA/QC) protocol are discussed in the Quality Assurance Project Plan (QAPP), which is included as Appendix B. Health and Safety procedures are discussed in the Site Health and Safety Plan, which is included as Appendix C.

a. Phase 1- Source Characterization

i. Soil Investigation

As discussed in the AOC and SOW, soils in the following areas will be investigated in Phase 1:

- The on-Site areas previously defined as Areas 1 through 4;
- On-Site areas located north and west of the manufacturing plant which have not been sampled to date; and
- The manufacturing plant area.

A total of 130 soil borings will be completed in on-Site areas to characterize the nature and extent of organic and inorganic contaminant concentrations in soils at the Site. The majority of soil borings will be completed in the areas west and southwest of the manufacturing plant, on which raw materials and residual materials were historically stockpiled. Twenty-five (25) soil borings will be completed in each of these areas, which are identified as Areas 1 through 4. Soil borings will also be completed in the manufacturing plant area and in the historically undeveloped northern and western portions of the site property. Ten (10) shallow soil borings will be completed in each of these three areas.

Soil samples will be screened on Site for metals using a portable X-ray fluorescence (XRF) analyzer. Concentrations of the PCOCs identified during the PSE for soil and sediment (antimony, arsenic, beryllium, cadmium, lead, silver, thallium and zinc) will be estimated by XRF screening. The XRF results will be used to identify samples that will be retained for metals analysis at a fixed-based laboratory, as well as to identify locations and depths of additional soil borings.

Samples from 20% of the soil borings completed (a total of 26 samples) will be retained for fixed-base laboratory analysis of TAL metals.

The core soil samples obtained from the soil borings will also be screened for organic vapors using a PID. Samples from 10% of the soil borings completed (a total of 13 samples) will be retained for analysis of TCL organic compounds and PCBs. Details concerning the selection process for the soil borings and samples selected for laboratory analysis are provided in Appendix A (FSP).

ii. Sediment Investigation

A sediment investigation will be conducted in on-Site and off-Site portions of the storm water/surface water drainageways systems that receive storm water discharges from the Site, border the Site, or enter the Site from adjacent upgradient properties. The principal objective of the sediment investigation will be to characterize the nature and extent of metals impacts on sediments in the drainageways. Samples will be collected as transect composites in sediment accumulation areas or at representative locations in the drainage ditches/streams.

Sixteen (16) sediment samples will be collected for fixed-base laboratory analysis. All sediment samples will be analyzed for TAL metals. In addition, 25% of sediment samples (4 samples) will be analyzed for TCL organic compounds and PCBs.

iii. Residue Investigation

Each residue pile or group of piles, identified by type of material or spatial considerations, will be evaluated by collecting representative samples in accordance with SW 846 procedures and testing the samples for metals at a fixed-based laboratory using the Toxicity Characteristic Leaching Procedure (TCLP) and the Synthetic Precipitation Leaching Procedure (SPLP). The samples will be collected from trenches excavated to the base of the piles. It is estimated that 21 residue samples will be collected and analyzed.

b. Phase 2- Migration Pathway Assessment

i. Surface Water Investigation

An evaluation will be made of potential on-Site sources of storm and surface water impacts and potential locations of surface and storm water discharges. Results of the Phase 1 sediment sampling will be used in the determination of final Phase 2 surface water sampling locations, some of which will be located at Phase 1 sediment sampling locations. The surface water investigation will include collection of samples located immediately downstream of storm water Outfalls 001 and 002.

ii. Ground Water Investigation

Temporary monitoring wells will be installed in the southwestern portion of the property, in the manufacturing plant area, and in the northern and western portions of the Site. The temporary wells will be used to 1) provide information concerning the hydraulic relationship between the shallow groundwater and the southwest pond; 2) better characterize the pattern of groundwater flow beneath the site; 3) assist in identifying locations for additional permanent monitoring wells. Twenty (20) temporary wells will be installed: ten (10) in the southwestern portion of the Site[8], four (4) in the manufacturing area, and six (6) in the northern and western portions of the Site.

Groundwater screening samples will be collected from the temporary wells for fixed-base laboratory analysis of TAL metals. Ground water screening samples will be collected from 10 of the 20 temporary wells for analysis of TAL inorganic compounds and 4 of the 20 wells for TCL organic compounds and PCBs. In concert with the full-list data collected from the existing monitoring wells, the screening analyses and piezometric measurements are expected to provide all

[8] The southwestern portion of the Site was designated as a Potential Area of Concern (PCOC) for ground water in the PSE Report.

necessary information concerning the optimal locations for additional wells. In addition, the elevations of all temporary wells will be surveyed and water level measurements will be made from the wells.

A minimum of ten (10) of the temporary wells will be converted to permanent monitoring wells. The locations of the permanent wells will be determined based on the analytical results of the groundwater screening samples and the groundwater flow patterns at the Site. A minimum of four (4) of the permanent wells will be installed in the southwestern portion of the Site. The remaining permanent wells (a minimum of 6 wells) will be installed in the manufacturing, northern and western portions of the Site.

Following the completion and development of the permanent monitoring wells, all existing and newly installed monitoring wells[9] will be sampled for TAL metals. The metals analyses will be conducted using filtered and unfiltered samples to determine total and dissolved metals concentrations. In addition, four of the ground water samples will be analyzed for TCL organic compounds and PCBs. Prior to sampling, a complete round of water level measurements will be made to allow for the construction of a Site-wide groundwater elevation contour map.

c. Baseline Human Health and Ecological Risk Assessment

Based upon the specific chemicals and concentrations detected at the Site, the number and location of the surrounding population, and migration pathways, a Baseline Human Health and Ecological Risk Assessment will be conducted to evaluate potential threats to human health, welfare, or the environment. Potential risks will be quantified whenever possible. A general outline of work for the Baseline Risk Assessment follows:

[9] Excluding existing wells MW-A, MW-B, MW-D and MW-E, which have been and continue to be sampled under a UST compliance program under the oversight of IEPA.

- Select target chemicals for evaluation based on their degree of contribution to the risks associated with the Site.

- Conduct exposure assessments that include the identification of acute and chronic hazards of concerns and the population(s) at risk.

- Evaluate existing toxicity information and determine the potential acute and chronic effects of the Site contaminants as well as the specific effects such as carcinogenicity, reproductive dysfunction, teratogenicity, neurotoxicity, and other metabolic alterations; and environmental effects of aquatic and terrestrial toxicities.

- Assess impact by identifying acceptable exposure guidelines or standards and comparing estimated doses with these guidelines or standards.

This assessment will be conducted in accordance with the procedures described in the *Risk Assessment Guidance for Superfund, Human Health Evaluation Manual* (USEPA 1991) and any current guidance. A report documenting the risk assessment methodologies and results will be prepared and submitted to USEPA for approval. The risk assessment will also include the preparation of an ecological risk assessment consistent with current USEPA guidance. More details concerning the Baseline Human Health and Ecological Risk Assessment are included in the Baseline Risk Assessment Plan (Appendix D).

3. Preliminary Response Objectives and Remedial Action Alternatives

Preliminary response action objectives established for the Eagle Zinc Site will be to reduce or eliminate any threat to public health and the environment resulting from COCs in soil, sediments, surface water and ground water. The acceptable contaminant concentrations or preliminary remediation goals (PRGs) for the COCs

will be established during the RI. Response action objectives for protecting human receptors will be based on both a contaminant concentration and an exposure route. Protection may be achieved by a combination of eliminating exposure and reduction in concentration. The response action goals will be developed on the basis of chemical-specific applicable or relevant and appropriate requirements (ARARs), when available, as well as Site-specific risk factors. Cleanup concentrations will be established based on achieving acceptable levels of risk to receptors as defined in the baseline risk assessment to be conducted as a part of the RI.

General response action alternatives will be identified for each medium of concern, defining the contaminant and its treatment, removal, or other actions, singly or in combination to satisfy response action objectives.

4. Applicable or Relevant and Appropriate Requirements

If unacceptable risk is identified at the Site, CERCLA §121, as amended by SARA, requires the selection of a remedial action that is protective of human health and the environment. If a remedy is necessary to address an unacceptable risk, USEPA defines protectiveness based on comparison to a baseline risk assessment that considers both ARARs and other standards to be considered (TBCs). The risk assessment includes the consideration of site-specific data such as types of hazardous substances present, potentials for exposure, and presence of sensitive populations.

A requirement may be either applicable or relevant and appropriate but not both. According to the *CERCLA Compliance With Other Laws Manual* (USEPA 1988a), "applicable requirements are defined as those cleanup standards, standards of control, and other substantive environmental protection requirements, criteria, or limitations promulgated under Federal or State law that specifically address a hazardous substance, pollutant, contaminant, remedial action, location, or other circumstance at a CERCLA site." Relevant and appropriate requirements are "those cleanup standards, standards of control, and other substantive environmental protection requirements, criteria, or limitations promulgated under Federal or State law that, while not applicable to a hazardous substance, pollutant, contaminant,

remedial action, location, or other circumstance at a CERCLA site, address problems or situations sufficiently similar to those encountered at the CERCLA site that their use is well suited to a particular site."

There are three types of ARARs applicable to actions: chemical-specific, location-specific, and action-specific. A brief description of each is provided below. As part of the RI process for the Eagle Zinc Site, potential ARARs and TBCs will be identified to assist in the development of potential remedial alternatives and remediation goals. ARARs that are eventually applied will be dependent upon accumulated site contaminant data, site-specific conditions, and the selected remedial action alternatives.

a. Chemical-Specific ARARs

Chemical-specific ARARs are usually health- or risk-derived numerical values that establish an acceptable level or concentration of chemicals that may remain in specific environmental media after remediation is complete. These levels are used to help establish remedial cleanup goals. As a general rule, if more than one chemical-specific ARAR exists for a particular contaminant, the most stringent should be applied (USEPA, 1988a). Table II-8 lists potential chemical-specific ARARs for the Eagle Zinc Site based on currently available information. The list will be evaluated and may be modified during the RI/FS based on information obtained about contaminants of concern and affected media.

b. Location-Specific ARARs

Location-specific ARARs are restrictions on the concentration of hazardous substances or the conduct of activities in environmentally sensitive areas. An example of a location-specific restriction on the concentration of hazardous substances is the RCRA land disposal restrictions (LDR) prohibiting hazardous waste placement into or onto the land (e.g., landfills) until waste-specific treatment standards are met. Examples of restrictions on the conduct of activities in environmentally sensitive areas include restrictions

on activities in floodplains, wetlands, and locations where endangered species or historically significant cultural resources are present. Potential location-specific ARARs for the Eagle Zinc Site are listed on Table II-9 and will be evaluated during the RI/FS. The list may be modified based on information obtained during the RI/FS.

c. Action-Specific ARARs

Action-specific ARARs are usually technology- or activity-based requirements or limitations on actions or conditions taken with respect to specific hazardous substances. Action-specific ARARs do not determine the remedial alternative; rather, they indicate how a selected alternative must be achieved. Table II-10 lists potential action-specific ARARs for the Site based on current information about the site. The list will be evaluated and may require modification based on information obtained during the RI/FS.

d. TBCs

Other information that does not qualify as an ARAR may be considered during the development of remedies. This information, referred to as to-be-considered (TBC) information consists of non-promulgated advisories, criteria, or guidance issued by federal, state, or local governmental agencies that are not legally binding. TBC information generally falls within three categories:

- Health effects information with a high degree of credibility
- Technical information on how to perform or evaluate site investigations or response actions
- Policy of administrative agencies

While TBCs do not carry the weight of ARARs in the determination of remediation goals, TBCs are considered in conjunction with ARARs during a site evaluation and may be used as guidance in determining remediation goals

and/or in developing remedies. Although TBCs are not legally binding regulations, they do become enforceable once incorporated into an accepted Record of Decision (ROD).

e. Waivers to ARARs

Under certain circumstances, ARARs may be waived. 40 CFR 300.430(f)(ii)(C)(1) through (6) identifies circumstances under which ARARs may be waived in a private party-lead action:

- The alternative is an interim measure and will become part of a total remedial action that will attain the ARAR;
- Compliance with the ARAR will result in a greater risk to human health and the environment than other alternatives;
- Compliance with the ARAR is technically impracticable from an engineering perspective;
- The alternative will attain a standard of performance that is equivalent to that required under the otherwise applicable standard, requirement, or limitation through use of another method or approach; or
- With respect to a state requirement, the state has not consistently applied, or demonstrated the intent to consistently apply, the promulgated requirement in similar circumstances at other remedial actions within the state.

5. Schedule

Consistent with the general RI/FS Schedule presented as Task 10 in the SOW, Figure II-15 includes the current projected schedule for completion of the RI/FS. Work items and project milestones noted on the schedule include: projected submission dates for draft and final work plans, technical memoranda and reports, estimated dates for receipt of Agency comments and approvals, and estimated dates for technical review meetings with USEPA and IEPA. All dates noted on the

schedule are in terms of elapsed time from receipt of approval of RI/FS project plans, technical memoranda and reports. The schedule will be updated regularly to reflect actual completion dates for project milestones.

6. Project Management

ENVIRON has been designated as the principal contractor for the RI/FS. Dr. Roy O. Ball, P.E. of ENVIRON has been designated as the Project Coordinator. Dr. Ball will be responsible for administration of all the Group's actions required by the AOC. Mr. F. Ross Jones of ENVIRON will be the Project Manager, with responsibility for coordination and oversight of the field investigations, data collection, and data management for the RI. Ms. Janet Kester, PhD, ENVIRON, will be responsible for the completion of the Baseline Risk Assessment for the RI. Subcontractors required for implementation of the RI include a drilling contractor, a surveyor, an excavation contractor, and a commercial laboratory. In addition, subconsultants will be used for data validation and to complete the field survey for the ecological risk assessment.

C. Task 4: Bench/Pilot Testing Studies

As discussed in the SOW, bench and pilot testing studies will be performed, as necessary, to determine the applicability of selected remedial technologies to site-specific conditions. If necessary, these studies will be conducted in the later stages of the RI/FS, after initial screening of remedial technologies and actions conducted during the FS.

D. Task 5: Reports

Technical memoranda will be prepared and submitted at key points during the RI, including at the completion of RI Phases 1 and 2, as well as the Baseline Risk Assessment. The purpose of the technical memoranda will be to transmit data to USEPA and IEPA, as well as to ensure that at the time of preparation of the final RI report, USEPA and IEPA are in agreement that the site characterization activities have been completed satisfactorily.

The RI Report will be prepared following the completion of all field activities and Agency approval of the three technical memoranda. The RI Report will follow the format provided in the guidance document, *Guidance for Conducting Remedial Investigations and Feasibility Studies Under CERCLA* (USEPA, 1988b). All pertinent information contained in the PSE Report, the Phase 1 and Phase 2 technical memoranda, and the baseline risk assessment report, will be presented in the RI Report.

In accordance with the AOC for the RI/FS, written progress reports are to be submitted to the USEPA on a monthly basis, and will be submitted until termination of the Order. The progress reports present a description of all significant developments which occurred during the reporting period, the responses to those significant developments, if any required, problems encountered during the reporting period, a schedule of activities planned for the subsequent reporting period, and any planned resolutions for past problems. In addition, the monthly progress reports will transmit field and laboratory data as they become available.

E. Task 6: Community Relations Support

As discussed in the SOW, a community relations program will be implemented by the USEPA, in consultation with the IEPA. The Group will cooperate with the USEPA and the IEPA in providing RI/FS information to the public. The responsible parties will, at the request of the USEPA, participate in the preparation of information distributed to the public, such as fact sheets, and in public meetings that may be held or sponsored by the USEPA to describe activities at, or concerning, the site, including the findings of the RI/FS.

F. References

USEPA (1988a) *CERCLA Compliance with Other Laws Manual*, OSWER Directive 9234.1-01, August 8, 1988.

USEPA (1988b) *Guidance for Conducting Remedial Investigations and Feasibility Studies Under CERCLA*, OSWER Directive 9355.3-01, EPA/540/6-89/004.

USEPA Office of Emergency and Remedial Response. (1989) *Risk assessment guidance for Superfund.* Vol. I, *Human Health Evaluation Manual.* Washington, D.C. EPA/540-1-89-002. OSWER Directive 9285.7-01a. December.

III. FEASIBILITY STUDY

A. Introduction

This Feasibility Study Work Plan details the activities comprising a Feasibility Study (FS) at the Eagle Zinc Company Site. The objective of the FS is to evaluate remedial alternatives applicable to addressing unacceptable risk identified in the HHRA. The FS for the Site will be conducted in accordance with the *Guidance for Conducting Remedial Investigations and Feasibility Studies Under CERCLA* (USEPA, 1988), as applicable.

To the extent practicable, the FS shall follow presumptive remedy guidance for metals in soils (USEPA, 1999) and any other appropriate presumptive remedy guidance (e.g., USEPA, 1995; USEPA, 1996a). For example, for metals in soils, the presumptive remedies are reclamation/recovery and immobilization for "principal threat" wastes, and containment for "low-level threat " wastes. If certain presumptive remedies are determined by USEPA to be appropriate for certain contaminants and media, the FS will consider only the "no action" alternative and the applicable presumptive remedy technologies for those contaminants/media. By applying presumptive remedies, the remedial alternatives screening process will be streamlined, and certain FS subtasks described below for a non-streamlined FS may be reduced in scope or eliminated pursuant to the presumptive remedy guidance. It is anticipated that the selection of presumptive remedies for use in the FS will be discussed during the technical review meeting for review of the RI Report.

In accordance with the SOW, the FS will consist of the following three tasks:

- Task 7: Remedial Alternatives Screening
- Task 8: Remedial Alternatives Evaluation
- Task 9: Feasibility Study Report

B. Task 7: Remedial Alternatives Screening

The first phase of the Feasibility Study will be the identification of remediation alternatives. A range of alternatives will be identified and evaluated to respond to contaminated media identified at the Site and/or other investigated areas in order to

provide adequate protection of human health and the environment. The potential range of alternatives will include options for reducing toxicity, mobility or volume of waste and will vary in degree to which long-term management of residuals or untreated waste is required. Options will also include containment with little or no treatment, limited action alternatives (e.g. deed restrictions), and a no-action alternative. Alternatives will be developed and evaluated concurrently with the RI process.

1. Establish Remedial Action Objectives

Remedial action objectives established for the Site and/or other investigated areas will specify the contaminants and media of interest, exposure pathways, and the acceptable contaminant level or range of levels for each exposure route. Remedial action objectives for protecting human receptors will identify a contaminant level and an exposure route, instead of a contaminant level alone, because protectiveness may be achieved by reducing exposure as well as by reducing contaminant levels. The goals are developed on the basis of chemical-specific Applicable or Relevant and Appropriate Requirements (ARARs), when available, and site-specific risk related factors. Cleanup levels will be established based on achieving acceptable levels of risk to receptors as defined by the Human Health Risk Assessment and Ecological Screening Evaluation.

2. General Response Actions

General response actions will be identified for each medium of interest defining the contaminant and its treatment, removal, or other actions, singly or in combination to satisfy remedial action objectives. Media requiring treatment shall be identified taking into account requirements for protectiveness as identified in the remedial action objectives and the chemical and physical characteristics of the site.

3. Identify and Screen Technologies

Based on the response actions, treatment technologies will be identified and screened to ensure that technologies considered are applicable to the contaminant and media affected. The screening will be based primarily on a technology's ability

to effectively address the contaminants at the site, as well as implementability, and cost. Applicable process options will be identified for alternative development.

4. Configure and Screen Alternatives

Potential technologies and process options will be combined into media-specific alternatives. The developed alternatives will be defined with respect to size and configuration of the representative process options; time for remediation; rates of flow or treatment; spatial requirements; distances for disposal; and required permits, imposed limitations, and other factors necessary to evaluate the alternatives. If many distinct viable options are available, screening of alternatives will be conducted to limit the number of alternatives that undergo the detailed analysis and to provide consideration of the most promising process options. The alternatives will be screened on a general basis with respect to their effectiveness, implementability, and cost.

C. Task 8: Remedial Alternatives Evaluation

After the initial screening, the range of potential options will be narrowed down to feasible options for further evaluation. A detailed analysis of each feasible alternative will be conducted which consists of an individual analysis of each alternative against the following set of nine evaluation criteria and a comparative analysis of all options against the nine evaluation criteria. The individual analyses will include a technical description of each alternative that outlines the waste management strategy involved and identifies the key ARARs associated with each alternative and an assessment that profiles the performance of that alternative with respect to each of the evaluation criteria. A table summarizing the results of this analysis will also be prepared.

1. Overall Protection of Human Health and the Environment

Each alternative will be evaluated to determine whether or not the remedy provides adequate protection of human health and the environment. The elimination, reduction, or control of risk through treatment, engineering controls, or institutional controls will be examined under this criterion.

2. Compliance with ARARs

ARARs will initially be identified during the Remedial Investigation. During the FS, an evaluation of each alternative in terms of compliance with chemical-specific (e.g., maximum contaminant levels), location-specific (e.g., preservation of historic sites) and action-specific (e.g., permits for offsite discharge) ARARs will be conducted. A determination will be made on whether or not an ARAR can be met or be waived for each alternative evaluated under the Detailed Analysis.

3. Long-term Effectiveness and Permanence

The ability of a remedy to maintain reliable protection of human health and the environment over time once the cleanup goals have been met will be evaluated for each option. Alternatives that provide the highest degrees of long-term effectiveness and permanence are those that leave little or no waste remaining at the site thereby minimizing long term maintenance and monitoring.

4. Reduction of Toxicity, Mobility, or Volume through Treatment

The anticipated performance of each treatment technology will be compared in reducing toxicity, mobility or volume of contamination for each media identified. Included in this evaluation is the amount of waste treated or destroyed, the irreversibility of the treatment process, and the type and quantity of residuals resulting from the treatment.

5. Short Term Effectiveness

The period of time needed to achieve protection and any adverse impacts on human health and the environment that may occur during the construction and implementation period until cleanup goals are achieved will be determined for each technology. Protection of workers and the community are considered under this criterion.

6. Implementability

The technical and administrative feasibility of a remedy, including the availability of materials and services needed to implement a particular option will be evaluated. Technical feasibility includes construction and operation, reliability of technology, and monitoring consideration. Administrative feasibility involves coordinating with state agencies or offices that have permits or other requirements that will need to be obtained or followed to implement a particular option. The availability of particular services and materials will be evaluated in order to determine whether a technology can be implemented.

7. Cost

A cost analysis involving the evaluation of estimated capital and operation and maintenance costs, and net worth costs will be conducted for each option. The accuracy of cost estimates, a present worth analyses, and cost sensitivity analyses will be part of the evaluation of costs for feasible remedial alternatives.

8. State Acceptance

The technical or administrative issues and concerns the IEPA may have regarding each alternative will be discussed under this criterion.

9. Community Acceptance

The issues and concerns the public may have for each of the alternatives will be discussed under this criterion.

D. Task 9: Feasibility Study Report

The FS Report will be prepared in accordance with the *Guidance for Conducting Remedial Investigations and Feasibility Studies Under CERCLA* (USEPA, 1988), as applicable. The FS Report will include the evaluation of remedial alternatives as described in the above subsections for the contaminated media identified in the RI. The objective of the FS Report will be to identify and evaluate remedial technologies that

provide for the protection of human health and the environment, and be in compliance with applicable ARARs.

E. Task 10: RI/FS Schedule

As discussed above, the general RI/FS Schedule, which is current as of the date of this work plan, is presented as Attachment II-15.

F. References

USEPA (1988b) *Guidance for Conducting Remedial Investigations and Feasibility Studies Under CERCLA*, United States Environmental Protection Agency, OSWER Directive 9355.3-01, EPA/540/6-89/004.

USEPA (1995) *Presumptive Remedies: CERCLA Landfill Caps RI/FS Data Collection Guide*, United State Environmental Protection Agency, OSWER Directive 9355.3-18FS, EPS/540/F-95/009.

USEPA (1996a) *Presumptive Response Strategy and Ex-Situ Treatment Technologies for Contaminated Ground Water at CERCLA Sites,* United State Environmental Protection Agency, OSWER Directive 9283.1-12, EPA/540/R-96/023.

USEPA (1996b) *Soil Screening Guidance: Users Guide*, United States Environmental Protection Agency, EPA/S40/R.96/018.

USEPA (1999) *Presumptive Remedy for Metals-in-Soil Sites,* United States Environmental Protection Agency, OSWER Directive 9355.0-72FS, EPA/540-F-98-054.

TABLES

Table II-1: Summary of Historical Site Investigations

Dates	Sampler	Locations	Media	No. Samples	Analytical Parameters	On/Off Site	Purpose
1980-1982	IEPA	Surface Runoff Areas	Storm Water	Unknown	Metals	On-Site	IEPA Stormwater Runoff Concerns
Oct-93	IEPA	On-Site/Off-Site Areas	Soil, Residuals, Sediments	Soil - 19; Sediment - 8; Residuals - 2	Soils-TAL Inorganics; Sediments-Full TCL/TAL List	On-Site/Off-Site	CERCLA/HRS Ranking Data Requirements
May-98	GBI; IEPA Split	On-Site Soils; residual piles	Soil, Residuals	Soils - 44; Residuals - 68	Lead, Cadmium (also selected samples for TCLP lead and cadmium)	On-Site	Interim Consent Order Requirements
Jul-98	GBI; IEPA Split	Outfalls 001 and 002[2]	Storm Water	4	Selected Metals, Other Inorganics, Physical Parameters	On-Site	NPDES Permitting
Dec-98	GBI; IEPA Split	Site Monitoring Wells	Ground Water	10	35 IAC Part 620.410 Inorganic and Organic Parameters	On-Site	Ground Water Assessment

[1] As per 1993 IEPA CERCLA Expanded Site Inspection Report and 1982 Environmental Risk Assessment.

[2] Outfall 002 also sampled monthly pursuant to general storm water permit

GBI - Goodwin & Broms, Inc.
IEPA - Illinois Environmental Protection Agency

Table II-2: Historical Sampling Results - Off-Site Soil

Date	1993		1993		1993		1993		1993		1993		1993	
Sample	X101-B/G		X102-B/G		X104*		X106		X107		X108		X109	
Parameter														
Aluminum (mg/kg)	12400		10000		6880		13000		13000		11500		10200	
Antimony (mg/kg)	8.9	J	9.2	J	10.6	J	9.4	J	10.5	J	13	J	9.3	J
Arsenic (mg/kg)	5.8		5.7		6.6		6.2		8.7		13.4		4.6	
Barium (mg/kg)	230		265		181		224		124		267		130	
Beryllium (mg/kg)	0.8	B	0.81	B	0.49	B	0.63	B	0.72	B	1	B	0.6	B
Cadmium (mg/kg)	--		--		3.2		0.89	B	3.5		11.3		0.71	B
Calcium (mg/kg)	10600		9880		598	B	11600		5360		5430		2580	
Chromium (mg/kg)	16.2		14.4		10.3		15.1		16.1		23.4		13.4	
Cobalt (mg/kg)	4.1	B	6.5	B	13.7		11.1		5.6	B	14.8		6.9	B
Copper (mg/kg)	20	J	19.7	J	30.6	J	24.7	J	36.4	J	104		15.3	
Iron (mg/kg)	14700		14400		11500		15400		14900		33900		12600	
Lead (mg/kg)	148		236		61		28.5		105		388		47	
Magnesium (mg/kg)	2370		2090		1040	B	2150		2090		1630		1530	
Manganese (mg/kg)	434		686		1180		922		600		1670		660	
Mercury (mg/kg)	0.17		0.18		--		--		0.16		0.16		0.11	B
Nickel (mg/kg)	13.5		11.5		20		14		15.9		35.1		11	
Potassium (mg/kg)	1890		1600		491	J	1060	J	1160	J	--		1650	
Selenium (mg/kg)	--		1.3	J	0.27	J	--		--		0.84	J	0.31	J
Silver (mg/kg)	--		--		--		--		--		--		--	
Sodium (mg/kg)	106	B	87.9	B	47.5	B	37.4	B	71.8	B	178	B	65.7	B
Thallium (mg/kg)	0.33	B	0.34	J	1.2	J	0.26	J	0.35	J	1.4	J	0.28	J
Vanadium (mg/kg)	28.5		27.1		27.5		28.5		27.3		37.7		24.7	
Zinc (mg/kg)	136		138		4770		1490		2480		2280		360	

B	The reported value is less than the CRDL but greater than the instrument detection limit.
J	Estimated value. Used in data validation when the quality control data indicate that a value may not be accurate.
--	Not Detected
mg/kg	milligrams per kilograms

*While technically located on-site sample is grouped with other 1993 off-site samples and hence had been compared to more stringent residential values.

Source: 1993 CERCLA Expanded Site Inspection Report

Table II-2: Historical Sampling Results - Off-Site Soil

Date	1993		1993		1993		1993		1993		1993		1993	
Sample	X110*		X111		X112		X113		X114		X115		X116	
Parameter														
Aluminum (mg/kg)	15000		13500		9950		16600		9750		14800		12500	
Antimony (mg/kg)	7.9	J	9	J	10.2	J	7.8	J	8.4	J	11.1	J	9.9	J
Arsenic (mg/kg)	13.6		8.5		6.2		5.6		11.9		10.5		7.1	
Barium (mg/kg)	150		193		233		116		183		181		227	
Beryllium (mg/kg)	0.78	B	0.94	B	0.85	B	0.85	B	1		0.8	B	0.93	B
Cadmium (mg/kg)	2		1.6		2.8		0.68	B	2.9		1.48		2.3	
Calcium (mg/kg)	3450		8380		2800		5940		4230		4970		8430	
Chromium (mg/kg)	20.7		20.2		14.8		21.7		15.9		19.4		18.9	
Cobalt (mg/kg)	8.5	B	7.8	B	11.3	B	10.6		5.8	B	7	B	9.8	B
Copper (mg/kg)	22.5		33.8		15.9		22.5		28.3	J	27.8	J	25.5	J
Iron (mg/kg)	20700		19300		13900		20400		28600		19700		18900	
Lead (mg/kg)	87.6		70.8		70.1		75.1		137		76.2		147	
Magnesium (mg/kg)	2500		1950		17.6		4870		1130		2030		2020	
Manganese (mg/kg)	563		491		2070		568		314		538		851	
Mercury (mg/kg)	--		0.11	B	0.11	B	--		--		0.42		0.24	
Nickel (mg/kg)	15.9		16.5		22.9		18.6		14.4		10.9		16.5	
Potassium (mg/kg)	1980		1920		1970		2400		1040		1470		1750	
Selenium (mg/kg)	0.49	J	0.42	J	0.39	J	0.27	J	0.76	J	0.52	J	0.53	J
Silver (mg/kg)	--		--		--		--		--		1.2		--	
Sodium (mg/kg)	62.8	B	120	B	52.4	B	45.8		293	B	61.5	B	89.9	B
Thallium (mg/kg)	--		0.25	J	0.28	J	0.27	J	0.71	J	0.57	J	0.53	J
Vanadium (mg/kg)	38.7		34.2		28.2		33.7		29.7		34.8		35.1	
Zinc (mg/kg)	606		488		489		451		1580		638		998	

B	The reported value is less than the CRDL but greater than the instrument detection limit.
J	Estimated value. Used in data validation when the quality control data indicate that a value may not be accurate.
--	Not Detected
mg/kg	milligrams per kilograms

*While technically located on-site sample is grouped with other 1993 off-site samples and hence had been compared to more stringent residential values.
Source: 1993 CERCLA Expanded Site Inspection Report

Table II-2: Historical Sampling Results - Off-Site Soil

Date	1993		1993		1993		1993	
Sample	X117		X118		X119		X120	
Parameter								
Aluminum (mg/kg)	13800		1410		9390		16300	
Antimony (mg/kg)	14.5	J	10.9	J	8.3	J	8	J
Arsenic (mg/kg)	8.5		5.9		6.7		10.7	
Barium (mg/kg)	222		106		196		155	
Beryllium (mg/kg)	1.7		0.73	B	0.6	B	0.95	
Cadmium (mg/kg)	4.8		--		2.8		--	
Calcium (mg/kg)	19300		1720		12100		2870	
Chromium (mg/kg)	17.3		18.5		13.7		20.4	
Cobalt (mg/kg)	10.6	B	11.1	B	14.9		7.4	B
Copper (mg/kg)	57.2	J	15.9	J	17.5	J	17.2	J
Iron (mg/kg)	21100		18200		14100		22900	
Lead (mg/kg)	186		30.4		51.9		32.7	
Magnesium (mg/kg)	2140		2120		1790		2870	
Manganese (mg/kg)	995		795		1520		889	
Mercury (mg/kg)	0.14	B	--		0.32		--	
Nickel (mg/kg)	27.5		12.8		14.8		16.9	
Potassium (mg/kg)	1460	J	1210	J	1670		1490	
Selenium (mg/kg)	0.35	J	0.27	J	0.55	J	0.38	J
Silver (mg/kg)	--		--		--		--	
Sodium (mg/kg)	1020	B	--		--		27.7	B
Thallium (mg/kg)	0.35	J	0.27	J	0.5	J	0.25	J
Vanadium (mg/kg)	34.3		34.5	B	26.7		39	
Zinc (mg/kg)	7420		354		1570		371	

B	The reported value is less than the CRDL but greater than the instrument detection limit.
J	Estimated value. Used in data validation when the quality control data indicate that a value may not be
--	Not Detected
mg/kg	milligrams per kilograms

*While technically located on-site sample is grouped with other 1993 off-site samples and hence had been compared to more stringent residential values.
Source: 1993 CERCLA Expanded Site Inspection Report

Table II-3: Historical Sampling Results - On-Site Soil

Date	May-98	May-98	May-98	May-98	May-98	May-98	May-98	May-98	May-98	May-98
Sample ID	Area 1 - 1S	X201	Area 1 - 1B	Area 1 - 108-8	Area 1 - 2S	Area 1 - 2B	Area 1 - 3S	Area 1 - 3B	X203	Area 1 - 4S
Parameter										
Lead (T) mg/kg	4342	2700	511	2330	4330	15.3	4772	3151	6300	4423
Cadmium (T) mg/kg	16.3	35	9.5	NA	16.8	6.6	30.5	20.8	21	21.5
Lead (TCLP) mg/L	8	3.7	NA	3.37	14.4	NA	23.4	31	29.6	36.3
Cadmium (TCLP) mg/L	0.193	0.17	NA	NA	NA	NA	NA	0.164	0.18	NA

Date	May-98	May-98	May-98	May-98	May-98	May-98	May-98	May-98	May-98	May-98
Sample ID	Area 1 - 4B	X205	Area 1 - 5S	Area 1 - 5B	X206	Area 2 - 1S	Area 2 - 2S	Area 2 - 2D	Area 2 - 2B	Area 2 - 3S
Parameter										
Lead (T) mg/kg	1385	4500	1305	1745	2500	1900	2583	2318	552	378
Cadmium (T) mg/kg	15.7	30	7.7	14.5	27	31.2	11.7	45.4	84.1	9.6
Lead (TCLP) mg/L	32	32.4	4.3	5.1	3.8	5.98	2.5	2.96	NA	NA
Cadmium (TCLP) mg/L	0.202	0.16	NA	0.219	0.19	NA	0.149	NA	NA	NA

Date	May-98	May-98	May-98	May-98	May-98	May-98	May-98	May-98	May-98	May-98
Sample ID	Area 2 - 3B	Area 2 - 4S	Area 2 - 4B	Area 3 - 1S	Area 3 - 2S	Area 3 - 3S	Area 3 - 4S	Area 3 - 4B	Area 3 - 5S	X212
Parameter										
Lead (T) mg/kg	9.2	328	158	650	13.8	438	4.6	48.6	7.1	18
Cadmium (T) mg/kg	2.5	6.5	9.2	22.1	8.8	15.9	3.3	7.7	8.8	NA
Lead (TCLP) mg/L	NA	NA	NA	0.04	NA	NA	NA	NA	0.3	NA
Cadmium (TCLP) mg/L	NA	NA	NA	NA	NA	NA	NA	NA	0.064	0.015

B	The reported value is less than the CRDL but greater than the instrument detection limit.
J	Estimated value. Used in data validation when the quality control data indicate that a value may not be accurate.
NA	Not Analyzed
(T)	Total
(TCLP)	Toxicity Characteristic Leaching Procedure
mg/kg	milligrams per kilograms
mg/l	milligrams per liter

Note: "200 Series" sample identification numbers also used for unrelated sediment samples collected by IEPA in 1993.
Source: 1993 CERCLA Expanded Site Inspection Report
1998 GBI Sampling Event and IEPA Spilt Samples

Table II-3: Historical Sampling Results - On-Site Soil

Date	May-98	May-98	May-98	May-98	May-98	May-98	May-98	May-98	May-98	May-98
Sample ID	Area 3 - 6S	Area 3 - 7S	Area 3 - 7B	Area 4 - 1S	Area 4 - 2S	Area 4 - 3S	Area 4 - 3B	Area 4 - 4S	Area 4 - 4B	Area 4 - 5S
Parameter										
Lead (T) mg/kg	887	1260	47.1	5075	267	1.2	20.6	0.4	30.3	8.6
Cadmium (T) mg/kg	485	27.5	69.6	83.4	22.7	8.8	5	4.3	15.9	3.2
Lead (TCLP) mg/L	0.56	0.54	NA	13.6	NA	NA	0.1	NA	NA	NA
Cadmium (TCLP) mg/L	NA	NA	NA	NA	NA	NA	0.074	NA	NA	NA

Date	May-98	May-98	May-98	May-98	May-98	May-98	May-98	May-98	May-98	May-98
Sample ID	Area 4 - 5B	Area 4 - 6S	X217	Area 4 - 6D	Area 4 - 6B	Area 4 - 7S	Area 4 - 8S	Area 4 - 9S	Area 4 - 9D	Area 4 - 9M
Parameter										
Lead (T) mg/kg	17.1	8137	9800	6273	30	160	35.4	789	177	148
Cadmium (T) mg/kg	17.9	53	88	45	24.6	15	24.6	17.9	9.1	6.3
Lead (TCLP) mg/L	NA	4.73	6.7	5.25	NA	NA	0.305	1.48	NA	NA
Cadmium (TCLP) mg/L	NA	0.679	0.001	0.487	NA	NA	0.244	NA	NA	NA

B	The reported value is less than the CRDL but greater than the instrument detection limit.
J	Estimated value. Used in data validation when the quality control data indicate that a value may not be accurate.
NA	Not Analyzed
(T)	Total
(TCLP)	Toxicity Characteristic Leaching Procedure
mg/kg	milligrams per kilograms
mg/l	milligrams per liter

Note: "200 Series" sample identification numbers also used for unrelated sediment samples collected by IEPA in 1993.
Source: 1993 CERCLA Expanded Site Inspection Report
1998 GBI Sampling Event and IEPA Spilt Samples

Table II-4: Historical Sampling Results - Sediment

Date	1993		1993		1993		1993		1993		1993		1993		1993	
Sample	X201-B/G		X202 (X201 Dup)		X203		X204		X205		X206		X207		X208	
Parameter																
Volatiles (ug/kg)																
Methylene Chloride	--		--		--		--		--		160	J	--		--	
Acetone	11	J	22		12	J	--		37	J	78	J	--		--	
2-Butanone (MEK)	--		4	J	6	J	--		20	J	48	J	--		--	
1,1,1-Trichloroethane	--		--		--		27	J	9	J	290	J	--		8	J
Toluene	--		--		--		--		--		36	J	--		--	
Semi-volatiles (ug/kg)																
2-Methylnaphthalene	--		--				--		100	J	--		--		--	
Phenanthrene	--		--		280	J	1900		--		--		--		--	
Anthracene	--		--		--		320	J	--		--		--		--	
Carbazole	--		--		--		290	J	--		--		--		--	
Fluoranthene	--		--		520	J	1700		--		--		130	J	--	
Pyrene	--		--		520	J	1600		--		--		140	J	--	
Benzo(a)anthracene	--		--		230	J	850		--		--		100	J	--	
Chrysene	--		--		310	J	670	J	--		--		120	J	--	
bis(2-ethylhexyl)phthalate	--		--		660		--		--		--		--		--	
Benzo(b)fluoranthene	--		--		480	J	--		--		--		140	J	--	
Benzo(k)fluoranthene	--		--		--		1200		--		--		--		--	
Benzo(a)pyrene	--		--		230	J	810		--		--		--		--	
PCBs (ug/kg)																
Aroclor-1254	--		--		250		120		--		--		--		24	J
Aroclor-1260	17	J	9.3	J	110		100		--		--		--		--	

B	Analyte was found in the associated blank as well as in the sample.
J	Estimated value.
--	Not Detected
ug/kg	micrograms per kilograms

Source: 1993 CERCLA Expanded Site Inspection Report

Table II-4: Historical Sampling Results - Sediment

Date	1993		1993		1993		1993		1993		1993		1993		1993	
Sample	X201-B/G		X202 (X201 Dup)		X203		X204		X205		X206		X207		X208	
Parameter																
Inorganics (mg/kg)																
Aluminum	6630		6390		7370		14900		8360		16300		10700		9810	
Antimony	9	J	10.4	J	10.3	J	17.4	J	9.3	J	62.7	J	10.7	J	10.8	J
Arsenic	4.5		4.3		6.4		10.9		2.9		19.4		6		8	
Barium	79.5		70.4		99.9		97.4		89.6		383		167		92.5	
Beryllium	0.4	B	0.4	B	0.5	B	0.6	B	0.5	B	1.5	B	0.7	B	0.6	B
Cadmium	0.7	B	--		8.6		7.4		1.8		523		11.1		19.6	
Calcium	6360		5520		20300		12000		4660		6260		1510		3020	
Chromium	9.9		9.9		12.1		13.2		11		28.6		14.6		13.7	
Cobalt	6.1	B	4.9	B	6	B	8.1	B	4.5	B	353		10.8	B	4.7	
Copper	11.9		11.2		37.9		41.9		9		1420		20.8		52.2	
Iron	10100		9120		12400		14300		10900		82400		14900		14500	
Lead	46.4		35		101		72.5		10.2		932		76		125	
Magnesium	2760		2390		3330		2960		2620		4970		1500		1930	
Manganese	501		384		722		451		85.9		3500		1470		461	
Mercury	--		--		0.2		0.1	B	--		0.7		--		0.3	
Nickel	9.2	B	8.7	B	11.5		14.7	B	12.6		583		11.9		12.7	
Selenium	0.3	J	0.3	J	0.3	J	0.4	J	0.3	J	4.1		0.3	J	0.4	J
Silver	0.2		--		--		--		--		14.1		--		--	
Sodium	73.3	B	79.5	B	132	B	150	B	84.7	B	470	B	82	B	110	B
Thallium	0.3	J	--		--		0.4	J	0.3	J	3.8	J	0.3	J	0.4	J
Vanadium	17.9		17.4		19		26.3		20.8		52.9		41.2		27.2	
Zinc	326		291		2200		3040		5690		156000		2410		3280	

B	Analyte was found in the associated blank as well as in the sample.
J	Estimated value.
--	Not Detected
ug/kg	milligrams per kilograms

Source: 1993 CERCLA Expanded Site Inspection Report

Table II-4: Historical Sampling Results - Sediment

Date	1993		1993		1993		1993		1993		1993		1993		1993	
Sample	X201-B/G		X202 (X201 Dup)		X203		X204		X205		X206		X207		X208	
Parameter																
Pesticides (ug/kg)																
alpha-BHC	--		--		--		--		--		1.5	J	--		--	
beta-BHC	--		--		--		--		--		1	J	--		--	
gamma-BHC (Lindane)	--		--		--		--		--		1.1	J	--		--	
Aldrin	--		--		4.4		--		--		--		--		--	
Heptachlor epoxide	--		0.2	J	--		1.3	J	--		4.7	J	--		--	
Diedrin	2.3	J	2.6	J	16		12		--		10	J	--		1.3	J
4,4'-DDE	--		0.4	J	--		--		--		0.7	J	--		--	
Endrin	0.3	J	0.9	J	18		12		2.4	J	--		--		2.8	J
Endosulfan II	--		--		--		--		--		--		--		3.6	J
4,4'-DDD	0.4	J	0.9	J	7.5		6	J	--		1.8	J	--		5.1	J
4,4'-DDT	3.7	J	0.4	J	11		15		--		4.8	J	--		--	
Methoyxyclor (Mariate)	--		--		--		--		--		13	J	--		--	
Endrin Ketone	--		0.5	J	--		--		1.6	J	--		--		--	
alpha-Chlorodane	2	J	3.1		16		7		--		1.7	J	--		0.6	J
gamma-Chlorodane	2	J	2.5		15		7.4		--		3	J	--		0.7	J
Toxaphene	--		110	J	--		--		--		--		--		320	P

B	Analyte was found in the associated blank as well as in the sample.
J	Estimated value.
--	Not Detected
ug/kg	micrograms per kilograms

Source: 1993 CERCLA Expanded Site Inspection Report

Table II-5: Historical Sampling Results - Residues

Date	1993		1993	
Sample ID	X103		X105	
Parameter				
Lead (T) mg/kg	260		1040	B
Cadmium (T) mg/kg	3.2		3.2	
Lead (TCLP) mg/L	NA		NA	
Cadmium (TCLP) mg/L	NA		NA	
Aluminum (mg/kg)	14900		7430	
Antimony (mg/kg)	13.9	J	11.4	J
Arsenic (mg/kg)	5		86.3	
Barium (mg/kg)	112		379	
Beryllium (mg/kg)	0.68	B	0.83	B
Calcium (mg/kg)	2010		1930	
Chromium (mg/kg)	15.9		22.6	
Cobalt (mg/kg)	12	B	20.1	
Copper (mg/kg)	201	J	911	J
Iron (mg/kg)	13900		104000	
Magnesium (mg/kg)	1970		1630	
Manganese (mg/kg)	915		178	
Mercury (mg/kg)	--		--	
Nickel (mg/kg)	20		55.9	
Potassium (mg/kg)	1120	B	300	J
Selenium (mg/kg)	0.31	J	1.3	
Silver (mg/kg)	--		6.3	
Sodium (mg/kg)	47.8	B	39.6	B
Thallium (mg/kg)	0.31	J	1.3	J
Vanadium (mg/kg)	28.2		22.6	
Zinc (mg/kg)	5580		31700	

B	The reported value is less than the CRDL but greater than the instrument detection limit.
J	Estimated value. Used in data validation when the quality control data indicate that a value may not be accurate.
NA	Not Analyzed
--	Not Detected
mg/l	milligrams per liter
mg/kg	milligrams per kilogram

Source: 1998 GBI Sampling
1993 IEPA Sampling

Table II-5: Historical Sampling Results - Residues

Date	1998	1998	1998	1998	1998	1998	1998	1998	1998	1998	1998
Sample ID	RR1-P1-1S	RR1-P1-1B	X202	RR1-P2-1S	RR1-P2-1B	RR1-P3-1S	X204	RR1-P3-1M	RR1-P3-1B	RR1-P4-1S	RR1-P4-1M
Parameter											
Lead (T) mg/kg	9970	2241	3000	61.8	986	4339	4000	15000	5292	40	423
Cadmium (T) mg/kg	19.6	15	44	11.8	11.9	30.7	29	14.5	35.4	6.8	8.1
Lead (TCLP) mg/L	123	NA	NA	NA	7.11	24.7	21.3	NA	NA	NA	NA
Cadmium (TCLP) mg/L	0.204	NA	NA	NA	0.123	0.27	0.24	NA	NA	NA	NA

Date	1998	1998	1998	1998	1998	1998	1998	1998	1998	1998	1998
Sample ID	RR1-P4-1B	RR1-P4-1B2	X209	RR1-P5-1S	RR1-P5-1M	RR1-P5-1B	RR1-P5-2S	RR1-P5-2M	RR1-P5-2B	RR1-P5-2D	RR1-P5-3S
Parameter											
Lead (T) mg/kg	55	2644	3100	23.2	15	4.3	20.8	31.7	5.8	8.2	124
Cadmium (T) mg/kg	6.54	66.7	120	5	1.5	1.4	1.8	5	2.7	1.2	7.5
Lead (TCLP) mg/L	NA	14.8	16.7	NA	NA	NA	NA	NA	NA	NA	NA
Cadmium (TCLP) mg/L	NA	0.8	0.99	NA	NA	NA	NA	NA	NA	NA	NA

Date	1998	1998	1998	1998	1998	1998	1998	1998	1998	1998	1998
Sample ID	RR1-P5-3M	RR1-P5-3B	RR1-P5-4S	RR1-P5-4M	RR1-P5-4B	RR1-P6-1S	RR1-P6-1M	RR1-P6-1B	RR1-P7-1S	RR1-P7-1M	RR1-P7-1B
Parameter											
Lead (T) mg/kg	65.4	153	0.9	2.9	5	5012	2338	3059	16.5	40.7	49.3
Cadmium (T) mg/kg	3.89	8.5	0.1	11.1	12.3	19.3	19.7	25	1.2	5.7	3.6
Lead (TCLP) mg/L	0.2	NA	NA	NA	NA	NA	8.61	NA	NA	NA	NA
Cadmium (TCLP) mg/L	0.01	NA	NA	NA	NA	NA	0.273	NA	NA	NA	NA

NA	Not Analyzed
mg/l	milligrams per liter
mg/kg	milligrams per kilogram

Source: 1998 GBI Sampling
1993 IEPA Sampling

Table II-5: Historical Sampling Results - Residues

Date	1998	1998	1998	1998	1998	1998	1998	1998	1998	1998	1998
Sample ID	RR1-P7-2S	RR1-P7-2M	RR1-P7-2B	RR1-P7-3S	RR1-P7-3M	RR1-P7-3D	RR1-P7-3B	RR1-P7-4S	RR1-P7-4M	RR1-P7-4B	X210
Parameter											
Lead (T) mg/kg	24	6.7	11.6	25	31.4	25.9	20	85	26.9	41.4	46
Cadmium (T) mg/kg	4	1.8	5.6	3.9	1.8	0.5	5	7.1	3.8	6.8	3.8
Lead (TCLP) mg/L	NA	0.203	NA	NA	NA	NA	NA	NA	NA	0.255	0.011
Cadmium (TCLP) mg/L	NA	0.055	NA	NA	NA	NA	NA	NA	NA	0.019	0.021

Date	1998	1998	1998	1998	1998	1998	1998	1998	1998	1998	1998
Sample ID	RR1-P8-1S	RR1-P8-1B	MP1-1S	MP1-1B	RCO-P1-1S	X207	RCO-P1-1B	RCO-P2-1S	RCO-P2-1M	X208	RCO-P2-1B
Parameter											
Lead (T) mg/kg	8.8	45	48695	50290	1.43	49	12.5	120	22.9	36	54.6
Cadmium (T) mg/kg	8	6.1	15.8	24.2	0.61	NA	0.4	5.8	1.94	3.3	6.5
Lead (TCLP) mg/L	NA	NA	255	288	0.41	0.012	NA	NA	0.22	0.007	NA
Cadmium (TCLP) mg/L	NA	0.028	NA	0.22	0.027	0.044	NA	NA	0.038	0.052	NA

Date	1998	1998	1998	1998	1998	1998	1998	1998	1998	1998	1998
Sample ID	RCO-P3-1M	RCO-P4-1M	CPH-P1-1S	CPH-P1-1B	CPH-P2-1S	CPH-P2-1B	X211	RRO-P1-1S	RRO-P1-1M	RRO-P1-1B	RRO-P1-2S
Parameter											
Lead (T) mg/kg	2.8	47.9	637	198	65	35.4	77	36.8	44.3	37.2	84.8
Cadmium (T) mg/kg	1.1	10.8	15	13.7	14.2	10	6.3	5	4.3	5.6	3.5
Lead (TCLP) mg/L	NA	NA	1.51	NA	NA	NA	NA	NA	NA	NA	NA
Cadmium (TCLP) mg/L	NA	NA	0.071	NA	NA	NA	NA	NA	NA	NA	NA

NA	Not Analyzed
mg/l	milligrams per liter
mg/kg	milligrams per kilogram

Source: 1998 GBI Sampling
1993 IEPA Sampling

Table II-5: Historical Sampling Results - Residues

Date	1998	1998	1998	1998	1998	1998	1998	1998	1998	1998	1998	1998
Sample ID	RRO-P1-2M	RCO-P3-1M	RCO-P4-1M	CPH-P1-1S	CPH-P1-1B	CPH-P2-1S	CPH-P2-1B	X211	RRO-P1-1S	RRO-P1-1M	RRO-P1-1B	RRO-P1-2S
Parameter												
Lead (T) mg/kg	272	2.8	47.9	637	198	65	35.4	77	36.8	44.3	37.2	84.8
Cadmium (T) mg/kg	3.6	1.1	10.8	15	13.7	14.2	10	6.3	5	4.3	5.6	3.5
Lead (TCLP) mg/L	0.166	NA	NA	1.51	NA	NA	NA	NA	NA	NA	NA	NA
Cadmium (TCLP) mg/L	NA	NA	NA	0.071	NA	NA	NA	0.015	NA	NA	NA	NA

Date	1998	1998	1998	1998	1998	1998	1998	1998	1998	1998	1998	1998
Sample ID	RRO-P1-2M	RRO-P1-2B	RRO-P1-3S	RRO-P1-3M	RRO-P1-3B	X218	RR2-P1-1S	RR2-P1-1M	RR2-P1-1B	X216	RR2-P1-2S	RR2-P1-2M
Parameter												
Lead (T) mg/kg	272	1292	80	842	1696	670	2722	2372	3385	4600	13797	456
Cadmium (T) mg/kg	3.6	8.4	16.7	6.7	6.1	6.1	7.3	9.2	5	50	5.8	1.5
Lead (TCLP) mg/L	0.166	0.081	NA	NA	0.158	0.084	4.18	5.3	7.11	8.5	16.9	2.74
Cadmium (TCLP) mg/L	NA	0.085	NA	NA	0.028	0.035	0.114	0.069	0.039	0.038	0.057	0.016

Date	1998	1998	1998	1998	1998	1998	1998	1998	1998	1998	1998
Sample ID	X213	RR2-P1-2B	RR2-P1-3S	RR2-P1-3M	RR2-P1-3B	X214	RR2-P1-3D	RR2-P1-4S	RR2-P1-4M	X215	RR2-P1-4B
Parameter											
Lead (T) mg/kg	860	3186	1589	4362	690	160	274	3147	2781	3300	14298
Cadmium (T) mg/kg	3	3.6	3.6	7.7	4.2	11	10.4	9.1	2	5.5	46.5
Lead (TCLP) mg/L	2.6	1.94	5.63	10.6	1.21	0.12	0.29	3.84	5.71	6.7	2.33
Cadmium (TCLP) mg/L	0.02	0.031	0.016	0.094	0.062	0.11	0.132	0.133	0.046	0.042	0.514

NA	Not Analyzed
mg/l	milligrams per liter
mg/kg	milligrams per kilogram

Source: 1998 GBI Sampling
1993 IEPA Sampling

Table II-6: Historical Sampling Results - Storm Water

OUTFALL	002	002	002	002	002	002	002	002	002	002	002	002	002	002	001	001	001	001
Type	First Flush	COMP	First Flush	COMP													First Flush	COMP
Date	6/30/1998	6/30/1998	6/29/1998	6/29/1998	7/18/2000	8/17/2000	9/12/2000	10/4/2000	11/16/2000	12/11/2000	2/9/2001	3/15/2001	5/5/2001	6/15/2001	11/19/1981	3/23/1982	6/9/1998	6/9/1998
Parameters																		
pH	7.6	8	7	7	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	7.5	7.2	7.4
Fluoride (mg/L)	0.67	0.98	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA
Sulfate (mg/L)	135	116	NA	NA	237	433	97	78	51.6	225	117	325	506	212	NA	NA	NA	NA
Ammonia (as N) (mg/L)	0.31	0.42	0.1	0.1	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	0.1	0.1
Phosphorus-P (T) (mg/L)	0.58	0.17	0.17	0.11	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	0.05	0.05
BOD5 (mg/L)	5	4	8.1	6.7	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	4.1	3.7
FOG (T)	NA	NA	0.8	0.6	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	0.1	0.1
Mercury (T) (mg/L)	0.0001	0.0001	0.0001	0.0001	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	0.0001	0.0001
Calcium (T) (mg/L)	50	41	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA
Sodium (T) (mg/L)	34	21	273	216	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA
Aluminum (T) (mg/L)	39	6.9	5.5	2.8	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	0.253	0.197
Boron (T) (mg/L)	0.16	0.1	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	0.1	NA	NA
Cadmium (T) (mg/L)	0.043	0.022	0.031	0.02	<0.1	0.002	0.005	0.004	0.004	0.009	0.016	0.02	0.003	0.009	0.018	<0.005	0.006	0.005
Copper (T) (mg/L)	0.64	0.32	0.346	0.235	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	0.03	<0.01	0.007	0.006
Iron (T) (mg/L)	52	11	3.05	2.78	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	3	1.8	0.6	0.438
Manganese (T) (mg/L)	1.3	0.43	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	0.83	0.37	NA	NA
Silver (T) (mg/L)	0.006	0.005	0.001	0.001	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	<0.005	<0.005	0.001	0.001
Vanadium (T) (mg/L)	0.1	0.024	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA
Hardness (mg/L)	NA	NA	88	98.8	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	198	196
(ROE) TDS (mg/L)	310	300	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	514	NA	NA
Chloride (T) (mg/L)	6.4	6.6	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA
Nitrate,Nitrite (N Total) (mg/L)	0.73	0.99	0.86	1.11	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	0.15	0.15
Cyanide T) (mg/L)	0.01	0.01	0.1	0.1	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	0.1	0.1
TSS (mg/L)	4120	1080	2400	538	200	1	233	131	24	54	577	NA	NA	113	NA	NA	20	20
Antimony (mg/L)	0.017	0.026	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA
Arsenic (T) (mg/L)	0.052	0.032	0.006	0.004	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	0.002	0.001	0.001	0.001
Selenium (T) (mg/L)	0.01	0.01	0.002	0.002	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	<0.001	<0.001	0.001	0.001
Magnesium (T) (mg/L)	9.9	4	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA
Potassium (T) (mg/L)	31	18	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA
Barium (T) (mg/L)	0.57	0.17	0.202	0.112	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	0.1	0.1	0.042	0.039
Beryllium (T) (mg/L)	0.0019	0.001	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA
Chromium (T) (mg/L)	0.067	0.017	0.016	0.006	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	<0.01	<0.05	0.001	0.001
Cobalt (T) (mg/L)	0.046	0.022	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA
Lead (T) (mg/L)	0.55	0.31	0.362	0.287	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	0.14	<0.05	0.017	0.013
Nickel (T) (mg/L)	0.16	0.07	0.076	0.051	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	0.07	<0.05	0.009	0.008
Strontium (T) (mg/L)	0.24	0.18	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA
Thallium (mg/L)	0.01	0.01	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA
Zinc (T) (mg/L)	291	200	NA	NA	56.5	8.63	15.9	10.2	10.1	10.3	43.3	1.08	5.76	42.6	2.84	2.2	0.947	0.885
COD (mg/L)	NA	NA	147	76	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	88	71
TDS (Conductivity)	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	460	NA	NA
Chromium Hexavalent) (mg/L)	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	0	NA	NA

NA Not Analyzed
mg/l milligrams per liter

NA Not Analyzed
mg/l milligrams per liter

Source: 1998 GBI Sampling Event
1988 IEPA Samples

Table II-7: Historical Sampling Results - Groundwater

With the exception of pH, all parameters are measured in mg/L (ppm).
Only detect values are presented. All wells sampled on December 1, 1998.

Well Number	Lab	Antimony	Arsenic	Barium	Beryllium	Boron	Cadmium	Chromium	Cobalt	Copper	Fluoride
G101	PAS		0.002	0.056				0.014	0.003	0.013	0.58
	IEPA			0.057		0.064		0.012		0.009	0.52
G102	PAS		0.001	0.109				0.005	0.002	0.009	0.42
	IEPA			0.1				0.005			0.41
G103	PAS		0.004	0.124				0.013	0.004	0.013	0.49
	IEPA			0.11		0.051		0.012	0.005	0.01	0.48
G104	PAS	0.001	0.006	0.11				0.022	0.006	0.021	0.37
	IEPA			0.08		0.1		0.012	0.006	0.016	0.34
G105	PAS			0.09				0.001		0.003	0.33
	IEPA			0.088							0.3
G106	PAS		0.001	0.037		0.44		0.003	0.001	0.008	0.32
	IEPA			0.036		0.38				0.007	0.34
G106 (Duplicate)	PAS	0.001	0.004	0.092		0.42		0.014	0.004	0.013	0.33
G107	PAS	0.002	0.003	0.071		0.64	0.044	0.002	0.006	0.019	0.52
	IEPA										
G108	PAS		0.003	0.054		0.66	0.029	0.002	0.016	0.017	0.4
	IEPA			0.053		0.56	0.032		0.02	0.006	0.38
G109	PAS	0.002	0.003	0.065		0.14		0.012	0.002	0.058	0.54
	IEPA			0.059		0.067		0.01		0.058	0.45

Source: Monitoring Well Installation and GW Sampling Interim Report, March 1999

Table II-7: Historical Sampling Results - Groundwater

With the exception of pH, all parameters are measured in mg/L (ppm).
Only detect values are presented. All wells sampled on December 1, 1998.

Well Number	Lab	Iron	Lead	Manganese	Mercury	Nickel	Selenium	Silver	Thallium	Zinc
G101	PAS	1.98	0.004	0.087		0.008	0.005			0.03
	IEPA	8.9	0.005	0.19		0.01				
G102	PAS	1.82	0.002	0.424		0.005	0.001			0.019
	IEPA	4.2		0.43						0.12
G103	PAS	3.19	0.007	0.206		0.013	0.002			0.08
	IEPA	12	0.007	0.41		0.014				
G104	PAS	6.62	0.016	0.177		0.019	0.002			0.6
	IEPA	13	0.018	0.38		0.015				0.99
G105	PAS	0.22	0.001	0.204		0.002				0.023
	IEPA	1		0.23						
G106	PAS	0.88	0.003	0.061		0.004	0.001			0.038
	IEPA	3.6		0.12						
G106 (Duplicate)	PAS	3.8	0.011	0.215		0.012	0.002			0.104
G107	PAS	5.96	0.182	2.56		0.005				1.86
	IEPA									
G108	PAS	3.68	0.011	6.1		0.014	0.007			5.34
	IEPA	7.7	0.01	6		0.016				5.4
G109	PAS	3.14	0.012	0.124		0.008	0.006			0.058
	IEPA	10	0.007	0.025		0.01				

Source: Monitoring Well Installation and GW Sampling Interim Report, March 1999

Table II-7: Historical Sampling Results - Groundwater

With the exception of pH, all parameters are measured in mg/L (ppm).
Only detect values are presented. All wells sampled on December 1, 1998.

Well Number	Lab	Chloride	Cyanide	Nitrate-N	Nitrate, Nitrite	pH	Sulfate	TDS
G101	PAS	2.21				7.5	71.3	425
	IEPA	2.1			0.12	7.2	60.8	375
G102	PAS	47.9				7.4	271	700
	IEPA	66.9				7.4	240	650
G103	PAS	51.1		0.7		7.3	319	1020
	IEPA	72.6			0.57	7.5	578	610
G104	PAS	31		0.4		7.5	470	995
	IEPA	45.9			0.39	7.2	673	701
G105	PAS	14.6				7.5	171	535
	IEPA	19.8				7.2	171	484
G106	PAS	23.4		0.3		7.5	398	895
	IEPA	30.3				7.9	621	628
G106 (Duplicate)	PAS	23		0.3		7.4	400	905
G107	PAS	11.5				7.2	410	790
	IEPA							
G108	PAS	11.2		0.1		7	231	675
	IEPA	16.7				7.4	204	501
G109	PAS	1.12		0.3		7.7	48	185
	IEPA				0.25	6.9	40.9	203

Source: Monitoring Well Installation and GW Sampling Interim Report, March 1999

Table II-8
Potential Chemical-Specific ARARs

Statute/Regulation Citation	Summary
Tiered Approach to Corrective Action Objectives (TACO) Title 35 Subtitle G Chapter I Part 740	Provides risk-based assessment process and objectives
Illinois Groundwater Protection Act 35 IAC 602	Provides numerical standards and groundwater classification
Water Quality Standards 35 IAC 302 Subparts B and D	Provides Illinois surface water standards
Federal Ambient Water Quality Criteria CWA 40 CFR 131	Establishes methods and requirements for states in the development of ambient water quality criteria for the protection of aquatic organisms and/or the protection of human health in surface water.
Lead; Identification of Dangerous Levels of Lead; Final Rule 40 CFR 745	Establishes acceptable residential lead levels.

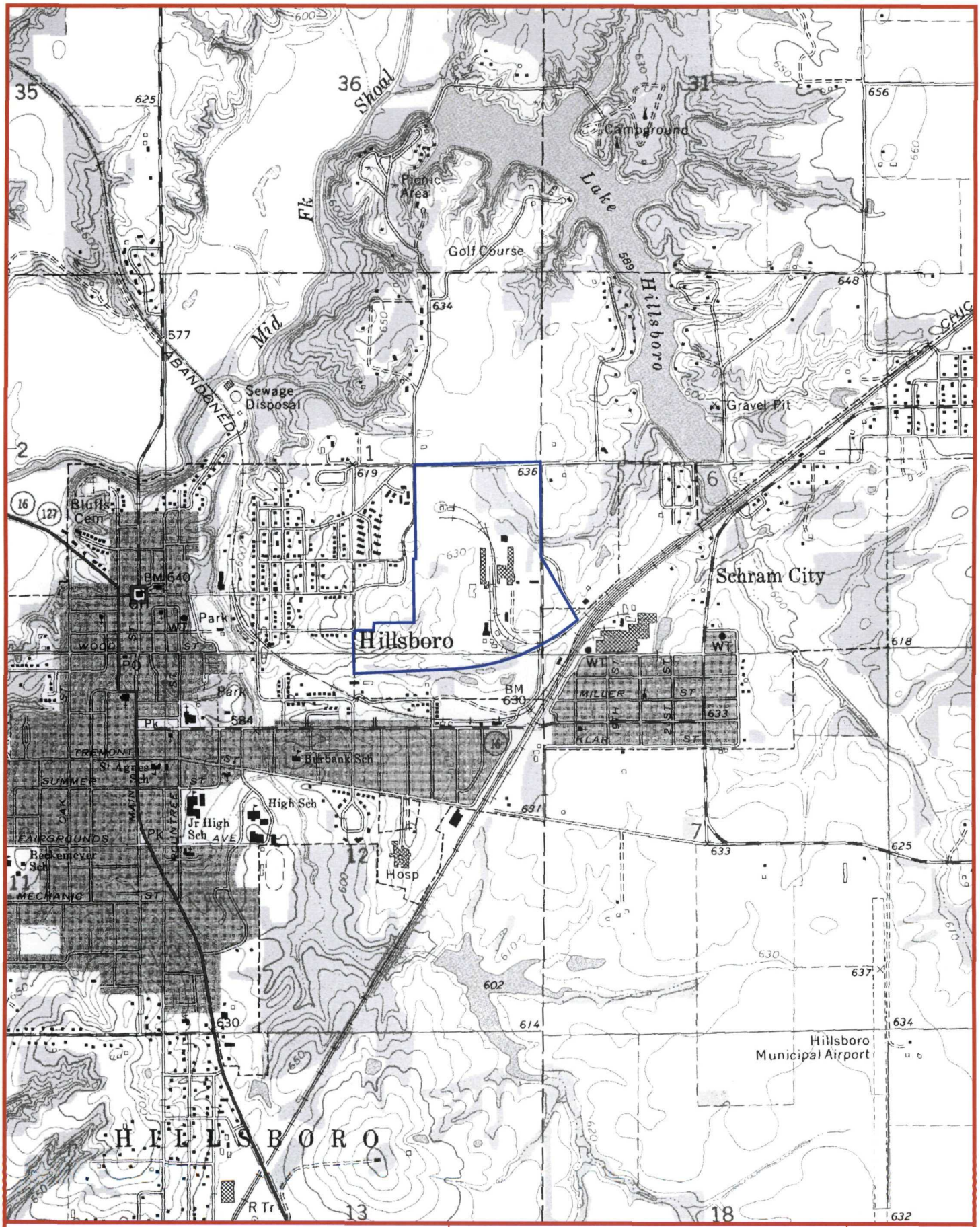
Table II-9
Potential Location-Specific ARARs

Statute/Regulation [Citation]	Summary
Endangered Species Act 16 USC 1531-1544, 50 CFR 200, 50 CFR 402	The purpose of this act is to conserve endangered, threatened, and rare species of wildlife and plants. This regulation prohibits federal agencies from jeopardizing habitat for endangered or threatened species.
Wetlands Protection [Executive Order 11990] 40 CFR 6.302, Appendix A	Regulates action involving construction of facilities or management of property in wetlands to avoid adverse effects, minimize potential harm, and preserve and enhance wetlands, to the extent possible.
Federal Floodplain Management [Executive Order 11988] 40 CFR 6.302, Appendix A	Regulates actions that will occur in floodplain to avoid adverse effects due to flooding.
National Historic Preservation Act Executive Order 11593 40 CFR 6.301(b)	Requires federal agencies to take into account the effect of any federally-assisted undertaking or licensing on any district, site, building, structure, or object that is included in or eligible for inclusion in the National Register of Historic Places. Provides for protection, enhancement, and preservation of sites with archeological or

Table II-10
Potential Action-Specific ARARs

Statute/Regulation Citation	Summary
Resource Conservation and Recovery Act (RCRA) 40 CFR Part 260-270	Establishes provisions covering USEPA permitting requirements, and establishes toxicity characteristics for hazardous waste.
Occupational Safety and Health Act 29 CFR Part 1910	Protects worker health and safety
Clean Water Act, National Pollutant Discharge Elimination System (NPDES) CWA 40 CFR 122	Regulates discharges of pollutants to surface waters.
Clean Air Act 42 USC 7401-7671, 40 CFR 50, 40 CFR 61	Protects ambient air quality through pollutant source control. It establishes National Ambient Air Quality Standards (NAAQS) and National Emission Standards for Hazardous Air Pollutants (NESHAP).

FIGURES



ENVIRON

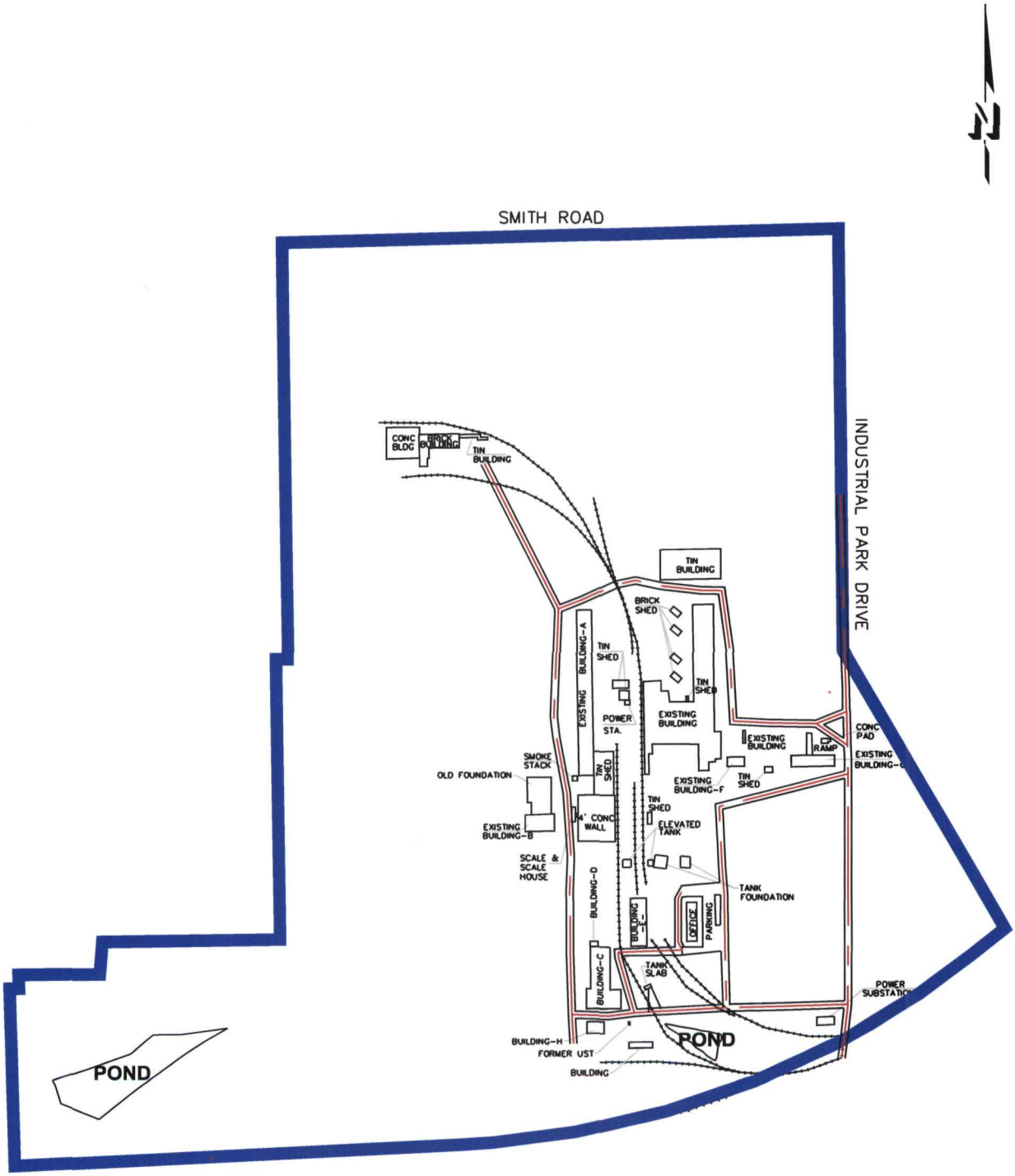
740 Waukegan Road, Suite 401, Deerfield, IL 60015

Site Location Map
Eagle Zinc
Hillsboro, Illinois

Figure
II-1

Drafter: CJG | Date: 06/05/02 | Contract Number: 21-7400E | Approved: | Revised:

CPF\Eagle Zinc\Ri... Workplan Report\Figures\Figure II-1



ENVIRON

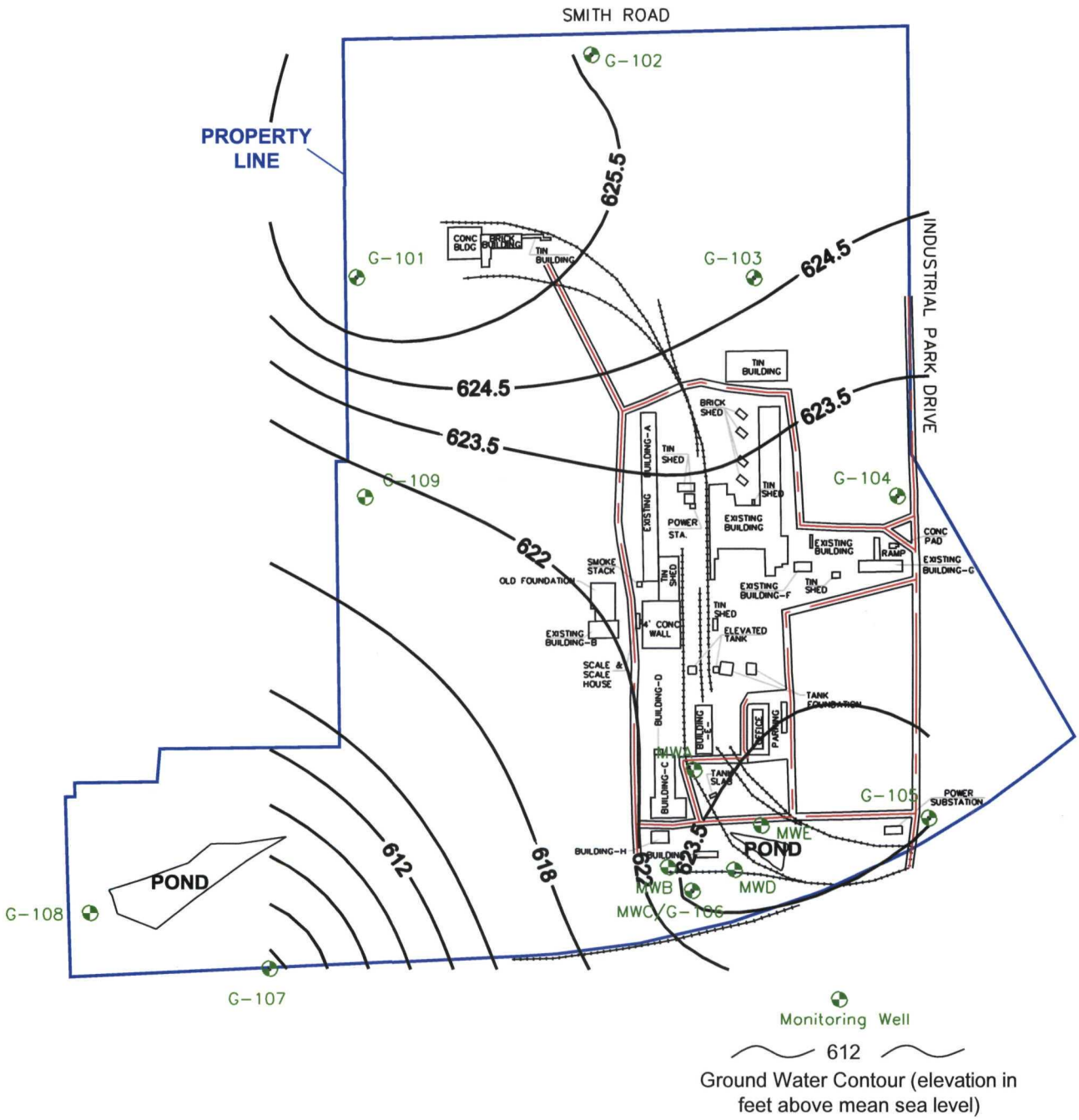
740 Waukegan Road, Suite 401, Deerfield, IL 60015

Site Layout
Eagle Zinc
Hillsboro, IL

Figure
II-2

Drafter: CJG | Date: 06/05/02 | Contract Number: 21-7400E | Approved: | Revised:

CPF\Eagle Zinc\RIP...\orkplan Report\Figures\II-2



ENVIRON

740 Waukegan Road, Suite 401, Deerfield, IL 60015

Shallow Ground Water Contour Map
Eagle Zinc, Hillsboro, Illinois

Figure II-3

Drafter: CJG | Date: 06/05/02 | Contract Number: 21-7400E | Approved: | Revised:

CPF\Eagle Zinc\RIFS workplan\Figures\II-3

1912 Site initially developed for zinc processing under the name Lanyon Zinc Company. Products: zinc, sulfuric acid

1919 Site purchased by Eagle-Picher Industries. Same products as Lanyon.

1935 Began manufacture of zinc oxide and leaded zinc oxide. Ceased zinc smelting and sulfuric acid production.

1958 Ceased production of leaded zinc oxide.

November 1980 Site purchased by The Sherwin-Williams Company.

June 1981 Site initially listed on CERCLIS as a Discovery Action.

1984 CERCLA Preliminary Assessment conducted by IEPA.

1984 Site purchased by Eagle Zinc Company, a Division of T.L. Diamond & Company.

October 1993 CERCLA Expanded Site Inspection conducted by IEPA.

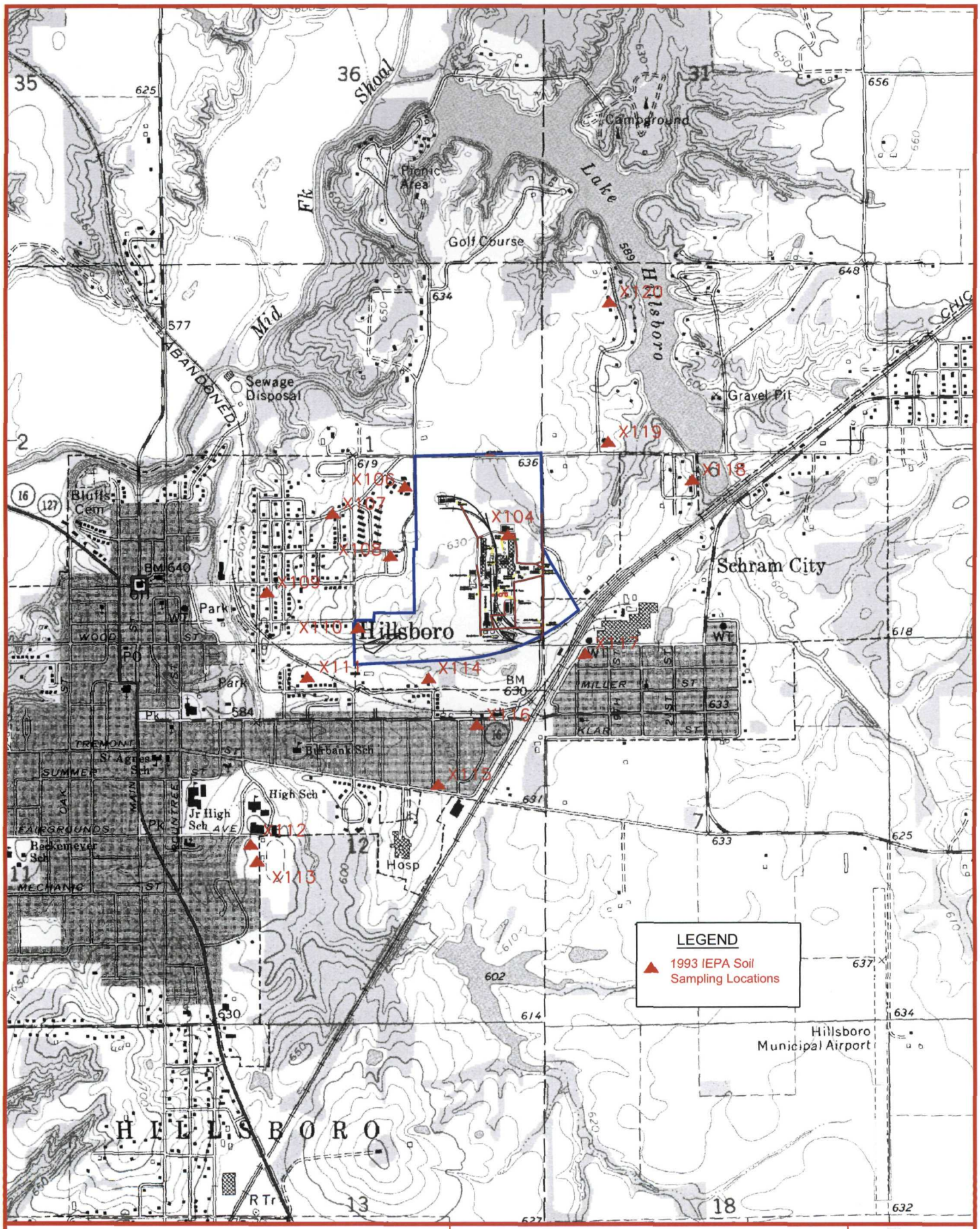
May 1998 Site entered into Interim Consent Order with IEPA.

March 1999 GBI Reports entitled *Monitoring Well Installation and Ground Water Sampling Interim Report and Interim Report of Residue Sampling and Analysis* submitted to IEPA.

June 2000 NPDES Storm Water Permit No. IL 0074519 issued to Eagle Zinc Company.

Summer 2001 Engineered storm water retention pond constructed at Outfall 002.

December 31, 2001 Eagle Zinc Parties enter into AOC with USEPA for completion of RI/FS.



ENVIRON

740 Waukegan Road, Suite 401, Deerfield, IL 60015

Previous Sampling Locations
Off-Site Soil
Eagle Zinc, Hillsboro, Illinois

Figure
II-5

Drafter: CJG | Date: 06/05/02 | Contract Number: 21-7400E | Approved: | Revised:

CPF\Eagle Zinc\RI/...ork Plan Report\Figures\Figure II-5

ENVIRON

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Previous Sampling Locations
On-Site Soil
Eagle Zinc, Hillsboro, Illinois

Figure
II-6

Drafter: CJG | Date: 06/05/02 | Contract Number: 21-7400E | Approved: | Revised:

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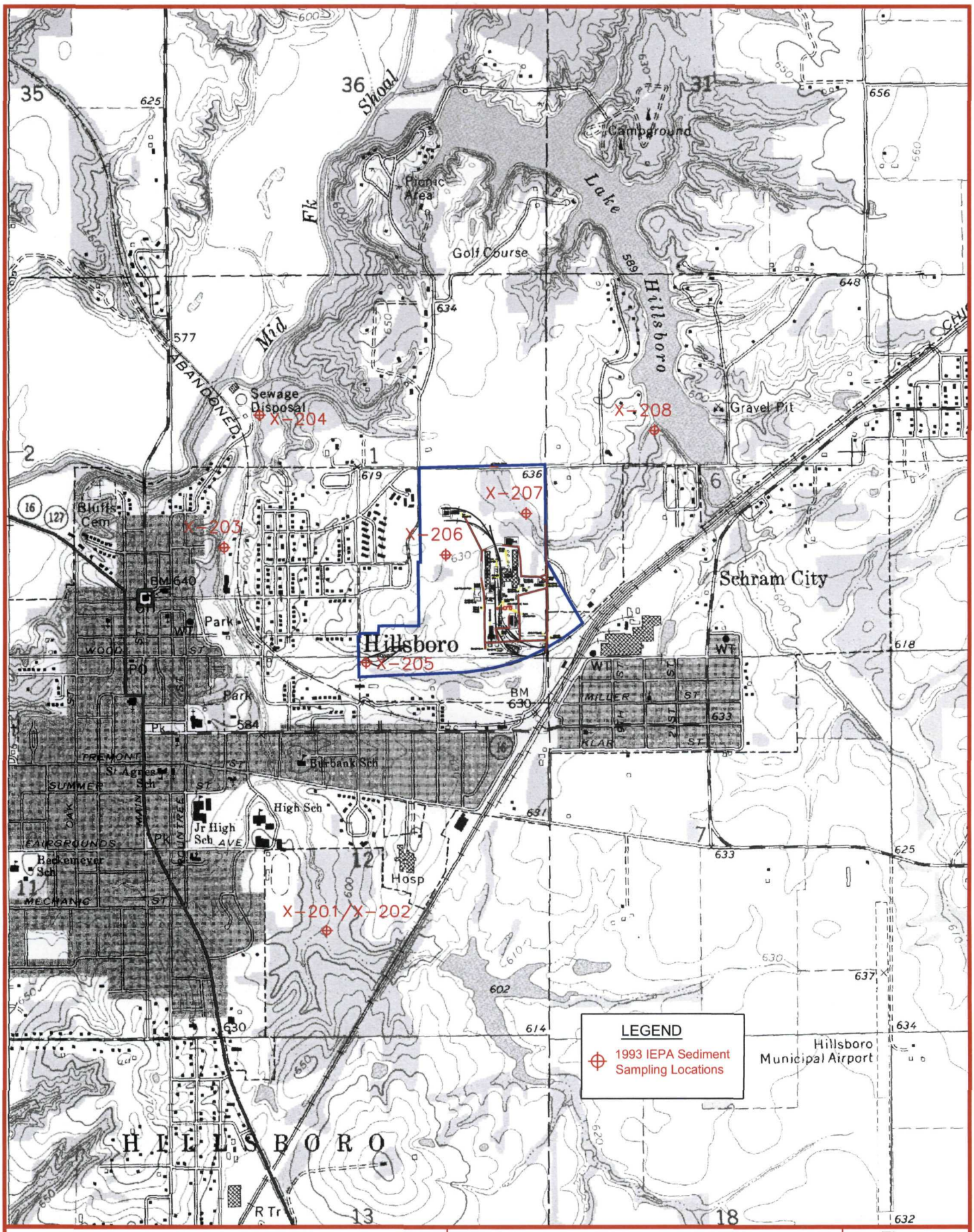

CPF\Eaige Zinc\RIFS Work Plan Report\Figures\Figure II-7

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740 Waukegan Road, Suite 401, Deerfield, IL 60015

Previous Sampling Locations
Sediments
Eagle Zinc, Hillsboro, Illinois

Figure
II-7

Drafter:	CJG	Date:	06/05/02	Contract Number:	21-7400E	Approved:	Revised:

Approximate Location of Piles identified in undeveloped areas

Rotary Residue Type 1

Rotary Clean Out

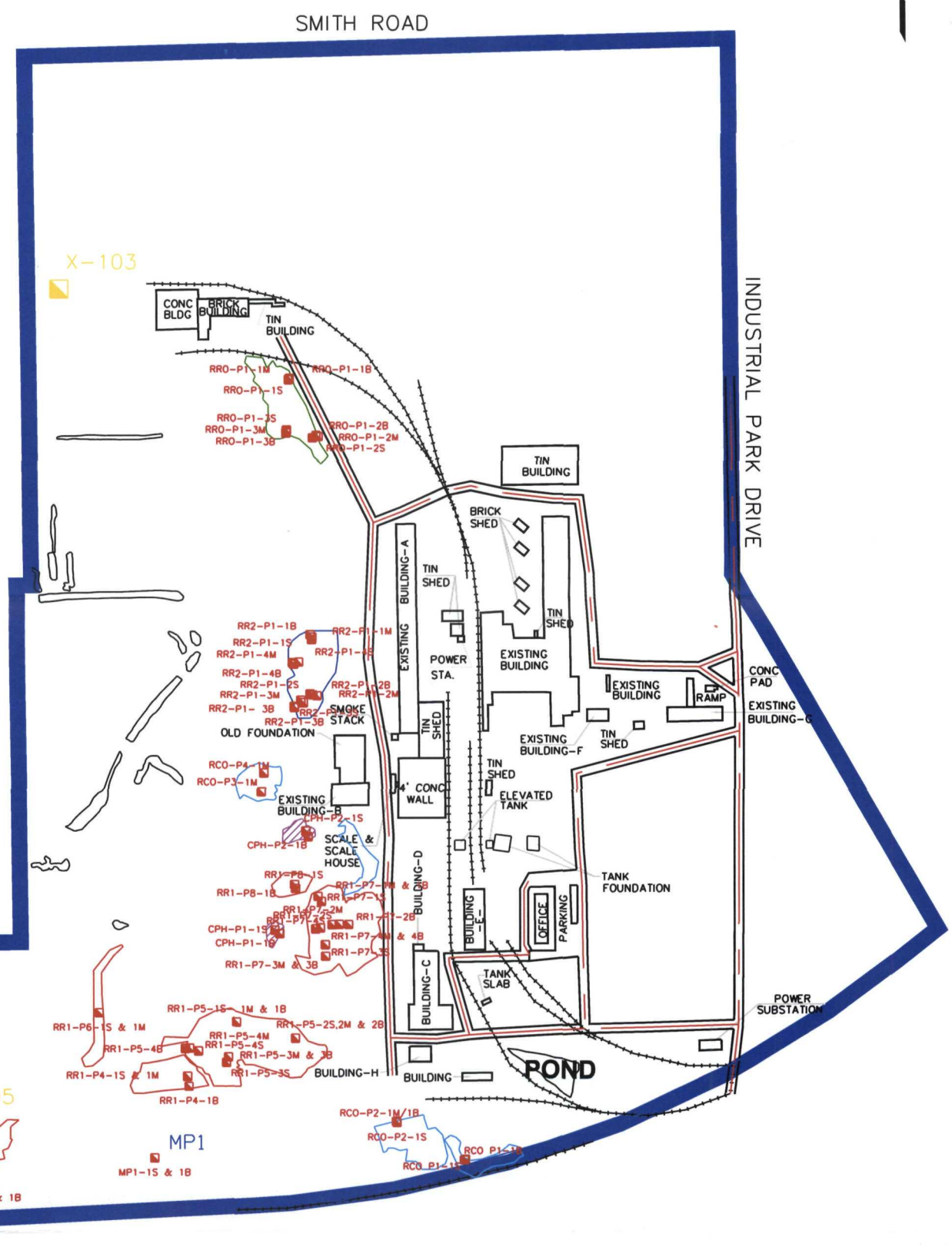
Rotary Residue Oversize

Carbon Plant Hutch

Rotary Residue Type 2

MP1 Miscellaneous Piles

Note: Extent of Piles at time of July 1998 residue sampling



LEGEND

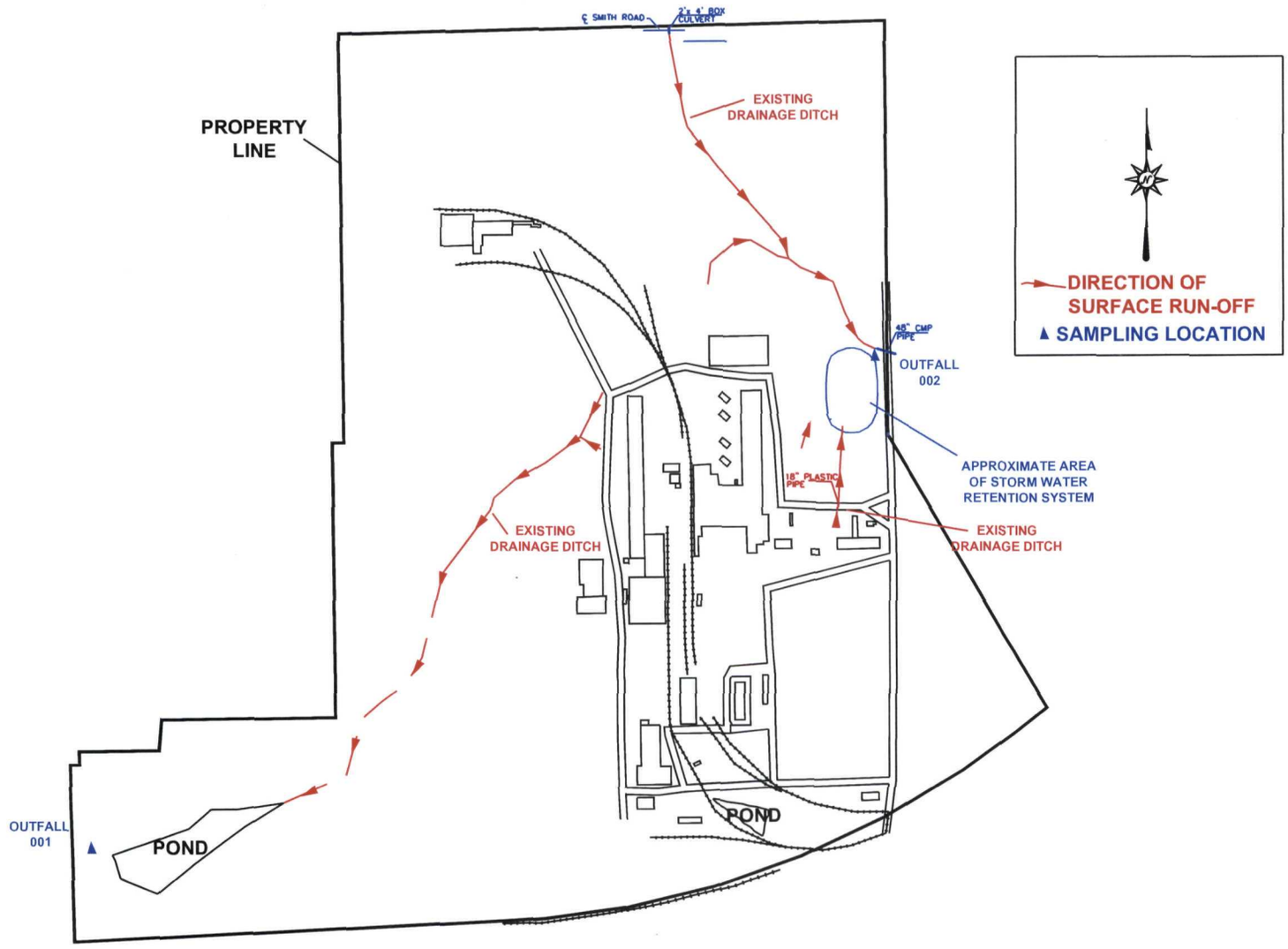
1993 Residue Pile Samples

1998 Residue Pile Samples

CPF\Eaige Zinc\RIFS Work Plan Report\Figures\Figure II-8

ENVIRON 740 Waukegan Road, Suite 401, Deerfield, IL 60015	Previous Sampling Locations Residues Eagle Zinc, Hillsboro, Illinois	Figure II-8

Drafter: CJG Date: 06/05/02 Contract Number: 21-7400e Approved: Revised:



CPF\Eaige Zinc\RIFS Work Plan Report\Figures\Figure II-9

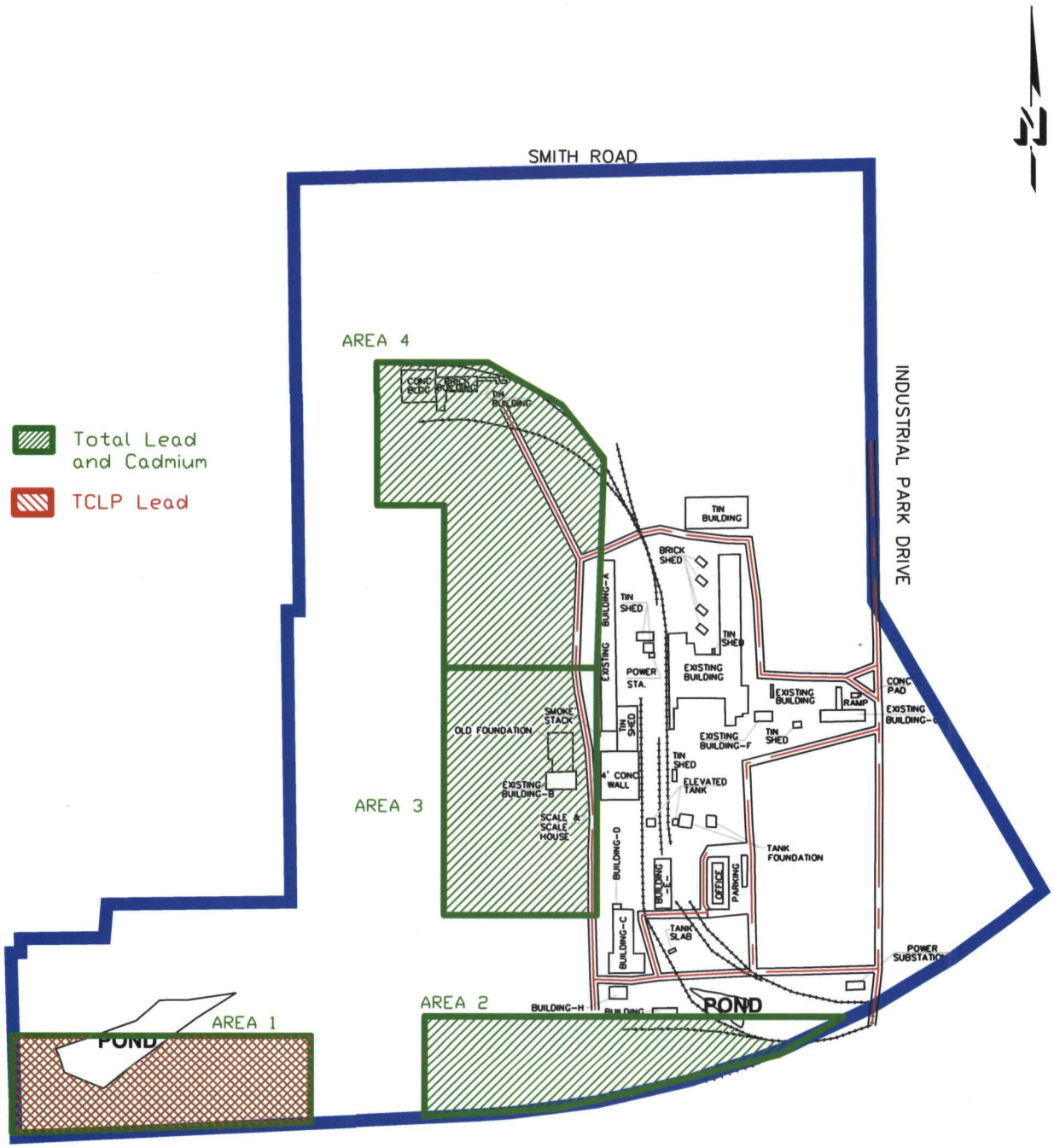
ENVIRON

740 Waukegan Road, Suite 401, Deerfield, IL 60015

Previous Sampling Locations
Storm Water
Eagle Zinc, Hillsboro, IL

Figure
II-9

Drafter:	CJG	Date:	06/05/02	Contract Number:	21-7400E	Approved:	Revised:



ENVIRON

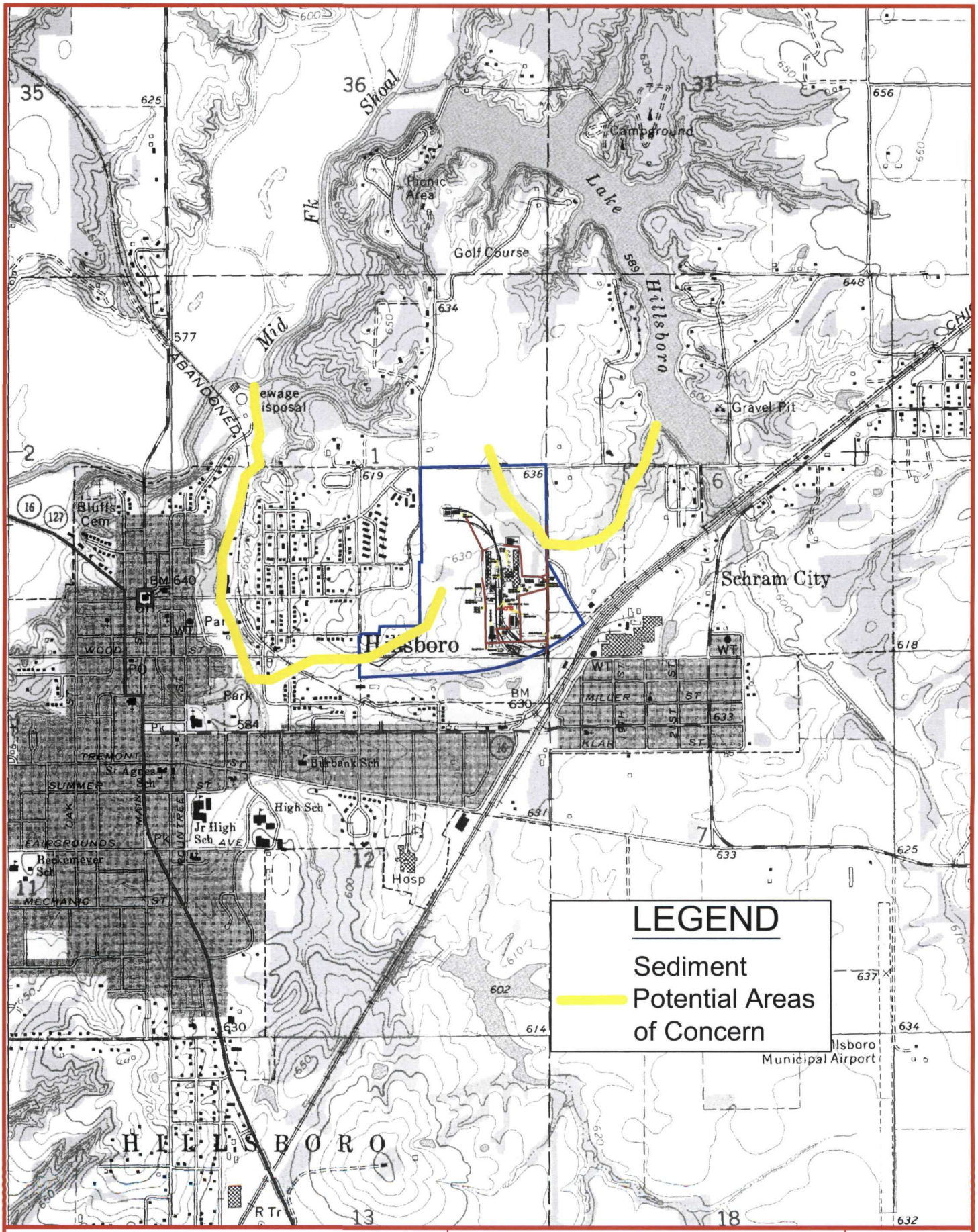
740 Waukegan Road, Suite 401, Deerfield, IL 60015

Potential Areas of Concern
On-Site Soil
Eagle Zinc, Hillsboro, Illinois

Figure
II-10

Drafter: CJG | Date: 06/05/02 | Contract Number: 21-7400E | Approved: | Revised:

CPF\Eagle Zinc\RIFS Work Plan Report\Figures\Figure II-10



ENVIRON

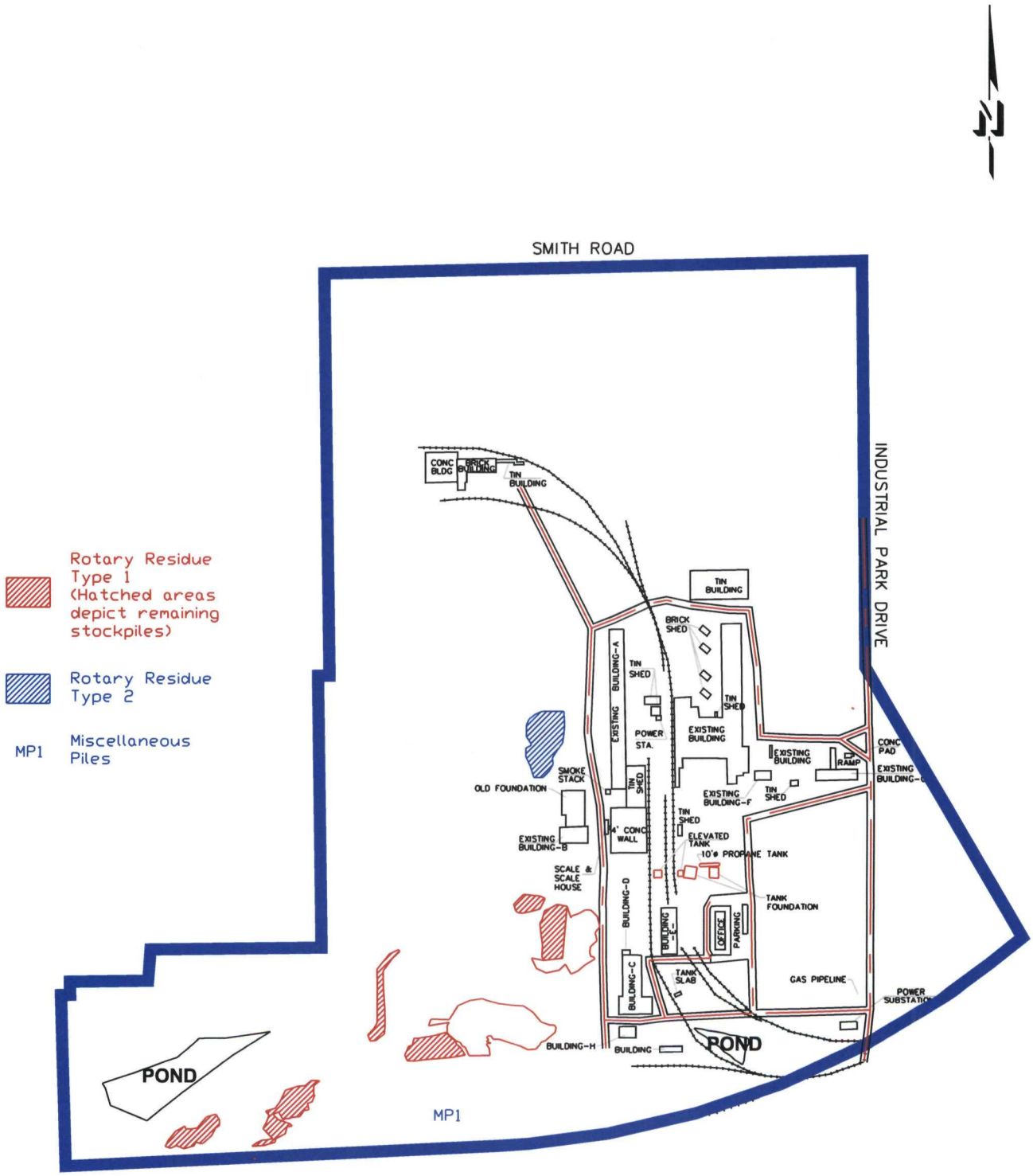
740 Waukegan Road, Suite 401, Deerfield, IL 60015

Potential Areas of Concern
Sediment
Eagle Zinc, Hillsboro, Illinois

Figure
II-11

Drafter: CJG | Date: 06/05/02 | Contract Number: 21-7400E | Approved: | Revised:

CPF\Eaige Zinc\RIFS work Plan Report\Figures\Figure II-11



ENVIRON

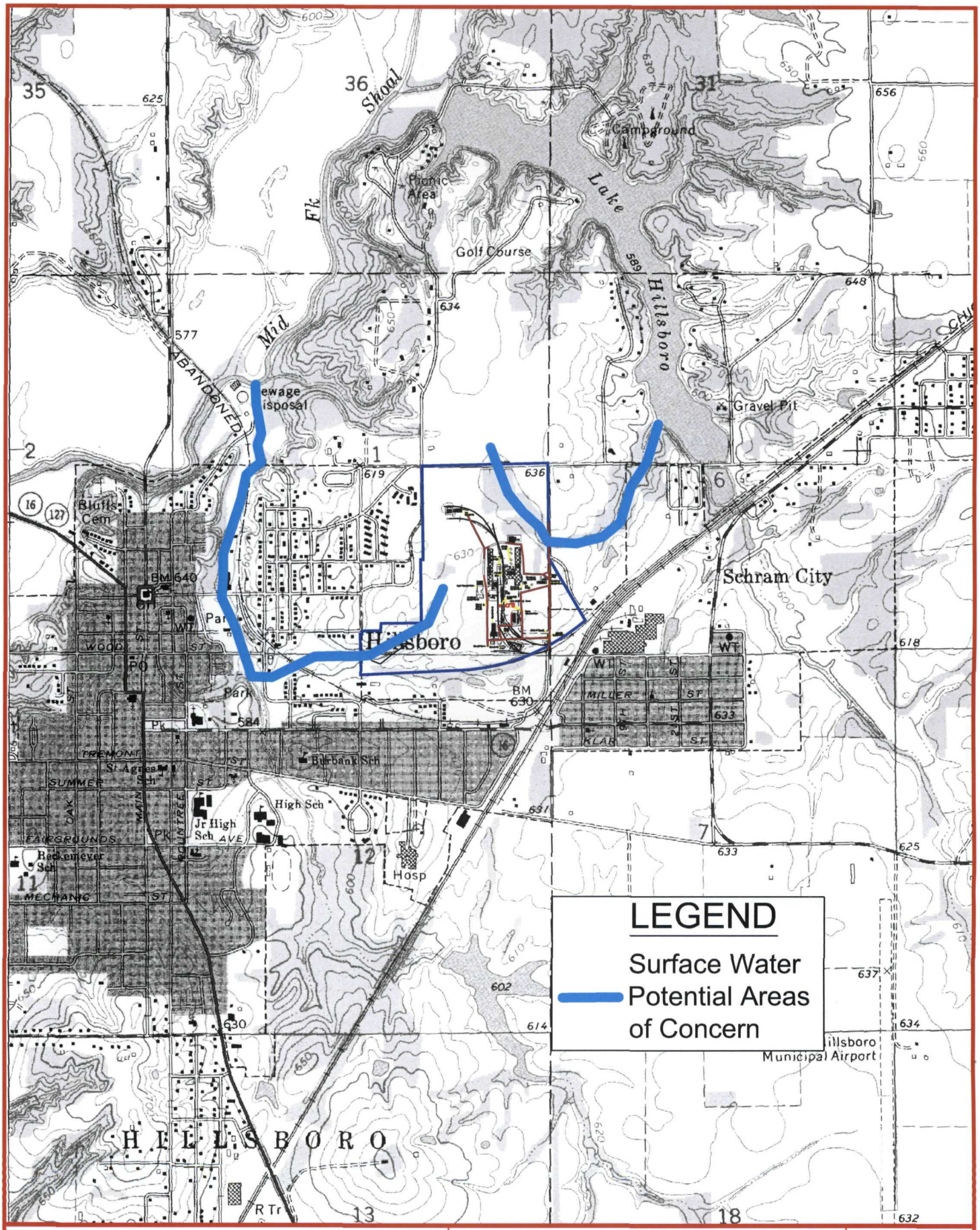
740 Waukegan Road, Suite 401, Deerfield, IL 60015

Potential Areas of Concern Residues
Eagle Zinc, Hillsboro, Illinois

Figure II-12

Drafter: CJG | Date: 06/05/02 | Contract Number: 21-7400E | Approved: | Revised:

CPF\Eaige Zinc\RFS Work Plan Report\Figures\Figure II-12



ENVIRON

740 Waukegan Road, Suite 401, Deerfield, IL 60015

Potential Areas of Cocern
Surface Water
Eagle Zinc, Hillsboro, Illinois

Figure
II-13

Drafter: CJG | Date: 06/05/02 | Contract Number: 21-7400E | Approved: | Revised:

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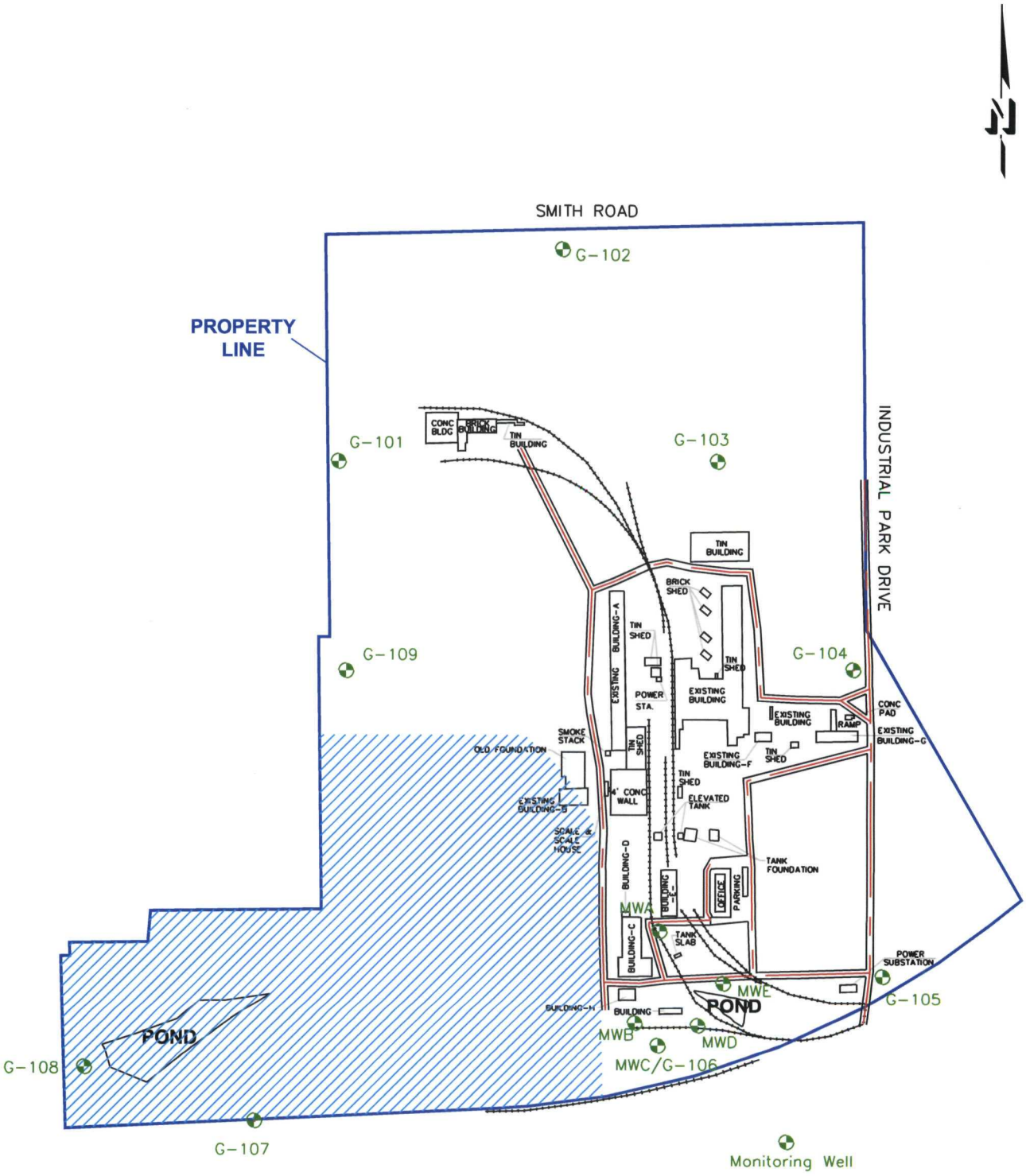


ENVIRON

740 Waukegan Road, Suite 401, Deerfield, IL 60015

Potential Areas of Concern
Ground Water
Eagle Zinc, Hillsboro, Illinois

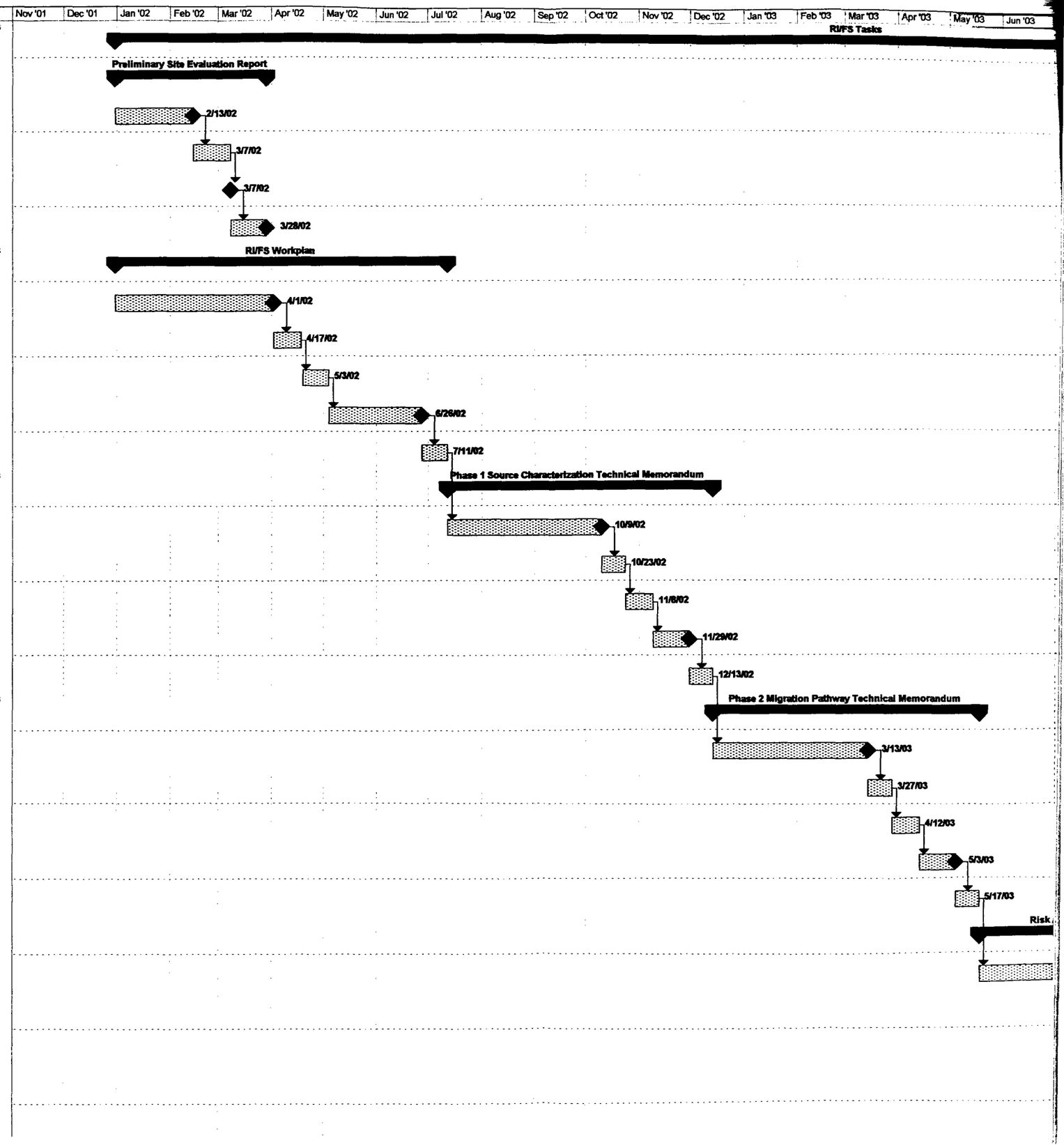
Figure
II-14

Drafter: CJG | Date: 06/05/02 | Contract Number: 21-7400E | Approved: | Revised:

CPF\Eagle Zinc\RIFS Work Plan Report\Figures\Figure II-14

Figure II-15
EAGLE ZINC -RI/FS SCHEDULE
EAGLE ZINC COMPANY SITE HILLSBORO, IL

ID	Task Name	Start	Finish	Duration
1	**RI/FS Tasks**	**Mon 12/31/01**	**Tue 5/11/04**	**863 days**
2	**Preliminary Site Evaluation Report**	**Mon 12/31/01**	**Thu 3/28/02**	**88 days**
3	Draft Preliminary Site Evaluation Report	Mon 12/31/01	Wed 2/13/02	45 days
4	PSE Report Review Meeting	Thu 2/14/02	Thu 3/7/02	22 days
5		Thu 3/7/02	Thu 3/7/02	0 days
6	Final PSE Report	Fri 3/8/02	Thu 3/28/02	21 days
7	**RI/FS Workplan**	**Mon 12/31/01**	**Thu 7/11/02**	**193 days**
8	Draft RI/FS Workplan	Mon 12/31/01	Mon 4/1/02	92 days
9	RI/FS Workplan Review Meeting	Tue 4/2/02	Wed 4/17/02	16 days
10	Agency Comments on Draft Draft RI/FS Work Plan	Fri 4/19/02	Fri 5/3/02	15 days
11	Final RI/FS Workplan	Sat 5/4/02	Wed 6/26/02	54 days
12	Agency Approval of Final RI/FS Workplan	Thu 6/27/02	Thu 7/11/02	15 days
13	**Phase 1 Source Characterization Technical Memorandum**	**Fri 7/12/02**	**Fri 12/13/02**	**155 days**
14	Draft Phase 1 Source Characterization Technical Memorandum	Fri 7/12/02	Wed 10/9/02	90 days
15	Phase 1Technical Memorandum Review Meeting	Thu 10/10/02	Wed 10/23/02	14 days
16	Agency Comments on Draft Phase 1 TM	Thu 10/24/02	Fri 11/8/02	16 days
17	Final Phase 1 Source Characterization TM	Sat 11/9/02	Fri 11/29/02	21 days
18	Agency Approval of Final Phase 1 TM	Sat 11/30/02	Fri 12/13/02	14 days
19	**Phase 2 Migration Pathway Technical Memorandum**	**Sat 12/14/02**	**Sat 5/17/03**	**155 days**
20	Draft Phase 2 Migration Pathway Technical Memorandum	Sat 12/14/02	Thu 3/13/03	90 days
21	Phase 2 Technical Memorandum Review Meeting	Fri 3/14/03	Thu 3/27/03	14 days
22	Agency Comments on Draft Phase 2 TM	Fri 3/28/03	Sat 4/12/03	16 days
23	Final Phase 2 Migration Pathway TM	Sun 4/13/03	Sat 5/3/03	21 days
24	Agency Approval of Final Phase 2 TM	Sun 5/4/03	Sat 5/17/03	14 days
25	**Risk Assessment Report**	**Sun 5/18/03**	**Fri 9/19/03**	**125 days**
26	Draft Risk Assessment Report	Sun 5/18/03	Wed 7/16/03	60 days
27	Risk Assessment Report Review Meeting	Thu 7/17/03	Wed 7/30/03	14 days
28	Agency Comments on Draft Risk Assessment Report	Thu 7/31/03	Fri 8/15/03	16 days
29	Final Risk Assessment Report	Sat 8/16/03	Fri 9/5/03	21 days
30	Agency Approval of Final Risk Assessment	Sat 9/6/03	Fri 9/19/03	14 days



APPENDIX A

Field Sampling Plan

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(continued)

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ATTACHMENTS

Attachment A-1: Standard Operating Procedures – Field Methods
Attachment A-2: Example Field Forms

FIELD SAMPLING PLAN

A. Introduction

The following Field Sampling Plan (FSP) describes the rationale and procedures for sample collection and analysis performed during the RI at the Eagle Zinc Site. Activities described in the FSP have been selected to complete the determination of the nature and distribution of contamination at the Site and in certain off-Site areas, as necessary to evaluate risk to human health and the environment.

B. Existing Data

Sampling of soil, sediment, residues, ground water, and storm water has been conducted at on-Site and off-Site locations on several occasions since the early 1980s. Those investigations and their findings are summarized in the PSE Report and in Section I of this work plan. Section I includes an evaluation of the historical sampling data using conservative screening criteria and identifies Potential Areas of Concern (PAOCs) and Potential Contaminants of Concern (PCOCs) for each medium investigated.

C. Sample Location And Frequency

The sampling and analysis program for the RI is described in Sections II.B.1 and II.B.2 and in Table A-1. The proposed locations for sediment, residue, surface water, and ground water samples are shown on Figures A-1 through A-5, respectively. The sample locations and frequencies are discussed further below. Sampling equipment and procedures are discussed in Section.E below.

1. Soil Sampling Locations

As discussed above, 130 shallow soil borings will be completed at the Site: 100 borings located in Areas 1 through 4, and 10 soil borings located in each of the manufacturing, northern and western areas of the Site. The soil areas are shown on Figure A-1. As discussed in the PSE Report, historical site activities conducted within each of these seven areas appear to have been relatively consistent over time, suggesting that a random sampling approach is appropriate. As such, proposed soil

sample locations for metals were developed as follows. First, an orthogonal grid was placed over each of the seven areas to be investigated. The grid spacing for each area was determined based on guidance used for RCRA closures in Illinois (IEPA, 1994). This method determines the grid interval using the following equation:

$$GI = (A\pi/GL)^{0.5}$$

Where:

GI= grid interval
A= area to be gridded (square feet)
GL = greatest length of the area to be gridded (feet)

Using this equation, the following grid spacing was determined:

Area 1: 30 feet
Area 2: 30 feet
Area 3: 40 feet
Area 4: 40 feet
Northern Area: 50 feet
Western Area: 40 feet
Manufacturing Plant Area: 50 feet

The sampling grids are shown on Figures A-1a through A-1g. Soil boring locations were selected using the random number generator program contained in the Microsoft Excel program. The randomly selected soil boring locations are shown on Figures A-1a through A-1g. Soil boring locations that occurred within the footprint of a building were moved to the closest grid node located outside the building.

The randomly selected soil borings will be located and staked out in the field by the surveyor. For locations that are determined to be inaccessible for drilling (e.g., located on a residue pile or within a ditch), the soil boring location will be moved the minimum distance required to complete the soil boring.

Based on the XRF screening results, 26 soil samples (20% of the soil borings) will be retained for laboratory analysis of TAL metals. All samples will be collected from soil borings exhibiting the highest estimated metals concentrations determined by XRF screening. While it is not expected that the highest metals concentrations will occur in the northern, western, or manufacturing plant areas, at least two samples will be collected from borings located in each of the seven designated soil areas for analysis of TAL metals (i.e., 14 of the 26 soil samples).

Since the current and historical processes at the Site have been limited to the manufacturing/processing of inorganic materials, and use of organic compounds, has been limited to maintenance and other support operations largely conducted inside buildings, organic contaminants are not expected in soils, particularly over the large areas planned for investigation[1]. Nevertheless, soil samples will be collected for analysis of organic constituents as follows. Each of the soil cores will be screened for organic vapors using a PID. Samples exhibiting the highest PID measurements or visual evidence of contamination potentially associated with organic constituents (e.g., staining or odors) will be retained for laboratory analysis of TCL organics and PCBs, for a total of 13 samples. A minimum of one sample will be retained for TCL organics/PCB analysis from each of the seven soil areas. Sample locations that cannot be selected based on above-background PID measurements (i.e., cannot be biased toward soils with elevated PID readings) will be randomly selected from the 26 soil sample locations already identified for TAL Metals analysis. To collect a randomly selected sample for TCL organics/PCB analysis, a second soil boring will be completed immediately adjacent to the original (randomly selected) soil boring location. The sample will be collected at the same depth as the sample retained for TAL metals analysis.

[1] Ground water in the area of a former gasoline UST is being monitored under IEPA's UST program.

If organic contaminants are identified as an issue, supplemental sampling may be necessary for further characterization.

Field duplicate samples will be collected and submitted for laboratory analysis at a rate of 1 out of 20. The field duplicates will be analyzed for TAL metals or TCL organic compounds plus PCBs, depending on the original sample analyses. Field duplicates will be collected from the samples exhibiting the greatest field evidence of potential contamination based on visual observations and sample screening using a PID and XRF analyzer. A minimum of 1 out of 20 samples will be designated as a Matrix Spike/Matrix Spike Duplicate (MS/MSD) sample.

2. Sediment Sampling Locations

Sixteen (16) sediment samples will be collected for laboratory analysis, including six (6) samples from the eastern drainageway, and ten (10) samples from the western drainageway. As shown on Figure A-2, 12 of the sediment samples are intended to investigate potential impacts from the Site, as they are downgradient of Outfalls 001 or 002 with respect to storm water discharges. In addition, 3 of the sediment samples are intended to investigate upgradient or background conditions in the drainageways. The samples will be collected as transect composites at sediment accumulation points or at representative locations. The exact locations of the sediment samples will be based in part on field observations, such as the distribution of areas of sediment accumulation in the streambed. All sediment samples will be analyzed for TAL metals. In addition, 25% of sediment samples (4 samples) will be analyzed for TCL organic compounds and PCBs.

Field duplicate samples will be collected and submitted for laboratory analysis at a rate of 1 out of 20. The field duplicates will be analyzed for TAL metals or TCL organic compounds plus PCBs, depending on the original sample analyses. Field duplicates will be collected from the samples exhibiting the greatest field evidence of potential contamination based on visual observations and sample screening using a PID. A minimum of 1 out of 20 samples will be designated as a Matrix Spike/Matrix Spike Duplicate (MS/MSD) sample.

3. Residue Sampling Locations

One gross sample will be collected from each discrete residue pile or groupings of piles of the same type. As shown on Figure A-3, 21 such piles or groups of piles have been identified. During the Phase 1 investigation, additional piles or pile groups may be identified and sampled as described below, based on their size, physical appearance, or other factors. Each gross sample will be collected as a composite of several sample increments. Based on tabulated values of the Student's "T" statistic contained in SW-846, six sample increments will be composited into a single gross sample for each pile or group of piles. The locations of the sample increments will be spaced evenly across the horizontal extent of each pile.

A test excavation or trench will be completed at each of the six sample increment locations, extending through the entire thickness of the pile. Equal-volume samples will be collected from the bucket of the excavator at three depths from within the excavation: approximately one-quarter, one-half, and three-quarter depths from the top of the excavation. The visual appearance of each sample will be logged, including color, composition, and estimated particle size(s). The three samples collected from the excavations will be mixed thoroughly to create the six sample increments. The six sample increments will then be mixed thoroughly to produce the gross sample for the pile(s). All residue samples will be analyzed for TCLP metals and SPLP metals.

Field duplicate samples will be collected and submitted for laboratory analysis at a rate of 1 out of 20. The field duplicates will be analyzed for TCLP Metals and SPLP Metals. A minimum of 1 out of 20 samples will be designated as a Matrix Spike/Matrix Spike Duplicate (MS/MSD) sample.

4. Surface Water Sampling Locations

Surface water samples will be collected from both of the major drainageways. The surface water samples will be collected as grab samples from the upper one to two inches of the water column in the ditch/stream. The ditch/stream channels are not expected to be more than a few inches deep.

The number and locations of the surface water samples will be selected based on the Phase 1 sediment sampling results, as well as field observations made at the time of sampling. At a minimum, one surface water sample will be collected immediately downstream of Outfalls 001 and 002. Surface water sampling will not be conducted during or immediately following a significant rainfall event. To the extent practicable, the surface water samples will be co-located with Phase 1 sediment sample locations. The locations of storm water drainageways to be evaluated through surface water sampling are shown on Figure A-4. As discussed above, surface water samples will be retained for analysis of TAL metals, TCL organic compounds, PCBs, or a combination of these constituent groups. Field duplicate samples will be collected and submitted for laboratory analysis at a rate of 1 out of 20. The field duplicates will be analyzed for TAL metals or TCL organic compounds plus PCBs, depending on the original sample analyses. A minimum of 1 out of 20 samples will be designated as a Matrix Spike/Matrix Spike Duplicate (MS/MSD) sample.

5. Ground Water Sampling Locations

As discussed above, the ground water investigation will include installation of 20 temporary wells, collection of ground water screening samples from a minimum of 10 temporary wells, installation of permanent monitoring wells at a minimum of 10 of the temporary well locations, abandonment of all temporary wells, and collection of a complete round of ground water samples from all newly installed and pre-existing monitoring wells (with the exception of existing wells MW-A, MW-B, MW-D and MW-E). The approximate locations of the temporary wells and the actual locations of the existing site monitoring wells are shown on Figure A-5.[2] Groundwater screening samples will be collected from 10 of the temporary wells for analysis of TAL metals. In addition, four of the temporary wells will be sampled for TCL organic compounds and PCBs.

[2] Phase I soil sampling data will be considered in the selection of actual locations for the temporary wells and the identification of temporary wells for collection of ground water screening samples.

All existing and newly installed permanent monitoring wells[3] will be sampled for TAL metals. The metals analyses will be conducted using filtered and unfiltered samples to determine total and dissolved metals concentrations. In addition, four of the ground water samples will be analyzed for TCL organic compounds and PCBs.

For the full ground water sampling round, field duplicate samples will be collected and submitted for laboratory analysis at a rate of 1 out of 20. The field duplicates will be analyzed for TAL metals or TCL organic compounds plus PCBs, depending on the original sample analyses. A minimum of 1 out of 20 samples will be designated as a Matrix Spike/Matrix Spike Duplicate (MS/MSD) sample.

D. Sample Designation

Identification of the individual sample collection locations and individual samples will be assigned as discussed in the following sections. Field duplicate samples will be identified by adding a "D" to the sample identification number. Each environmental sample collected during the RI will contain a unique alpha-numeric designation indicating the area where the sample was collected (e.g., "WD" for western drainageway), the sample location, the sample depth (if necessary), and the sample matrix. If necessary, the sample numbers will be modified to comply with any limits on the number of sample identification characters or other imposed by the laboratory.

1. Soil Samples

All soil samples will be collected within one of seven on-Site areas. These areas will be designated as follows:

Area 1: A1

Area 2: A2

Area 3: A3

Area 4: A4

[3] Excluding existing wells MW-A, MW-B, MW-D and MW-E, which have been and continue to be sampled under a UST compliance program under the oversight of IEPA.

Northern Area: NA

Western Area: WA

Manufacturing Plant Areas: MA

First, the soil matrix will be identified using an "S", followed by the appropriate area designation (e.g., "MA"). Next, each soil boring/sampling location will be assigned a two-digit number, such as 05. Finally, the soil sample depth in feet below the ground surface will be designated, representing the base of the sample interval (e.g., 3.5 would be used for a sample interval of 3.0 to 3.5 feet). As an example, soil sample number five collected in the manufacturing plant area at a depth of 2.5 feet would be designated S-MA-05-2.5 and its field duplicate would be designated S-MA-05-2.5D.

2. Sediment Samples

All sediment samples will be collected within one of the two major drainageways that receive runoff from the Site or their tributaries. These areas will be designated as follows:

Western Drainageway: WD

Eastern Drainageway: ED

The sediment matrix will be identified using an "SD", followed by the area designation (e.g., "WD"). Similar to the soil samples, the sediment sampling locations will be assigned a two-digit number, such as 05. It is assumed that all sediment samples collected during Phase 1 of the RI will be from the surface of the sediments; therefore, a depth designation is not required. As an example, sediment sample number five collected in the western drainageway would be designated SD-WD-05 and its field duplicate would be designated SD-WD-05D.

3. Residue Samples

All residue samples will be categorized as consisting of one of the six residue types, based on the previously established residue pile categories. The residue types include:

Rotary Residue Type 1: RR1
Rotary Residue Type 2: RR2
Rotary Clean Out: RCO
Rotary Residue Oversize: RRO
Carbon Plant Hutch: CPH
Miscellaneous Piles: MP

First, the matrix identifier "R" will be used followed by the residue type designation (e.g., RR1). A unique sample location number will then be assigned to the pile of group of piles sampled within the residue type category.[4] As an example, residue sample number two collected from a RRI stockpile would be designated R-RR1-02 and its field duplicate would be designated R-RR1-02D.

4. Surface Water Samples

All surface water samples will be collected within one of three drainageways that receive runoff from the Site. These areas and their sample designations are as follows:

Western Drainageway: WD
Eastern Drainageway: ED

The surface water sampling locations will be designated using a two-digit number, such as 05. No depth designation will be used, as all surface water samples will be collected at or immediately below the surface of the water bodies,

[4] The location and depth of the sample increments and depth composites used to create the gross sample will not be represented in the identification numbers for the residue samples.

which consist of shallow drainage ditches and streams. The surface water matrix will be identified using a "SW". As an example, surface sample number five collected in the western drainageway would be designated SW-WD05 and its field duplicate would be designated SW-WD05D.

5. Ground Water Samples

Ground water samples will be collected from newly installed temporary wells and permanent monitoring wells, as well as pre-existing monitoring wells G-101 through G-109. Therefore, the following sample type designations will be used:

Newly Installed Temporary Wells: TW
Newly Installed Permanent Monitoring Wells: MW
Pre-existing G-Series Monitoring Wells: G

The ground water sampling locations will be designated using the two- or three digit well number, such as 101 for the existing G-series wells and 05 for a newly installed temporary or permanent well. No depth designation will be used, as all ground water samples will be collected at or immediately below the piezometric level measured in the well following purging and stabilization. The six-digit sample collection date (year/month/day) will also be included in the sample identification number. As an example, a ground water sample collected from existing monitoring well G-106 on October 8, 2002 would be designated G106-021008 and its field duplicate would be designated G106-021008D.

6. Quality Assurance/Quality Control Samples

Samples designated for quality assurance/quality control analysis will be designated with the 6-digit sample date (year/month/day). In addition, the prefix MS/MSD will be used for matrix spike/matrix spike duplicate samples. Trip blanks placed in each shipment of aqueous samples to be analyzed for VOCs will be designated with the prefix TB and will be numbered sequentially starting with the

number 1. For example, a trip blank with the fourth shipment that is analyzed on June 1, 2002 would be identified as TB-04-020601.

E. Sampling Equipment And Procedures

A list of field equipment for each sampling program is provided in Table A-2. Detailed Standard Operating Procedures (SOPs) for field screening, soil sampling, surface water sampling, sediment sampling, well development, and ground water sampling are provided in Attachment A-1 to the FSP.

1. Soil Sampling

Soil borings performed during the Phase 1 investigation will be advanced using a direct-push drilling apparatus (e.g., Geoprobe) equipped with 4-foot-long, 2-inch outside diameter macro-core samplers with dedicated polyethylene liners. All soil borings will be sampled continuously from the ground surface to the completion depth. The completion depth will either be 4 feet below ground surface (bgs) or two feet below the depth at which undisturbed[5] native soils are encountered, whichever is deeper. An experienced ENVIRON field geologist or engineer will prepare a geological log for each soil boring. Field screening for volatile organic vapors will be conducted using a portable photoionization detector (PID) immediately after sample retrieval. In addition, each soil core will be screened for metals concentrations using a hand-held XRF analyzer. A PID and XRF measurement will be made from each recovered 1 foot interval of the native soil. As described in Section C.1 above, based on the field screening results, one soil sample will be retained from 20% of soil borings for analysis of TAL metals at a fixed-base laboratory. In addition, one analytical sample will be retained from 10% of the soil borings for analysis of TCL organic compounds and PCBs. The analytical samples will be collected from the uppermost one-foot interval of native soils exhibiting undisturbed characteristics. Samples for the fixed-based laboratory will be collected first, followed by QA/QC samples (Field Duplicate and MS/MSD).

[5] Includes soils exhibiting no visually observable evidence of disturbance or mixing with surfical materials, such as Plant residues.

2. Sediment Sampling

The following procedures will be used to collect sediment samples for laboratory analysis. Surface water flow in the upper reaches of the drainageways (i.e., those segments located on-site or close to the site) is intermittent. A sample of the stream sediment will be obtained using a sediment sampler for locations where there is actual water flow, or using stainless steel sampling tools for locations where the portion of the stream to be sampled is dry. Each sample retained for analysis of TAL Metals will be collected as a composite of three equally spaced/equal volume increments collected across the streambed at the selected sampling location. Samples retained for analysis of TCL organics/PCBs will be collected as grab samples based on field screening results and visual observations. All samples will be collected from the uppermost six inch interval. Upon completion of sampling, a stake or flag will be placed on the stream bank parallel to the sample location for future reference. In addition, the geographic coordinates of each sediment sample location will be logged using a hand-held Global Positioning System (GPS) unit. Where practicable, the sampling will proceed from downstream to upstream so that any disturbed sediment does not impact subsequent sampling.

3. Residue Sampling

Samples will be collected from each discrete residue pile or group of similar piles as discussed in Section C.3 above (as composites from trenches excavated to the base of the piles). All sample mixing will be conducted in a clean 5-gallon bucket. The appropriate sample volumes will be collected from the composited material for analysis of TCLP metals and SPLP metals. Any necessary particle size reduction will be performed at the laboratory.

4. Surface Water Sampling

The surface water samples will be collected as grab samples from the central portion of water flow by submerging the sample container with the open end facing upstream. For samples containing a preservative or fixing agent, the samples will be collected using a laboratory-cleaned glass sample jar and immediately

transferred to the proper sample container. Sample collection will be done in such a way that sediments are not disturbed prior to or during the collection of the surface water sample. If practicable, the sampling will proceed from downstream to upstream so that any disturbed sediment does not impact subsequent sampling. Upon completion of sampling, a stake or flag will be placed on the stream bank parallel to the sample location for future reference. In addition, the geographic coordinates of each surface water sample location will be logged using a hand-held Global Positioning System (GPS) unit.

5. Ground Water Investigation

The temporary monitoring wells will be installed using a direct-push drilling apparatus as described for the soil sampling program. Continuous 2-inch outside diameter macro-cores will be collected continuously to an appropriate depth below the top of the saturated zone and soil logs will be prepared by the field geologist/engineer. A one-inch diameter section of PVC screen and riser pipe will be placed in the core hole and a clean sand filter pack will be placed around the well screen. Ground water screening samples will be collected from temporary wells using a small-diameter bailer or a peristaltic pump. In addition, water level measurements will be collected from all temporary wells and converted to water level elevations using surveyed benchmarks at the top of the wells. Following the determination of locations for installation of permanent monitoring wells, all temporary wells will be abandoned by removing the PVC, returning the soil cores to the borehole, and sealing the remainder of the borehole with granular bentonite.

The permanent monitoring wells will be installed near selected temporary wells using a hollow-stem auger drilling rig. Continuous split-spoon samples will be collected at five-foot intervals from the ground surface to the completion depth of the soil boring and will be logged prepared by the field geologist/engineer. A 2-inch inside diameter section of schedule-40 PVC screen and riser pipe will be placed in the borehole and a clean sand filter pack will be placed around the screened interval to a depth of one foot above the top of the well screen. A one-foot thick bentonite seal will then be placed in the annular space of the well. Finally, the

well will be grouted to the surface with an appropriate mixture of Portland cement and bentonite. The permanent wells will be completed with a stick-up type protective casing with a locking cap.

Development of the permanent monitoring wells will be performed to allow for the collection of representative ground water samples. All monitoring wells will be developed no sooner than 12 hours following well installation. Well development will consist of the removal of a minimum of three times the measured casing volume of water plus three times the saturated volume of the monitoring well sand pack. Field-measured parameters of pH, specific conductance, dissolved oxygen, and temperature will be recorded at each well volume. Well development will be considered complete when these field-measured parameters have stabilized. Both visual clarity and field-measured parameters will be used to evaluate effectiveness of well development.

As discussed above, a full round of ground water samples will be collected following development of the newly installed permanent monitoring wells. If adequate water is present, a peristaltic pump will be used to purge and sample the monitoring wells. If adequate water is not available, a bailer will be used to purge and sample monitoring wells. During well purging, measurements for field parameters (pH, specific conductance, temperature, and dissolved oxygen) will be made. When using a peristaltic pump, continuous monitoring of these samples may be made using a flow-through sampling cell. Should it be necessary to use a bailer for purging, samples will be collected on a periodic basis during purging for field parameter measurement. The wells will be considered purged when a minimum of three times the measured casing volume has been removed, and the field-measured parameters of pH, specific conductance, and temperature have stabilized. If a well bails dry during purging, it will be considered purged and sampling will commence once a sufficient amount of water has recharged in the well. If non-dedicated sampling equipment is used, decontamination procedures described in the following section will be used for decontamination of the bailers.

Filtered and unfiltered samples collected from the permanent monitoring wells will be submitted to the laboratory for metals analyses. Field filtering will be

conducted using dedicated 0.45-micron filters either in line with the pump tubing or as an attachment to a bailer.

During the well sampling program, an electronic water level meter will be used to synoptically measure the depth to the surface of ground water within all sites monitoring wells. The measurements will be collected from a surveyed and marked location at the top of the well casing or from the north edge of the well casing if a surveyed mark is absent. The ground water measurements will be made to the nearest one hundredth (0.01) of a foot.

6. Decontamination Procedures

Dedicated sampling equipment and monitoring well construction materials which have been pre-cleaned or have been prepackaged will not require decontamination prior to use. Sample containers will be pre-cleaned as specified in the QAPP. All non-dedicated sampling equipment will require decontamination prior to its use.

Equipment will be decontaminated by either steam cleaning with a high-pressure washer or washing in a low phosphate detergent wash such as Alconox™ followed by a rinse with municipal water and a final distilled water rinse.

F. Sample Handling And Analysis

As soon as sample labeling is completed, each sample will be placed in an insulated container (cooler) that contains sufficient ice to lower the sample temperature to between 4° C and 6° C.

1. Sample Containers, Preservation Methods, and Volume Requirements

This information is included in the QAPP (Appendix B to RI/FS Work Plan).

2. Shipping Methods

This information is included in the QAPP (Appendix B to the RI/FS Work Plan).

3. Instructions and Samples for Filling Out Paperwork

This information is included in the QAPP (Appendix B to the RI/FS Work Plan). In addition, example field forms and instructions are included in Attachment A-2 of the FSP.

4. Handling and Disposal of Wastes

Five types of investigative-derived waste (IDW) will be generated during the course of this investigation. These IDWs are: (1) soils (hollow-stem auger and direct-push drill cuttings and excess soil samples), (2) monitoring well development water, (3) decontamination water, (4) monitoring well purge water, and (5) personal protective equipment (e.g., gloves).

Soil cores and soil samples generated from direct-push drilling will be returned to the borehole from which they were extracted. As organic contaminants are not known to be a concern for soils in either the unsaturated or saturated zone, soil cuttings generated by hollow-stem auger drilling for well installation will be spread on the ground surface at or near the well location. If field evidence of contamination is observed based on PID readings or visual observations, the drill cuttings will be placed in drums and labeled according to their source. The drums will be temporarily stored at a covered, contained area provided by Eagle Zinc Company. The contents of the drums will then be sampled for determination of hazardous waste characteristics. If determined to be non-hazardous, the waste will be disposed as a special industrial waste. If the waste is found to be hazardous, the waste will be disposed of at a permitted off-site treatment, storage and disposal (TSD) facility. Any off-site disposal of containerized solid waste will occur following Phase I of the RI, within the time frames specified by applicable regulations (e.g., within 90 days for hazardous waste).

Wastewater from equipment decontamination, well development, and well purging will be containerized. The analytical results for the ground water samples will be used to determine the disposition of these wastewaters. Disposal options include placement of the water on the ground in the area of the monitoring well from which it was purged (i.e., returning the water to the shallow aquifer) or off-

Site disposal as a nonhazardous waste. Off-site disposal of investigation-derived wastewater will occur immediately following Phase II of the RI.

While it is unlikely that waste personal protective equipment (PPE) generated during the site activities have the potential to accumulate significant amounts of hazardous waste due to the nature of the materials, soiled PPE will be placed in a drum for off-site disposal as a special waste.

APPENDIX A
TABLES

Table A-1
Summary of Sampling and Analysis Program

Sampling Area	*Matrix*	*Field Parameters*	*Analyses*	*SW-846 Laboratory Methods*	*Number of Samples Investigative Samples (Estimated)*	*Duplicates*	*Trip Blanks*	*MS/MSD*
On-Site areas 1-4, undeveloped site areas, manufacturing area	Soil	Visual, PID Screening, XRF	XRF	--	130	1/20		1/20
			TAL metals	6010/6020	26			
			TCL organics	8260B/8270C	13			
			PCBs	8082	13			
Residual Piles	Residue	Visual, PID	TCLP metals	1312/6010/6020	20	1/20		1/20
			SPLP metals	1311/6010	20			
Eastern and Western Drainageways	Sediment	Visual, PID	TAL metals	6010/6020	16	1/20		1/20
			TCL organics	8260B/8270C	4			
			PCBs	8082	4			
	Surface Water	Visual	TAL metals	6010/6020	15	1/20	1/shipment	1/20
			TCL organics	8260B/8270C	4			
			PCBs	8082	4			
			Sulfates	9036	15			
Site-wide	Ground Water (temporary wells)	Visual	TAL metals	6010/6020	10	1/20	1/shipment	1/20
			TCL organics	8260B, 8270C	4			
			PCBs	8082	4			
	Ground Water (permanent wells)	Visual, pH, conductivity, Temperature Dissolved Oxygen	TAL metals	6010/6020	23	1/20	1/shipment	1/20
			TCL organics	8260B,8270C	4			
			PCBs	8082	4			
			Sulfates	9036	23			

Key:

PID = Photoionization Detector
XRF = X-Ray Fluorescence Analyzer
PCOCs = Potential Contaminants of Concern for Groundwater (Cadmium, Lead, Manganese, Zinc, Iron, and Sulfate)
MS/MSD = Matrix Spike/Matrix Spike Duplicate

Table A-2
Field Equipment

Activity	*Field Equipment*
Soil Sampling	Stainless steel spatula/spoon
	Mixing bowl
	PID
	XRF
	Marking flags/stakes
	Ruler/Tape
	PPE
Sediment Sampling	Stainless steel spatula/spoon
	Mixing bowl
	PID
	XRF
	Marking flags/stakes
	PPE
Residue Sampling	Stainless steel spatula/spoon
	Soft sediment sampler
	Mixing bowl
	PID
	Marking flags/stakes
	PPE
Surface Water Sampling	Open sample container (laboratory cleaned)
	Marking flags/stakes
	PPE
Well installation/ground water sampling	Teflon bailers/cord
	Well pumps
	Flow thru meter for field measurements
	Water level indicator
	Drums to contain development/purge water
	PID
	PPE

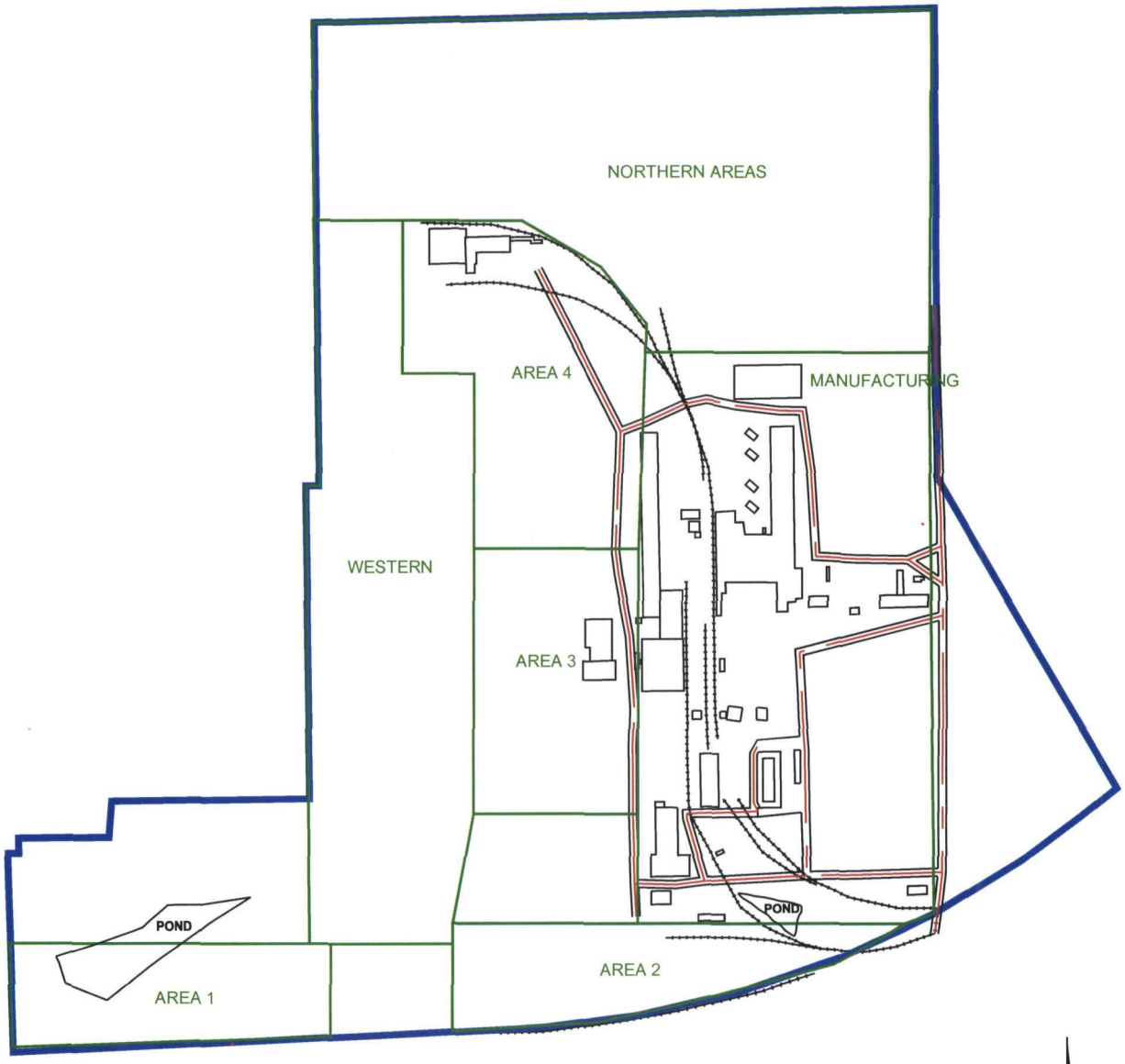
Key: PPE = Personnel Protective Equipment
PID = Photoionization Detector
XRF = X-Ray Fluorescence Analyzer

Table A-2
Field Equipment

Activity	*Field Equipment*
Decontamination	Alconox™
	Brushes
	Distilled Water
	Paper Towels
	Tap Water
	Water containers
	PPE
Miscellaneous	Instrument calibration equipment
	Field book
	Sample coolers
	Packing and shipping material
	Field forms and logs
	Permanent markers

Key: PPE = Personnel Protective Equipment
PID = Photoionization Detector
XRF = X-Ray Fluorescence Analyzer

APPENDIX A
FIGURES



APPROX. SCALE (ft.)
0 220

ENVIRON
740 Waukegan Road, Suite 401, Deerfield, IL 60015

Soil Investigation
Areas
Eagle Zinc, Hillsboro, Illinois

Figure
A-1

Drafter: APR Date: 06/05/02 Contract Number: 21-7400E Approved: Revised:

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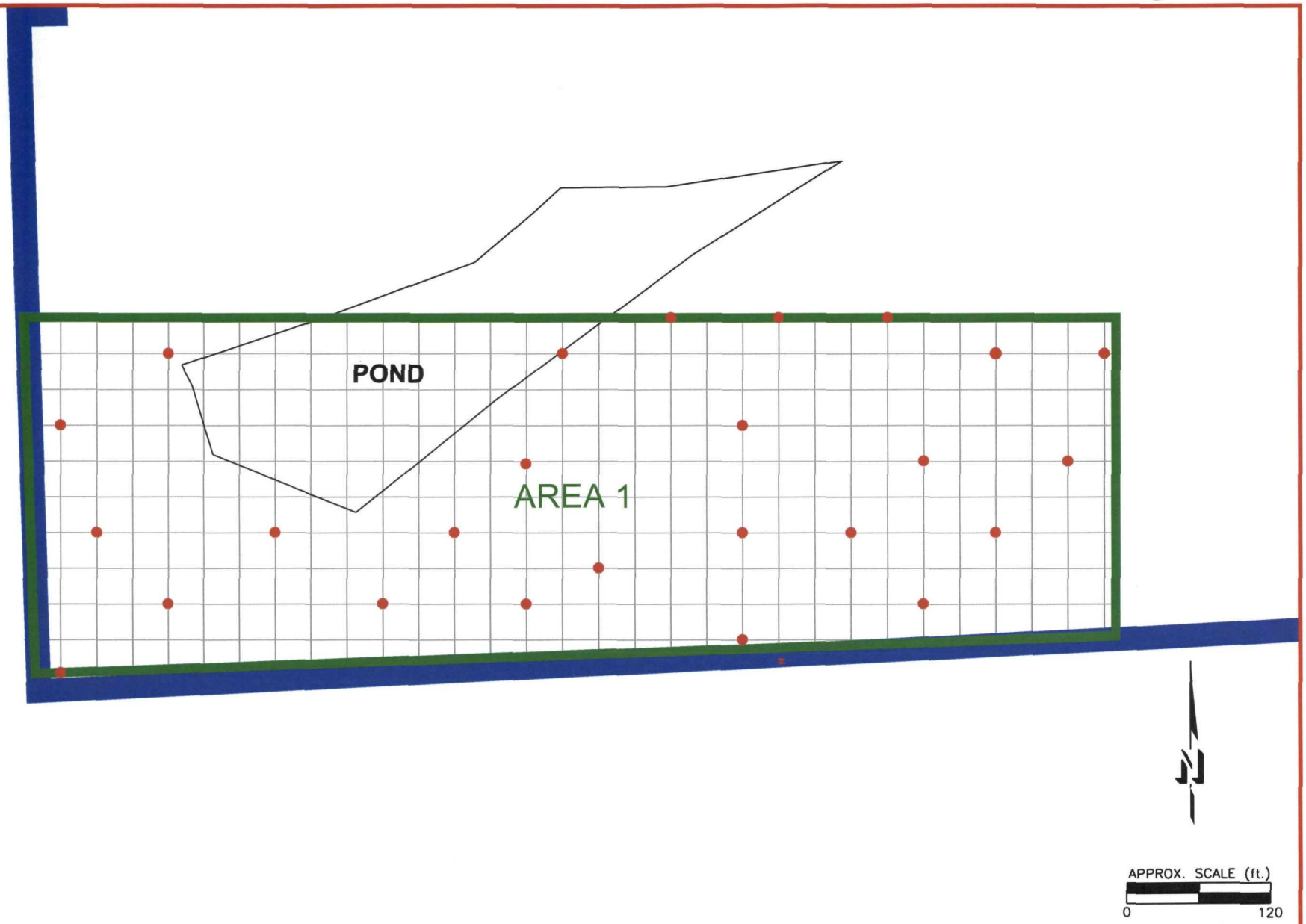

ENVIRON

740 Waukegan Road, Suite 401, Deerfield, IL 60015

Proposed Soil Sample Locations
Area 1
Eagle Zinc, Hillsboro, Illinois

Figure
A-1a

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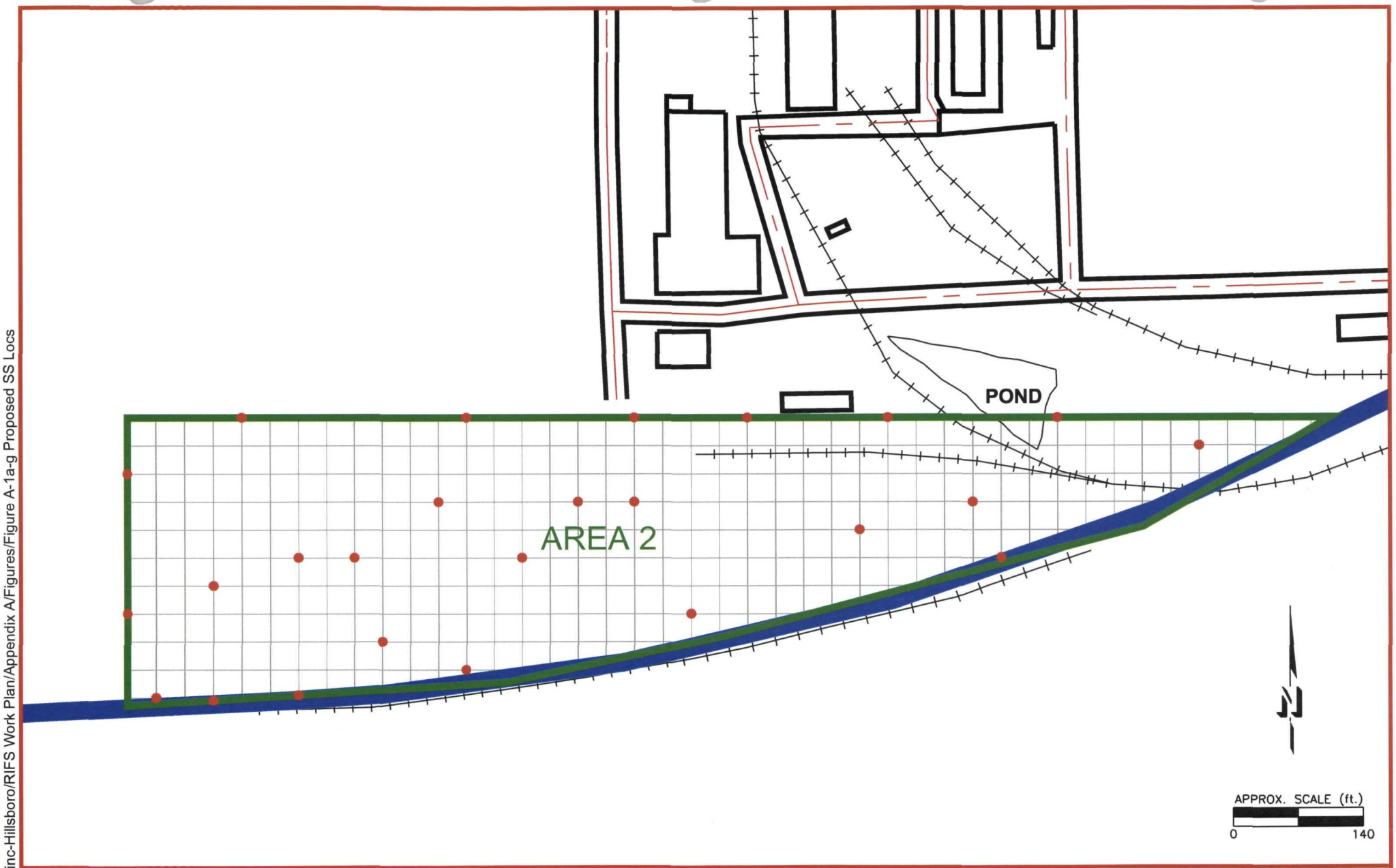

ENVIRON

740 Waukegan Road, Suite 401, Deerfield, IL 60015

Proposed Soil Sample Locations
Area 2
Eagle Zinc, Hillsboro, Illinois

Figure
A-1b

Drafter: APR | Date: 6/25/02 | Contract Number: 21-7400E | Approved: | Revised:

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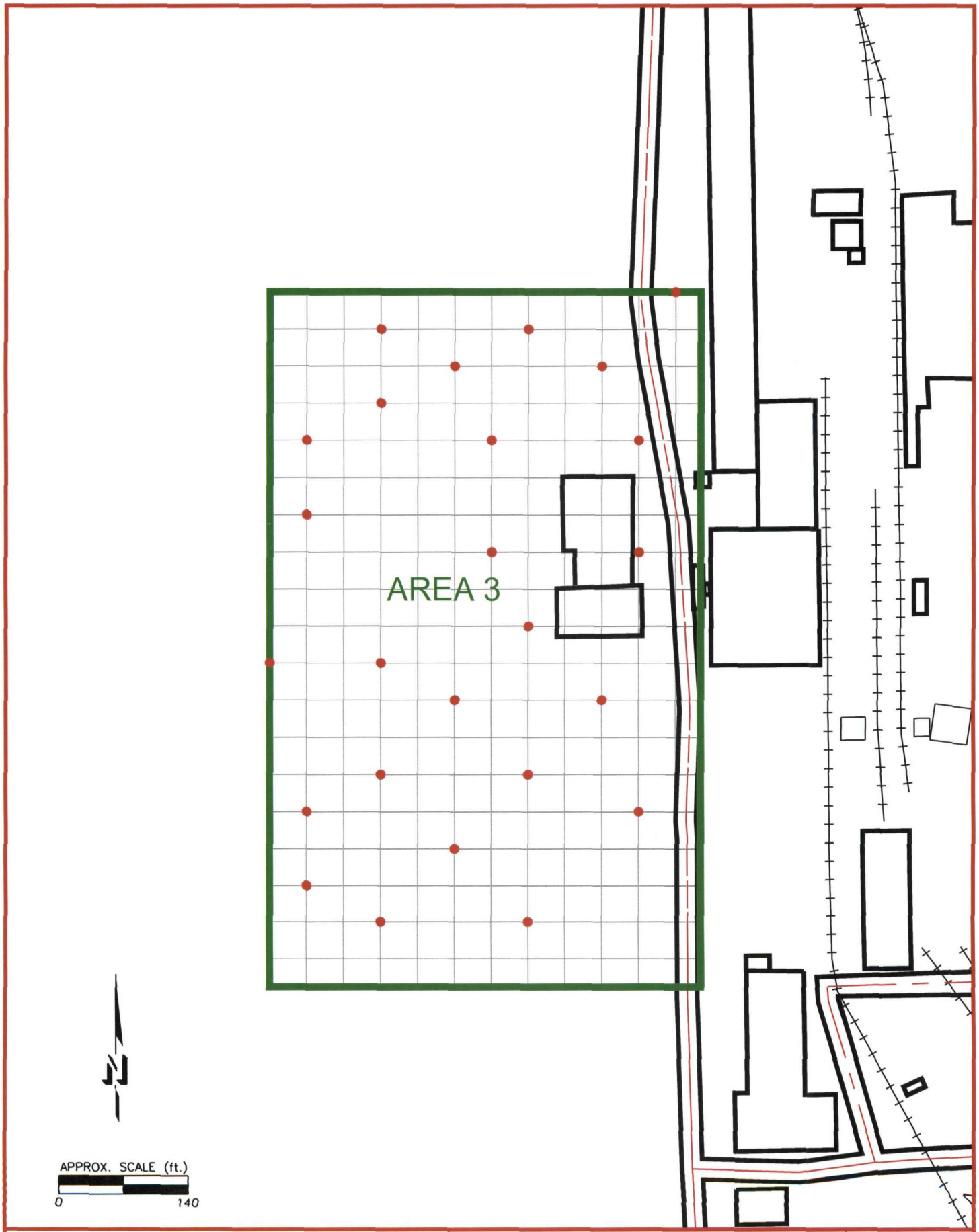

ENVIRON

740 Waukegan Road, Suite 401, Deerfield, IL 60015

Proposed Soil Sample Locations
Area 3
Eagle Zinc, Hillsboro, Illinois

Figure
A-1c

Drafter: APR | Date: 6/25/02 | Contract Number: 21-7400F | Approved: | Revised:

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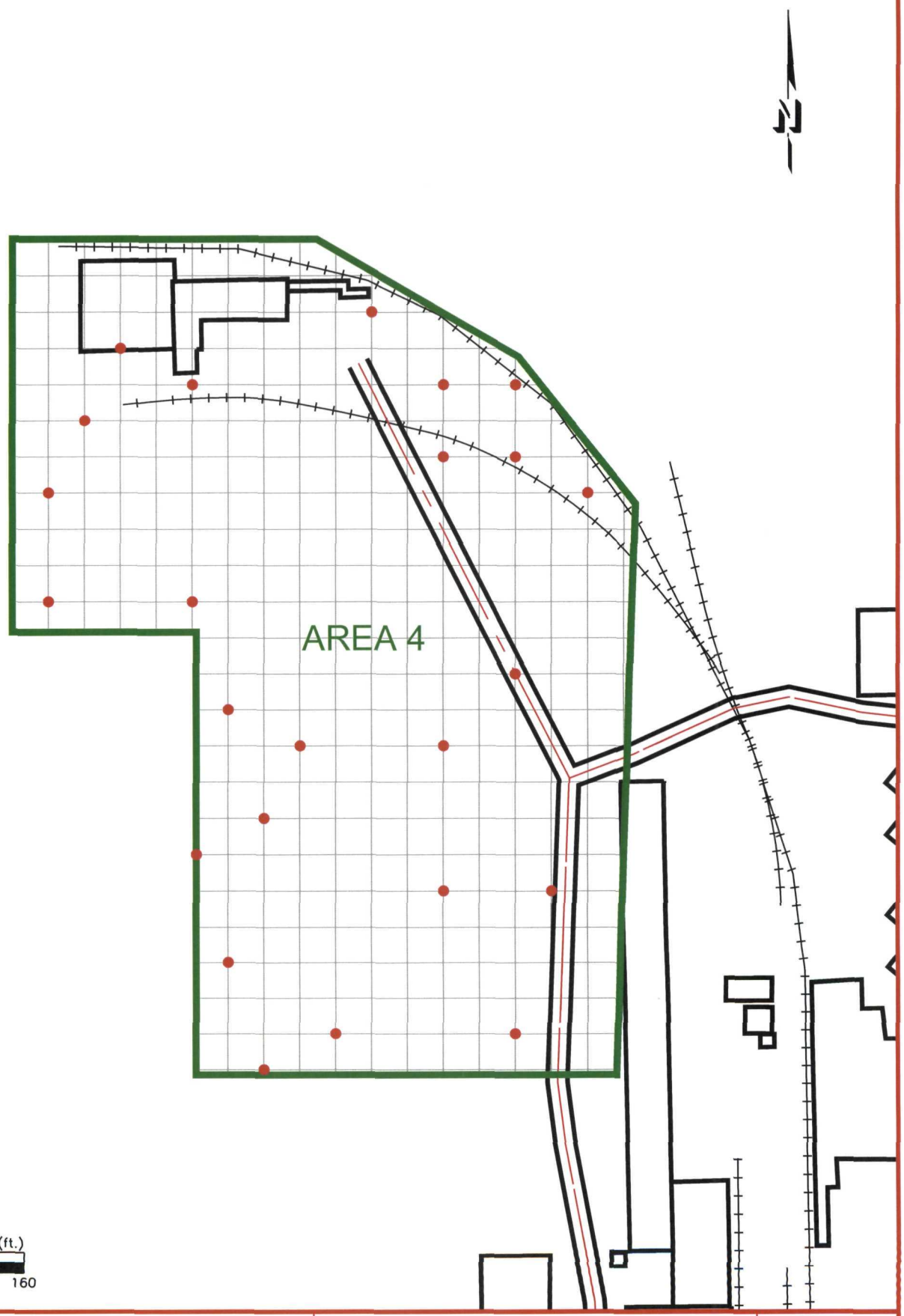

ENVIRON

740 Waukegan Road, Suite 401, Deerfield, IL 60015

Proposed Soil Sample Locations
Area 4
Eagle Zinc, Hillsboro, Illinois

Figure
A-1d

Drafter:	APR	Date:	6/25/02	Contract Number:	21-7400F	Approved:	Revised:

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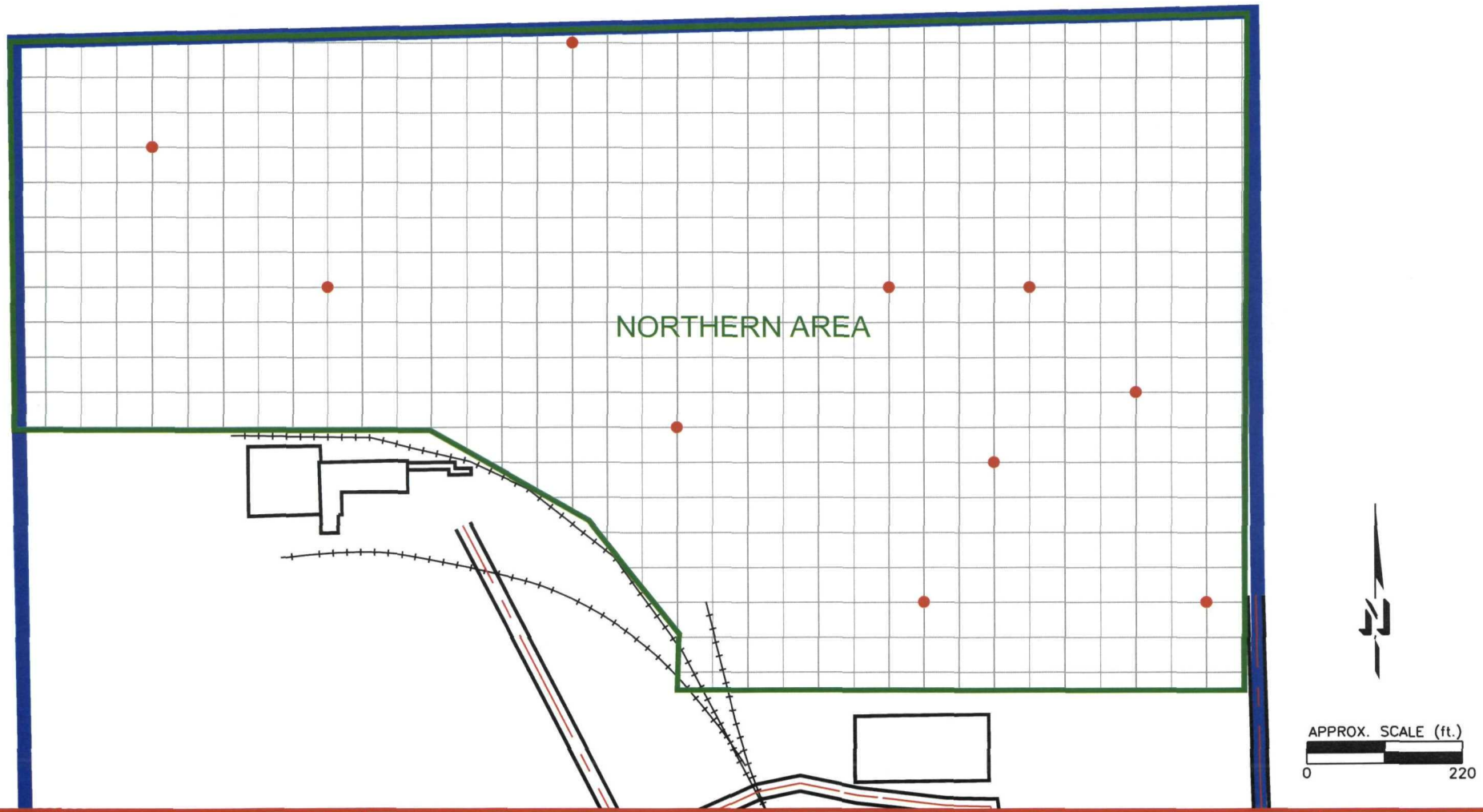

ENVIRON

740 Waukegan Road, Suite 401, Deerfield, IL 60015

Proposed Soil Sample Locations
Northern Area
Eagle Zinc, Hillsboro, Illinois

Figure
A-1e

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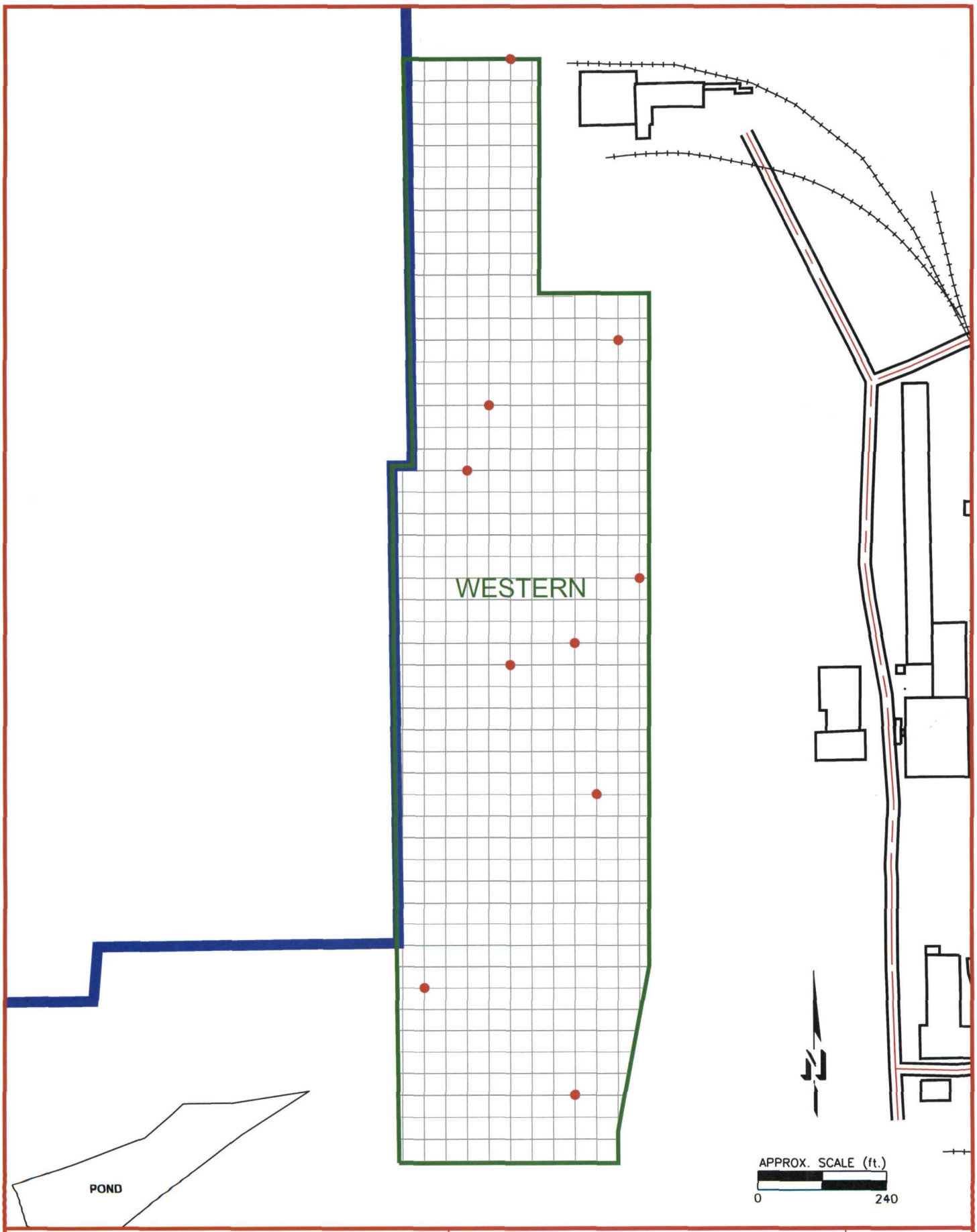

ENVIRON

740 Waukegan Road, Suite 401, Deerfield, IL 60015

Proposed Soil Sample Locations
Western Area
Eagle Zinc, Hillsboro, Illinois

Figure
A-1f

Drafter: APR | Date: 6/25/02 | Contract Number: 21-7400F | Approved: | Revised:

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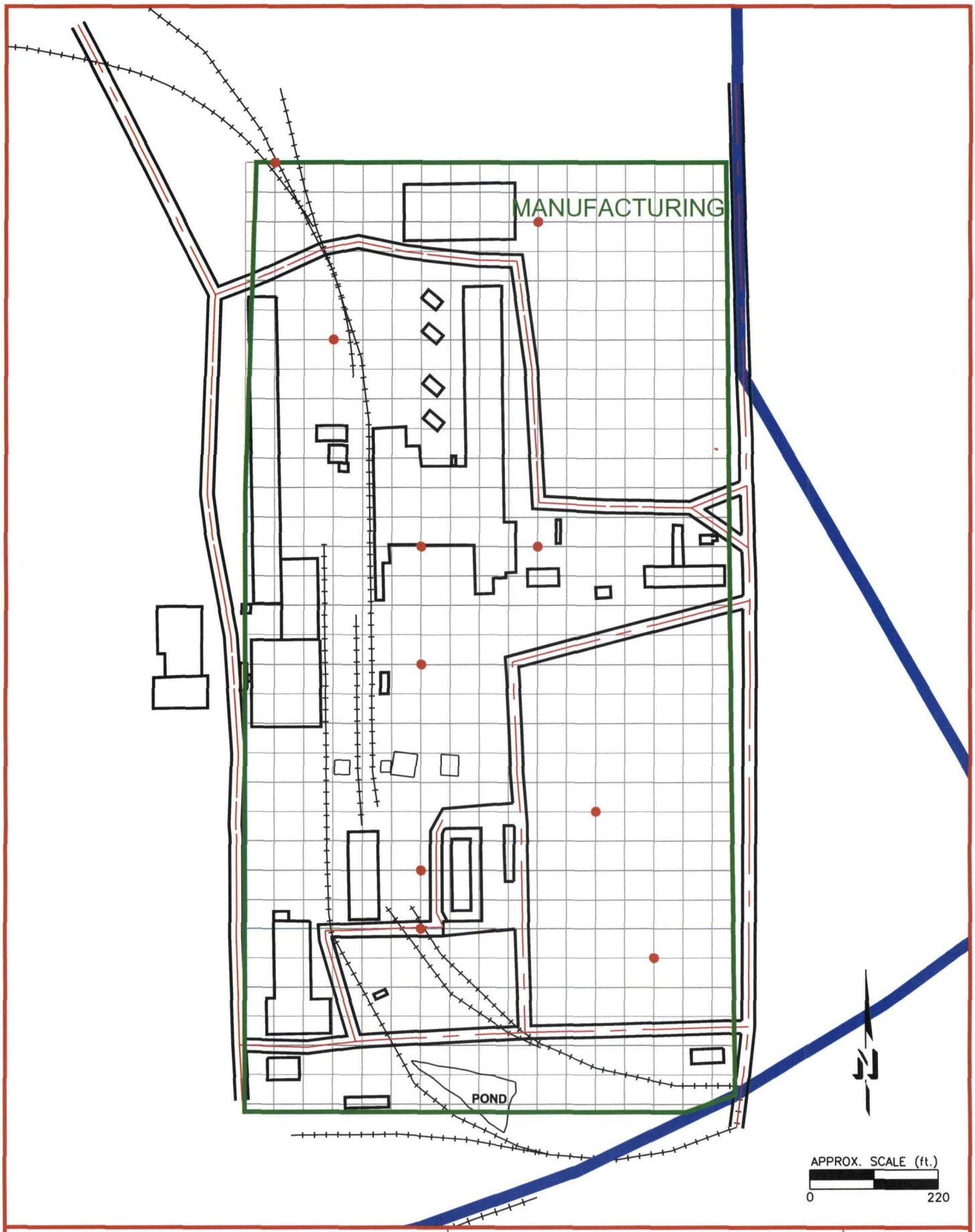

ENVIRON

740 Waukegan Road, Suite 401, Deerfield, IL 60015

Proposed Soil Sample Locations
Manufacturing Area
Eagle Zinc, Hillsboro, Illinois

Figure
A-1g

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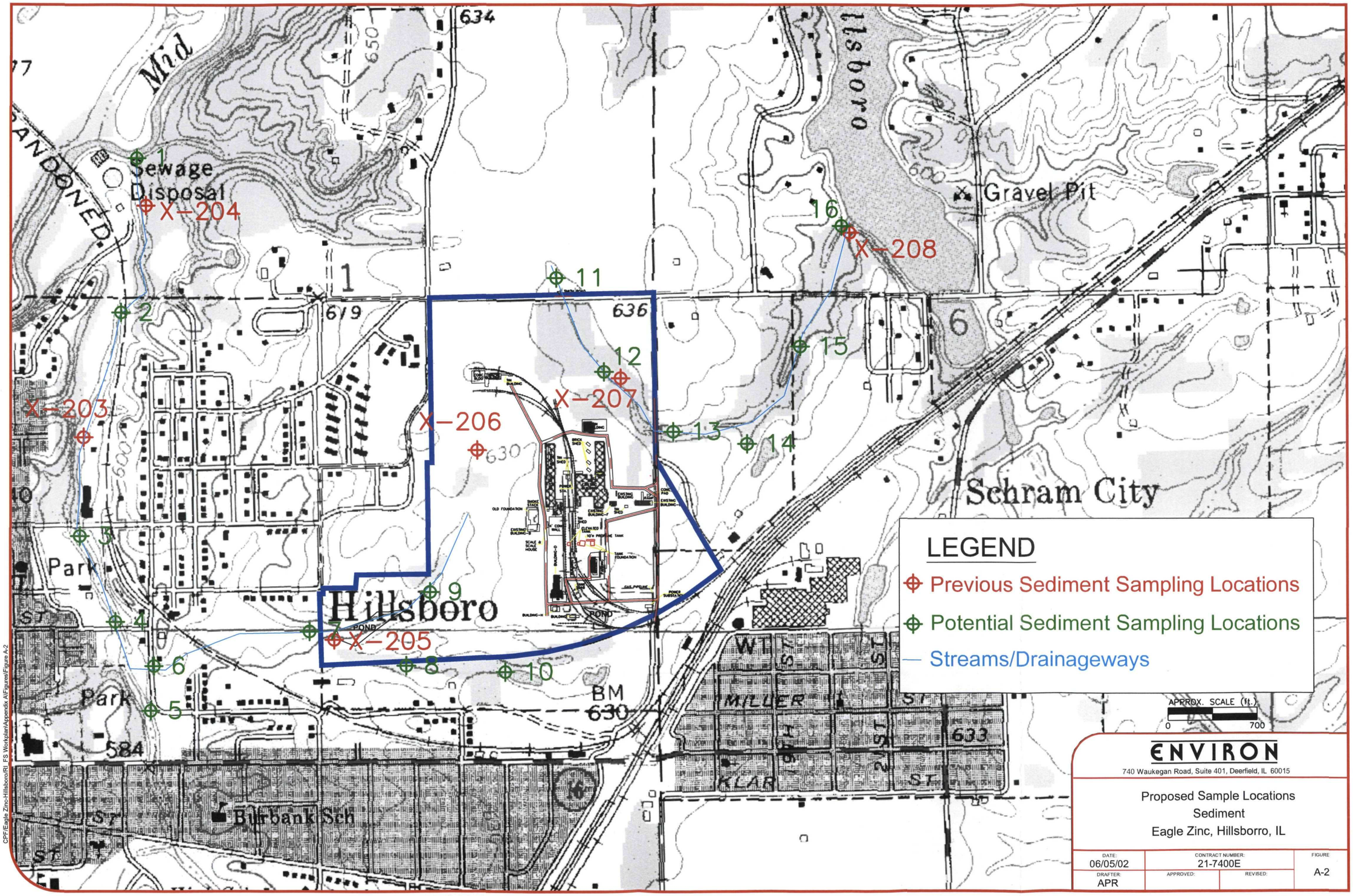
CPFs/Eagle Zinc-Hillsboro/RIFS Work Plan/Appendix A/Figures/Figure A-1a-g Proposed SS Locs



ENVIRON
740 Waukegan Road, Suite 401, Deerfield, IL 60015

Proposed Sample Locations
Sediment
Eagle Zinc, Hillsborro, IL

DATE:	CONTRACT NUMBER:		FIGURE
06/05/02	21-7400E		A-2
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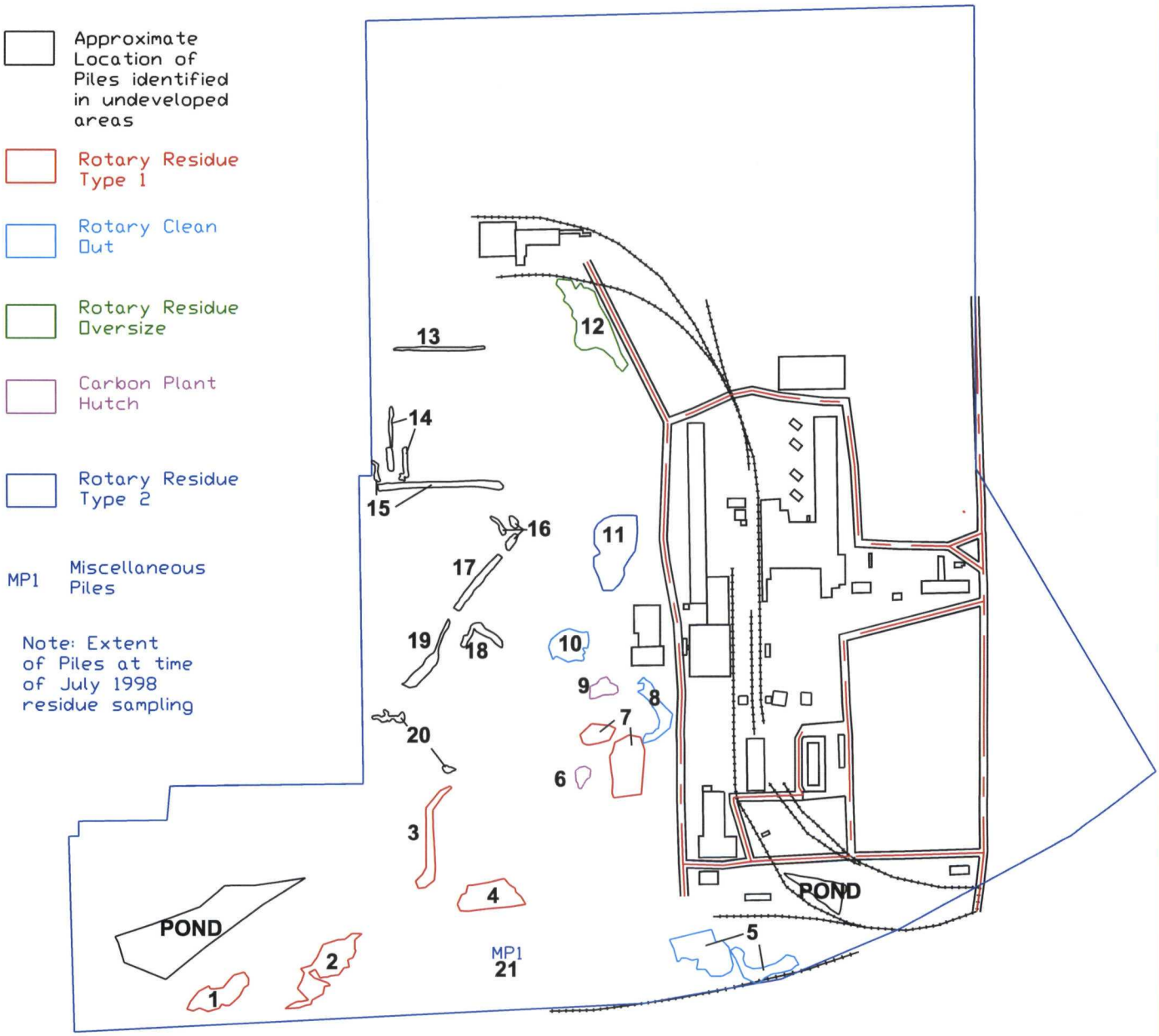

ENVIRON

740 Waukegan Road, Suite 401, Deerfield, IL 60015

Proposed Sample Locations
Residues
Eagle Zinc, Hillsobro, IL

Figure
A-3

Drafter: CJG | Date: 02/28/02 | Contract Number: 21-7400E | Approved: | Revised:

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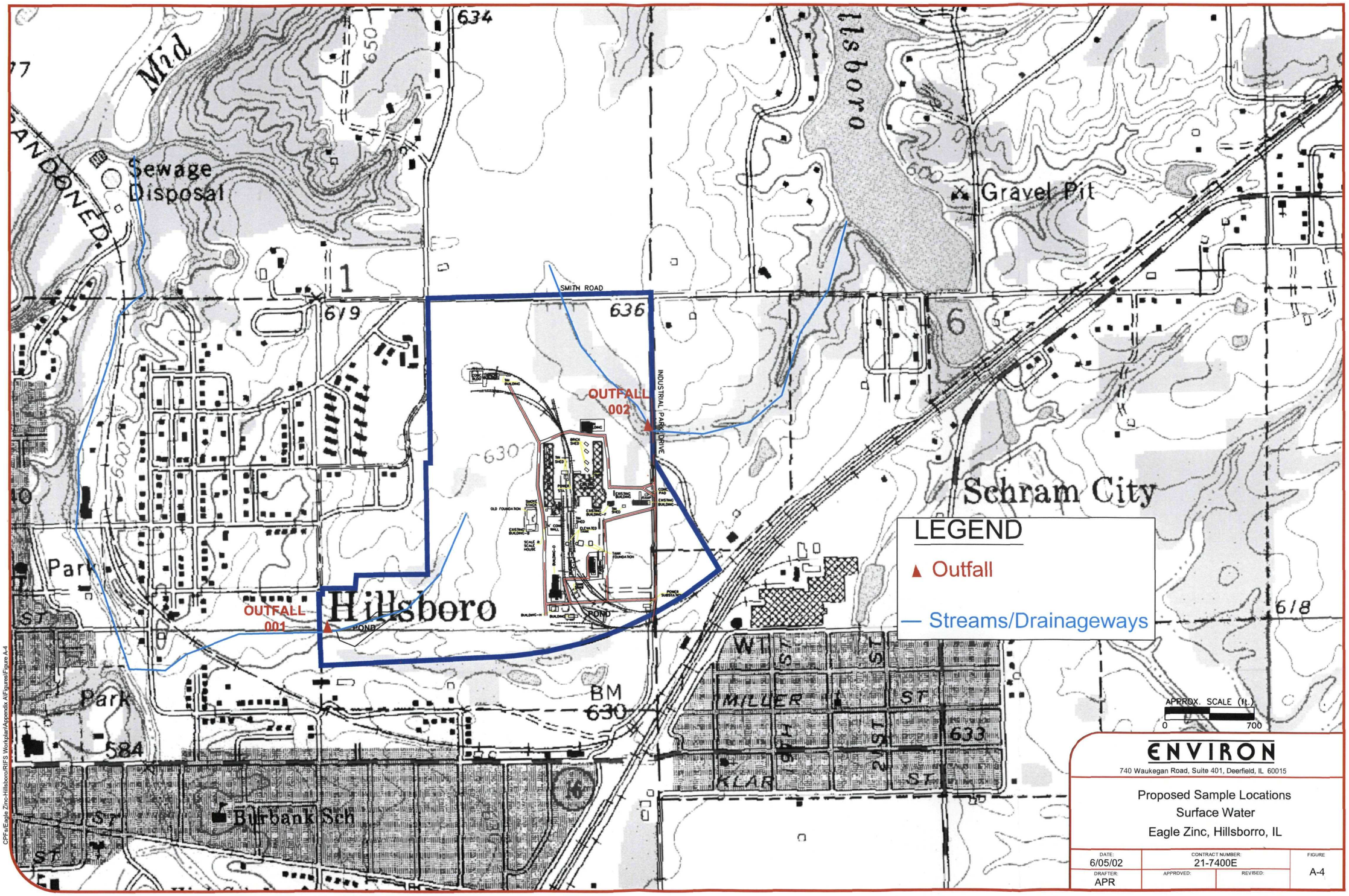

ENVIRON
740 Waukegan Road, Suite 401, Deerfield, IL 60015

Proposed Sample Locations
Surface Water
Eagle Zinc, Hillsborro, IL

DATE:	CONTRACT NUMBER:		FIGURE
6/05/02	21-7400E		A-4
DRAFTER: APR	APPROVED:	REVISED:	

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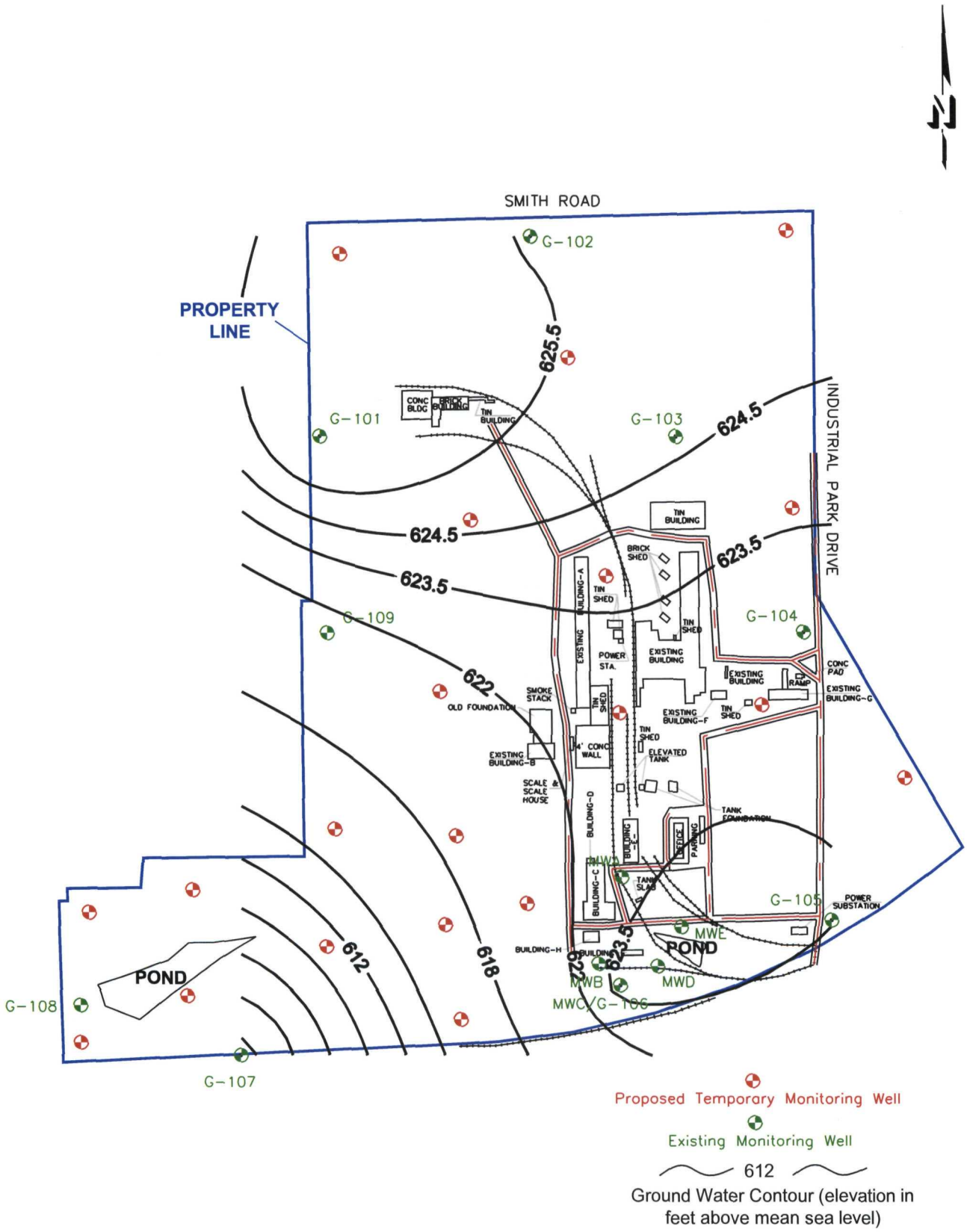

Proposed Temporary Monitoring Well

Existing Monitoring Well

612

Ground Water Contour (elevation in feet above mean sea level)

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ENVIRON 740 Waukegan Road, Suite 401, Deerfield, IL 60015	Proposed Sample Location Ground Water Eagle Zinc, Hillsboro, Illinois	Figure A-5

Drafter: CJG Date: 06/05/02 Contract Number: 21-7400E Approved: Revised:

ATTACHMENT A-1

Standard Operating Procedures – Field Methods

ATTACHMENT A-1

Standard Operating Procedures – Field Methods

1. Introduction

This Standard Operating Procedure (SOP) describes the protocols to be followed during the collection of soil, sediment, surface and ground water samples, and the decontamination of equipment. These activities will be performed as part of the Remedial Investigation (RI) at the Eagle Zinc Company Site. This SOP is included as part of the RI Field Sampling Plan (FSP).

2. Soil Sample Collection Procedures

This section describes the procedures that will be completed to collect soil samples. Soil borings will be completed using a direct-push drilling apparatus and macro-core samplers, which produce 4-foot long soil cores. An experienced geologist will prepare a detailed geological log for each soil boring. Initial field screening for volatile organic vapors will be conducted using a portable photoionization detector (PID) and XRF analyzer immediately after sample retrieval. Field measurement operating procedures are included in Section E.

A. Field Screening

The entire soil and sediment core recovered from the borehole will be field screened for VOCs using a PID and for metals using an XRF analyzer. The procedure for field screening the soil and sediment core is as follows:

- The soil will be field screened immediately after the sampling device is removed from the borehole and opened for inspection.
- The soil core will be screened with an organic vapor meter photoionization detector (PID) using a 11.8 eV bulb.
- For VOCs, the field screening will be performed by creating a headspace within the soil core using a stainless steel sampling spatula/spoon. The

probe of the PID will be inserted into this headspace for approximately 15 seconds, until the instrument has measured a maximum reading.

- For metals the field screening will be performed by making measurements at 6-inch increments along the soil core. To obtain measurements for the full range of metals of interest, a dual source analyzer will be used (i.e., requires two radioactive sources to measure the metals of interest).
- Field screening measurements will be collected at 6-inch intervals for the entire length of the recovered soil core. The measurements will be recorded on the boring log.

B. Soil Sampling

The procedure for collecting investigative soil samples is as follows:

- Soil samples will be collected for VOCs using SW-846 USEPA Method 5035 (methanol field preservation method or equivalent methods). Samples for all other parameters will be collected by filling the sample containers to obtain the required sample volumes.
- The investigative samples will be collected first, followed by the QA/QC samples (Field Duplicate and MS/MSD).

All samples will be preserved, handled, and shipped following the procedures described in Section B.2 of the QAPP.

C. Decontamination

All equipment inserted into a soil boring will be decontaminated prior to their use and before leaving the site. More specifically, the sampling equipment will be decontaminated as follows:

Step 1 Steam clean or scrub equipment with a non-phosphate detergent mixed with potable water.

Step 2 Rinse with potable water.

Step 3 Rinse twice with distilled water.

Step 4 Air dry.

Step 5 Place in a clean polyethylene bag or wrap in aluminum foil with shiny-side out when not in use and during transport.

In addition, all down hole drilling equipment (augers, drill bit, and rods) will be decontaminated with a pressurized steam cleaner.

3. Surface Water/Sediment Sample Collection Procedures

This section describes the procedure that will be completed to collect surface water and sediment samples.

A. Surface Water

The procedure for collecting surface water samples is as follows:

- Surface water samples will be taken from the horizontal and vertical center of the channel. Surface water will be collected by submerging the sampling container in the creek facing upstream. Care will be taken not to displace preservative from pre-preserved sample containers. Precautions to be exercised to minimize the risk of contamination from sediment include: (1) not allowing the sample container to contact the sediment during sampling, and (2) filling the sample container in a manner that prevents localized eddy phenomena from stirring up sediment.
- The surface water sampling effort will progress in the upstream direction to minimize cross-contamination of samples.
- Upon completion of sampling, a stake or flag will be placed on the stream bank parallel to the sample location for future resampling and/or surveying.

- The sample location, time, and other observations will be recorded in the field notebook.
- The field notebook, sample log sheets, labels, and chain-of-custody sheets will be filled out with the sample identification properly designated and logged.

The investigative samples will be preserved, handled, and shipped following the procedures described in Section B.2 of the QAPP.

B. Sediment

The procedure for collecting sediment samples is as follows:

- Sediment samples will be collected by submerging a soft sediment dredge such as a Peterson grab sampler into the surface water and carefully lowering the device to the base of the creek. The device will then be closed and raised to the surface where the sample will be will be immediately placed into the appropriate sample containers. A stainless steel spatula/spoon may be used to assist in filling the sample containers.
- Care will be taken to lower and raise the sampling device in a manner to minimize the loss of fine sediment particles.
- If the portion of stream to be sampled is dry the upper inch of sediment will be removed prior to sampling using a stainless steel spatula/spoon. A separate stainless steel spatula/spoon will be used to fill the sample containers.
- Upon completion of sampling, a stake or flag will be placed on the stream bank parallel to the sample location for future resampling and/or surveying.
- The sample location, time, and other observations will be recorded in the field notebook.

- The field notebook, sample log sheets. labels, and chain-of-custody sheets will be filled out with the sample identification properly designated and logged.

C. Decontamination

All surface water and sediment sampling equipment will be decontaminated prior to their use. More specifically, the sampling equipment will be decontaminated as follows:

Step 1 Steam clean or scrub equipment with a nonphosphate detergent mixed with potable water.

Step 2 Rinse with potable water.

Step 3 Rinse twice with distilled water.

Step 4 Air dry.

Step 5 Place in a clean polyethylene bag or wrap in aluminum foil with shiny-side out when not in use and during transport.

4. Ground Water Sample Collection Procedures

This section describes the procedures that will be completed to:

- Determine the ground water level and elevation at each well,
- Obtain ground water samples in open boreholes,
- Develop newly installed monitoring wells,
- Purge monitoring wells prior to sampling, and obtain ground water samples.

All equipment used for ground water level measurement and ground water sampling will be decontaminated prior to sampling and at the completion of the sampling activities by following the procedures described in Section 4.F of this Attachment.

A. Ground Water Elevation Determination

Prior to purging and sampling the monitoring wells, the depth to water and the total well depth will be measured using an electronic water level meter or equivalent. The instrument must have been calibrated by the manufacturer prior to its purchase and therefore should not require calibration in the field. The water levels will be determined to the nearest 0.01 of a foot with an accuracy of ±0.02 feet and the total well depth will be determined to the nearest 0.1 of a foot with an accuracy of ±0.2 feet. The water level meter probe will be decontaminated prior to their use as outlined in Section 4.F.

The depth to water and the total depth of the monitoring well will be used to determine the volume of ground water to be removed from the monitoring well as part of the purging activities.

Ground water level elevations will be calculated by subtracting the measured depth to water from the surveyed top of casing elevation. The field measurements will be recorded in a field notebook and used to prepare piezometric surface maps of the site. An Illinois-licensed professional land surveyor will measure the top of casing elevation of all monitoring wells.

B. Monitoring Well Development

Monitoring well development will be performed to allow the collection of water samples representative of the formation to be monitored. Well development will be conducted as follows:

- The water level and total depth will be measured, as described in section 4.A above, and be used to calculate casing volume. Any accumulation of sediment will be noted.
- Bailers or pumps may be used to remove water/sediment during the development
- At least three times the measured casing volume of water plus three times the saturated volume of the monitoring well sand pack will be removed. If any water is used during drilling activities three times the

quantity of water lost to the formation during drilling will be removed during development. Field-measured parameters of pH, specific conductance, dissolved oxygen, and temperature will be recorded at each casing volume. Both visual clarity and stabilization of field-measured parameters will be used to evaluate effectiveness of well development.

- A detailed description of each well's response during development and all of the field measurements will be recorded.

C. Monitoring Well Purging

Prior to collecting samples, each well will be purged of standing water using a submersible pump if adequate water is present. If adequate water is not available, a bailer will be used to purge monitoring wells. Monitoring wells screened in the overburden will be purged and sampled using low flow (low stress) techniques. Monitoring wells screened in bedrock intervals will be purged and sampled using low flow (low stress) low volume techniques.

Low Flow Purging

Low flow purging of overburden wells will be conducted as follows:

- The water level and total depth will be measured, as described in section 4.1, and be used to calculate casing volume. Any accumulation of sediment will be noted. Use of a submersible pump or bailer for purging/sampling will be determined.
- If a bailer is used, field parameters will be measured at least every casing volume. The well will be considered purged when a minimum of three times the measured casing volume has been removed, and the field-measured parameters of pH, specific conductance, oxygen reduction potential, dissolved oxygen, turbidity, and temperature have stabilized. Field parameters are considered stabilized when consecutive readings are within 10% or, in the case of pH, 0.1 standard units (SU). If a well bails dry during purging, it will

be considered purged and sampling will commence once a sufficient amount of water has recharged in the well.

- If a submersible pump is used, the pump intake will be placed at the approximate center of the screened interval. The water level in the well should be monitored during pumping, and ideally the pumping rate should equal the well recharge rate with little or no water level draw down in the well (the water level should stabilize at the appropriate pumping rate). If possible, draw down will be minimized (less than 0.3 feet). There should be at least 1 foot of water over the pump intake so there is no risk of the pump suction being broken, or entrainment of air in the sample. Record the pumping rate adjustments and depth(s) to water in the logbook.
- During purging, field parameters will be measured continuously using a flow through cell and recorded at least every casing volume until the parameters stabilize. The well will be considered purged when a minimum of three times the measured casing volume has been removed, and the field-measured parameters of pH, specific conductance, oxygen reduction potential, dissolved oxygen, turbidity, and temperature have stabilized. Field parameters are considered stabilized when consecutive readings are within 10% or, in the case of pH, 0.1 standard units (SU). If a well pumps dry during purging, it will be considered purged and sampling will commence once a sufficient amount of water has recharged in the well.
- A submersible pump intake will be placed at the approximate center of the screened interval. If the length of the screened interval is greater than 10 feet the pump intake will be placed approximately 5 feet from the bottom of the well. The water level in the well should be monitored during pumping, and ideally the pumping rate should equal the well recharge rate with little or no water level draw down in the well (the water level should stabilize at the appropriate pumping rate). If possible, draw down will be minimized (less than 0.3 feet). There should be at least 1 foot of water over the pump intake so there is no risk of the pump suction being broken, or entrainment of air in

the sample. Record the pumping rate adjustments and depth(s) to water in the logbook.

- During purging, field parameters will be measured continuously using a flow through cell until all field parameters stabilize. The field parameters will be recorded at least every half casing volume or every 5 minutes which ever is less. The well will be considered purged when the field-measured parameters of pH, specific conductance, dissolved oxygen, and temperature have stabilized. Field parameters are considered stabilized when 3 consecutive readings are within the following limits: pH within 0.1 SU, specific conductance within 3%, dissolved oxygen (DO) within 10%, and temperature within 3%. Consecutive readings will not taken at less than 3-minute intervals. If a well pumps dry during purging, it will be considered purged and sampling will commence once a sufficient amount of water has recharged in the well.

D. Ground Water Sampling

After each monitoring well has been adequately purged, one round of ground water samples will be collected for laboratory analysis. If sufficient water is present samples will be collected immediate following purging using the bailer/pump used during purging. If using a pump with a flow through cell the samples will be collected directly from the pump discharge prior to discharge to the flow through cell. The pumping rate decreased as low a possible to limit sample disturbance. The ground water sample will be obtained by directly filling the sampling bottles from the bailer or pump discharge in the following order: VOCs, SVOCs, PCBs, metals, sulfate. VOC samples will be collected in 40-milliliter vials, ensuring that there is zero headspace. QA/QC samples will be collected by alternately filling bottles for each sample parameter. VOC sample bottles for both the investigative and the QA/QC sample will be completely filled, purged of headspace, and sealed.

E. Field Instrument Operation, Calibration, and Maintenance

The soil and ground water sampling equipment, including the photoionization detector (PID), XRF analyzer, electric water level meter, and water quality meter (containing the pH, specific conductivity, temperature, and dissolved oxygen probes) will be operated, calibrated, and maintained in the field in accordance to the manufacturer's specifications. A summary of the operating procedures for these instruments is provided below.

Photoionization Detector Operating Procedures:

- Equipment: OVM/DataLogger Model 580B, Thermo Environmental Instruments, Inc. A 11.8 eV lamp will be used with Argon gas and a wavelength of approximately 105 nm
- The OVM 580B is an Organic Vapor Meter that detects and quantitates most organic vapors with a sensitive photoionization detector (PID). It is used to detect volatile organics in the field and in field samples.
- Set up includes: 1) Attach power plug from the back of the unit to the RUN/CHG plug-in and depress the ON/Off switch to turn the unit on. 2) Attach extension to sample inlet at front of unit, 3) Calibrate each day as detailed below, 4) Check battery by observing if low battery signal is on LCD display and charge if needed.
- ENVIRON uses rental PID equipment, which is calibrated by the factory and by the rental company. Field calibration will be performed using the cylinder method at least daily. Calibration by cylinder is described as follows: Isobutylene is chosen as a reference standard (250 ppm) for calibration. A constant flow pressure regulator sets the flow needed for calibration of the 580B unit. The inlet to the unit is connected to the "T" tube that is provided with the calibration cylinder. The other end of the "T" is connected to the regulator on the Standard Cylinder. It is important that a length of tubing is attached to the "T" as this prevents diffusion of ambient air

into the sample line. "Zero" air is assumed to be background air unless field conditions are such that air without hydrocarbons is not available. The calibration steps are:

1. Power up instrument
2. Depress on/off key to ignite lamp and initiate sample pump
3. Depress Mode/Store Key
4. Depress -/CRSR key in response to Log this Value?
5. Depress 1/CRSR key to select parameters mode from the main menu
6. Depress +/INC key to advance thru the Run Mode selection parameter prompt
7. Depress +/INC key to advance thru the Auto Logging Mode selection parameter prompt
8. Depress +/INC key to advance thru the Average Time selection parameter prompt
9. Depress +/INC key to advance thru the Alarm Setting parameter prompt
10. Depress +/INC key to advance thru the Lamp Selection Prompt
11. Depress the +/INC key to advance thru Response Factor Setting parameter prompt.
12. Depress RESET key to initiate calibration sequence
13. Depress the -/CRSR key to decline restoration of the backup calibration
14. Connect the outlet of the calibration tubing assembly to the Model 580 Detector Inlet as describe above
15. Introduce Zero Air to Model 580 by opening the flow regulator
16. Depress RESET Key to zero the unit
17. Close the flow regulator

18. Simultaneously depress RESET and -/CRSR keys to activate the movable cursor
19. Repeat step 18 until cursor is at the ones place
20. Simultaneously depress RESET and +/INC keys to increment the ones place value
21. Repeat step 20 until the ones place value reads 0
22. Repeat step 18 to move cursor to the tens place
23. Repeat step 20 until the tens place value reads 5
24. Repeat step 18 to move the cursor to the hundreds place
25. Repeat step 20 until the hundreds place value reads 2
26. Repeat step 18 to move the cursor to the thousands place
27. Repeat step 20 until the thousands place value reads 0
28. The LCD should now read *SPAN PPM=0250 "+" to continue*
29. Depress the +/INC to accept the span concentration value
30. Connect isobutylene cylinder (250 ppm) to calibration tubing assembly
31. Connect outlet of the calibration tubing assembly to the Model 580B detector Inlet
32. Introduce isobutylene standard to unit by opening flow regulator
33. Reset key and unit will calibrate
34. Close flow regulator
35. Depress +/inc key in response to *Reset to Calibrate* message
36. Depress MODE/STORE to return to Run mode
37. For other calibration gases, and procedures, see operation manual

- Routine Maintenance: Routine maintenance such as lamp replacement, lamp cleaning is performed by the instrument rental service.

- Normal Measurement Procedures include: 1) Turn on equipment as described in Set up procedures above, 2) Once the lamp is lit, the display will show the PPM on the bottom line, 3) set RUN MODE to Max Hold mode and the top line of the display will indicate the maximum reading, 4) Set alarm to project action limit by simultaneously pressing the RESET switch the with either the +/INC switch or the -/CRSR switch, 5) Place unit inlet in area (headspace of soil sample, well, jar or bag sample, breathing zone as appropriate) to be measured and wait for concentration to stabilize, 6) Record reading in field book or field data sheet.
- Data logging and printing features are not expected to be used however instructions are included in the operations manual.
- Instruction manual should be kept with unit at all times to allow for troubleshooting and use of other calibration gases.

XRF Analyzer Operating Procedures

- Equipment: Niton XL-722 X-ray Fluorescence Analyzer
- XRF is used to determine concentration of metals in bulk soil samples.
- Setup Includes: 1) Select SETUP on the menu and verify that XRF is in Bulk Sampling Mode.
- Measurement includes: 1) Turn power on 15 minutes prior to use, 2) Push safety slide out from under the shutter release, 3) Place the unit on the test guard so that the rectangular opening on the test guard is under the window pf the NITON, squeeze the shutter release and press the unit flat against the surface of the test guard, 4) Wait until reading has stabilized (Approximately 30-60 seconds)
- After measurement: 1) turn off the power, 2) wash test guard thoroughly with tap water.
- The XRF is calibrated at the factory but needs to be self-calibrated several prior to use and several times daily to ensure the highest

possible accuracy: 1) Select CALIBRATE AND TEST from the menu and press clear/enter to start self-calibration, 2) Immediately following self-calibration check the unit using the provided standard samples.

- Instruction Manual should accompany the unit at all time to allow troubleshooting using error codes and for instructions regarding replacement of faulty probes.

Electric Water Level Meter Operating Procedures:

- Equipment: Solinst Water Level Meter, Model 101
- Water Level Meter is used to determine depth to water in wells and other locations
- Set up includes: 1) make sure downhole cable and sensor is clean, 2) turn unit on by turn Sensitivity knob clockwise, 3) check battery power by depressing Battery Test button, 4) insert sensor in container of water and test for beep tone.
- Measurement includes: 1) Unit should be on, set Sensitivity knob to approximately the midpoint of the range, 2) Lower sensor into well or measurement location until beep tone is heard. Do not allow the sensor to fall unrestricted to the bottom, 3) Pull up slightly on the cable until tone stops, 4) If tone lingers or is erratic, adjust sensitivity as appropriate, 5) Re-lower sensor until beep tone is heard and read depth on cable markings. The cable is marked with in 0.1 foot divisions, 6) Record depth measurement in field notebook or field form, 7) Use unit handle to reel in the cable and sensor after measurement, 8) Place sensor in metal cylinder-holder located on back side of unit.
- After measurement: 1) Decontaminate cable and sensor as they are retracted from the well or measurement location using soap and water. If necessary they can be soaked in a bucket of soapy water. 2) Rinse cable and sensor with potable water, 3) Turn off unit.

- The sensor and battery should be checked each day. There is no calibration needed for this unit

Water Quality Meter Operating Procedures:

- Equipment: Horiba U-10 Water Quality Checker
- U-10 is used to monitor water parameters during well purge
- Set up includes: 1) Removal of protective rubber cap from pH sensor, 2) check for pH sensor, DO sensor, temperature sensor, conductivity sensor and condition of the probe guard.
- Measurement includes: 1) Turn power on (check battery if no power or if error code *Er1* shows in readout), 2) Gently place the probe into the water sample. Water should be above the level of the DO probe for measurement, 3) when the power is turned on, the U-10 will be in measurement mode. All parameters are measured at once. Use the Select key to toggle the upper cursor on the readout to the parameter to be read, 4) To get a uniform reading, slowly move the probe up and down to circulate the water through it then wait for the readout to stabilize.
- Expanded Readout methods: Use EXP key to see the results with one additional decimal place of accuracy.
- After measurement: 1) turn off the power, 2) wash the probe thoroughly with tap water. 3) Fill the small rubber cap with tap water and use it to cover the pH sensor, 4) Place vinyl tape around the O-ring portion of the reference sensor to prevent the KCl internal solution.
- The U-10 is to be calibrated daily prior to use: 1) Fill the calibration beaker to about 2/3 with the standard solution, to the line on the beaker. 2) Fit the probe over the beaker noting that the DO sensor is not to be immersed in the standard solution, 3) Turn the power on, 4) press the Mode key until cursor shows MAINT for maintenance mode; the lower cursor should be on the AutoSub-Mode, 5) Press the

ENT key and the upper cursor will gradually move across the four auto calibration parameters one by one. When the auto-calibration has stabilized, the upper cursor will stop blinking and automatically move to MEAS mode. 6) If the unit does not move to MEAS mode and the readout shows *Er3* or *Er4*, press the CLR key to cancel the error code and redo the calibration. See the operation manual for procedures if the unit is to be calibrated manually or if the unit will not calibrate automatically.

- Instruction Manual should accompany the unit at all time to allow troubleshooting using error codes and for instructions regarding replacement of faulty probes.

F. Decontamination

All equipment inserted into a monitoring well will be decontaminated prior to their use. More specifically, the sampling equipment will be decontaminated as follows:

Step 1 Scrub equipment with a non-phosphate detergent mixed with potable water.

Step 2 Rinse with potable water.

Step 3 Rinse twice with distilled water.

Step 4 Air dry.

Step 5 Place in a clean polyethylene bag or wrap in aluminum foil with shiny-side out when not in use and during transport.

Non-dedicated pump will be decontaminated as follows:

Step 1 The outside of the pump will be scrubbed with a non-phosphate detergent mixed with potable water.

Step 2 A volume of non-phosphate detergent mixed with potable water solution equal to at least three times the volume of the discharge tubing will be pumped through the pump.

Step 3 Deionized water will be pumped through the pump until specific conductance readings approach zero.

Disposable sampling equipment and sampling gloves will be discarded after sampling each well. All investigation-derived wastes will be handled as indicated in Section F.4 of the FSP.

ATTACHMENT A-2

Example Field Forms

ENVIRON

DAILY SAMPLE TRAFFIC REPORT

Site Name: _____ Date: _____ Page ___ of ____

Site Location/ Address: _____ Weather: _____

Case Name/Number: _____ Observers: _____

Manager: _____ Sampling Personnel: _____

Investigation Samples Collected

Sample Analysis	Matrix	Container Type/ Quantity	Preservative	Depth Sampled	Sample Date and Time	Field Lab or Fixed Lab	Sample Designation

QA/OC Samples Collected

Notes: _____

ENVIRON

GROUND WATER SAMPLING FORM

Site Name: _____ Well or Surface Site #: _____

Site Location/ Address: _____ Sample Designation: _____

Case Name/Number: _____ Date, Time: _____

Manager: _____ Weather: _____

Sampling Personnel: _____ Observers: _____

Water Level Measurements

DTW	TD	Well Diam.	Date	Time	Method Used	PID

Casing Volume ((TD-DTW) x Gal/Ft)= _____

Gallons per foot of casing/borehole: 2 in = 0.163; 4 in = 0.653; 6 in = 1.469; 8 in = 2.611; 10 in = 4.08

Purging Data

Well Volume (#)	Purge Volume (gallons)	pH	Specific Conductance (umhos/cm)	Temp. (°C)	Dissolved Oxygen (mg/L)	Redox (mV)	Notes

Purge Method _____ Total Purge Volume _____ Pump Intake Depth _____

Sampling Data

Sample Analysis	Date	Time	Container Type	Container Volume	Depth Sampled	Field Filtered	Preservative

Notes: _____

APPENDIX B

Quality Assurance Project Plan

QUALITY ASSURANCE PROJECT PLAN

REMEDIAL INVESTIGATION/FEASIBILITY STUDY
EAGLE ZINC COMPANY SITE
HILLSBORO, ILLINOIS

Submitted To:

U.S. Environmental Protection Agency, Region V

Submitted By:

ENVIRON International Corporation
Deerfield, Illinois

On behalf of

Eagle Zinc Group

July 2002

Revision 2

Don Novak — 7.9.02
U.S. EPA Region 5 Remedial Project Manager — Date

[signature] for AR — 7/9/02
U.S. EPA Region 5 Quality Assurance Reviewer — Date

F. Ross Jones — 7/16/02
Contractor Project Manager — Date

Cynthia Bongiovanni — 7/17/02
Contractor QA Officer — Date

Michael C. Suhr — 18-JUL-02
En Chem Laboratory QA Manager — Date

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LIST OF ACRONYMS/ABBREVIATIONS

ARARs	Applicable or Relevant and Appropriate Requirements
ASTM	American Standards for Testing Materials
BNA	Base-Neutral-Acid Extractables (Semivolatile Organics)
BPM	Brownfield Project Manager
CERCLA	Comprehensive Environmental Response, Compensation, and Liability Act (Superfund)
COC	Chain of Custody
CLP	Contract Laboratory Program
CRDL	Contract Required Detection Limits
CRQL	Contract Required Quanitation Limits
CRL	Central Regional Laboratory
DCF	Document Control Format
DQO	Data Quality Objective
EMSL	Environmental Monitoring and Support Laboratory
EAPM	Early Action Project Manager
FSP	Field Sampling Plan
FSS	Field Services Section
MDLs	Method Detection Limits
MS/MSD	Matrix Spike/Matrix Spike Duplicate
NIST	National Institute of Standard Technology
NPL	National Priorities List
OSC	On-Scene Coordinator
QA/QC	Quality Assurance/Quality Control
QAMP	Quality Assurance Management Plan
QAPP	Quality Assurance Project Plan
QLs	Quantitation Limits
PARCC	Precision, Accuracy, Representativeness, Completeness, and Comparability
PE	Performance Evaluation Sample
RAS	Routine Analytical Services
RCRA	Resource Conservation and Recovery Act
RI/FS	Remedial Investigation/Feasibility Study
RD/RA	Remedial Design/Remedial Action
RPD	Relative Percent Difference
RPM	Remedial Project Manager
SAP	Sampling and Analysis Plan
SARA	Superfund Amendments and Reauthorization Act
SAS	Special Analytical Services
SF	Superfund
SMC	Sample Management Coordinator
SOP	Standard Operating Procedure
SOW	Statement of Work
SW-846	Test Methods for Evaluating Solid Waste
TAL	Target Analytes List
TCL	Target Compound List
TIC	Tentatively Identified Compound
TSA	Technical System Audit
USEPA	United States Environmental Protection Agency
VOA	Volatile Organic Analysis
VOC	Volatile Organic Compound

A. Group A – Project Management

This QAPP presents the organization, objectives, functional activities and specific quality assurance (QA) and quality control (QC) activities associated with the Remedial Investigation (RI) activities planned for the Eagle Zinc Company Site. This QAPP also describes the specific protocols that will be followed for sampling, sample handling and storage, chain-of-custody, and laboratory analysis. All QA/QC procedures will be conducted in accordance with applicable professional technical standards, USEPA requirements and guidelines, and specific project goals and requirements.

This QAPP has been prepared in accordance with USEPA guidance documents, specifically, *EPA Requirements for Quality Assurance Project Plans, EPAQA/R-5 Interim Final* (USEPA 1999) and *Instructions on the Preparation of a Superfund Division Quality Assurance Project Plan, Region 5 (USEPA 2000).* This document is provided as Appendix B to the RI/FS Work Plan for the site and is to be read in conjunction with the RI Work Plan and the Field Sampling Plan (FSP), which is provided as Appendix A of the RI/FS Work Plan.

1. Distribution List

The following individuals will be provided with copies of the QAPP:

- USEPA Region 5 Project Manager – Dion Novak;
- IEPA Project Manager – Rick Lanham;
- Project Coordinator for Eagle Zinc Parties – Roy O. Ball (ENVIRON);
- ENVIRON Project Manager – F. Ross Jones;
- ENVIRON Field Activities Coordinators – Scott Hayter and Chris Greco;
- ENVIRON QA Director – Cynthia Bonczkiewicz;
- EnChem, Inc.– Laurie Woelfel and Mike Suha

- Heartland Environmental Services, Inc. (Heartland) – Paul Humburg

2. Project Organization

On behalf of the Eagle Zinc Parties, ENVIRON has overall responsibility for the direction and quality of all phases of the RI, including the management of field activities and the quality of the work. All fixed-base laboratory analyses will be performed by EnChem, Inc. (EnChem). Independent data validation will be performed by Heartland Environmental Services, Inc. (Heartland). The various quality assurance and management responsibilities of key project personnel are defined below.

a. Management Responsibilities

- *USEPA Project Manager:* The USEPA Remedial Project Manager, Dion Novak, has the overall responsibility for USEPA regulatory oversight of all phases of the RI scope of work. The USEPA Project Manager will be responsible for reviewing and approving the QAPP.
- *IEPA Project Manager:* The IEPA Project Manager, Rick Lanham, has the overall responsibility for IEPA regulatory oversight of all phases of the RI scope of work.
- *Project Coordinator for Eagle Zinc Parties:* Roy O. Ball, ENVIRON, has overall responsibility of insuring the quality of work on behalf of the Parties. Dr. Ball will:

  - Define project objectives and develop a detailed work plan schedule;

- Establish project policy and procedures to address the specific needs of the project as a whole, as well as the objectives of each task; and
- Acquire and apply technical and other resources as needed to ensure performance within budget and schedule constraints.
- Develop and meet ongoing project and/or task staffing requirements, including mechanisms to review and evaluate each task product;
- Review the work performed on each task to ensure its quality, responsiveness, and timeliness;
- Review and analyze overall task performance with respect to planned requirements and authorizations;
- Approve all external reports (deliverables) before their submission to USEPA Region 5 and IEPA;
- Ultimately be responsible for the preparation and quality of monthly progress reports, draft and final RI and FS Work Plans and Reports.

ENVIRON Project Manager: F. Ross Jones, P.G., the ENVIRON Project Manager, will:

- Define data collection objectives and develop a detailed field work plan schedule;
- Acquire and apply technical and other resources as needed to ensure performance of field work within budget and schedule constraints;
- Orient all field leaders and support staff concerning the project's special considerations;

- Monitor and direct the field leaders;
- Develop and meet ongoing project and/or task staffing requirements, including mechanisms to review and evaluate each task product;
- Review the field and laboratory work performed on each task to ensure its quality, responsiveness, and timeliness;
- Review and analyze overall task performance with respect to planned field requirements and authorizations;
- Approve all external reports (deliverables) before their submission to USEPA Region 5 and IEPA.

b. Quality Assurance Responsibilities

- *ENVIRON QA Director:* The ENVIRON QA Director is Cynthia Bonczkiewicz. The QA Director will remain independent of direct job involvement and day-to-day operations, and have direct access to project staff as necessary to resolve any QA dispute. The QA Director will be responsible for auditing the implementation of the QA program in conformance with the demands of specific investigations, ENVIRON's policies, and USEPA/IEPA requirements. Specific functions and duties include:

  - Reviewing and approving of QA plans and procedures;
  - Performing QA audit on various phases of the field operations;
  - Providing QA technical assistance to project staff; and
  - Reporting on the adequacy, status, and effectiveness of the QA program on a regular basis to the ENVIRON Project Manager.

- *EnChem Laboratory Quality Assurance Officer:* Mike Suha, the EnChem Laboratory Quality Assurance Officer, will:

  - Oversee laboratory quality assurance;
  - Oversee laboratory QA/QC documentation;
  - Oversee detailed laboratory data review;
  - Decide laboratory corrective actions, if required;
  - Present technical laboratory QA procedures;
  - Prepare laboratory standard operation procedures; and
  - Ensure that the laboratory protocols specified in this QAPP are followed.

c. Laboratory Responsibilities

- *EnChem Laboratory Project Manager:* Laurie Woelfel, the EnChem Laboratory Project Manager, will:
  - Coordinate laboratory analyses;
  - Supervise in-house chain-of-custody;
  - Oversee laboratory data review;
  - Oversee preparation of analytical reports; and
  - Approve final analytical reports prior to submittal to ENVIRON.
- *Independent Laboratory Data Validation*: Data validation will be provided by Paul Humburg of Heartland. Heartland is a consulting firm that specializes in the field of data validation.

The primary responsibility for project quality rests with the ENVIRON Project Manager in conjunction with the ENVIRON project team. Independent

quality assurance will be provided by the EnChem Project Manager and the ENVIRON QA Director prior to the release of data packages.

d. Field Responsibilities

- *ENVIRON Field Activities Coordinators:* ENVIRON Field Activities Coordinators, Scott Hayter and Chris Greco, will be supported by the technical and field staff from ENVIRON and other firms, as needed. They will be responsible for leading and coordinating the day-to-day activities of the various resource specialists under their supervision and will report directly to the ENVIRON Project Manager. Specific ENVIRON Field Activities Coordinators responsibilities include:

- Implementation of field-related plans, assurance of schedule compliance, and adherence to management-developed study requirements;
- Coordination and management of field staff including sampling and subcontractors;
- Implementation of QC for technical data provided by the field staff including field measurement data;
- Adherence to work schedules provided by the ENVIRON Project Manager;
- Authorship, review, and approval of text and graphics required for field team efforts; and
- Identification of problems at the field team level, discussion of resolutions with the ENVIRON Project Manager, and provision of communication between project team members and upper management.

3. Problem Definition/Background

a. Site/Facility History

i. Past Data Collection Activities

The following information concerning the regulatory history of the Site is largely summarized from prior site reports. The facility was initially listed on the Comprehensive Environmental Response Compensation and Liability Information System (CERCLIS) on June 1, 1981 as a discovery action initiated during Sherwin-Williams' ownership of the Site. Sherwin-Williams filed USEPA form 8900-1, Notification of Hazardous Waste Site, in accordance with Section 103(c) of CERCLA, which indicated that slag had been disposed on the Site property. A Preliminary Assessment (PA) of the Site was conducted in 1984 by the IEPA pursuant to CERCLA, which culminated in the submission of a PA Report to U.S. EPA Region V. Sampling of residual materials by IEPA in the early 1980s resulted in a determination that the materials were not hazardous waste and the Site was not subject to RCRA permitting.

In addition to the CERCLA activities described above, several sets of surface water samples were collected by the IEPA from the southwest storm water discharge between 1980 and 1982 and analyzed for metals. Detected concentrations of zinc, iron, lead and copper in the surface runoff above applicable state surface water quality standards on one or more occasion resulted in a Notice of Violation (NOV) from the IEPA. This prompted Sherwin-Williams to remove approximately 18,000 tons of residue materials from 10 acres of the Site.

A CERCLA Expanded Site Inspection was conducted by IEPA on October 26 and 27, 1993, including the collection of 28 environmental samples. Based on information provided by IEPA and as reported in the Expanded Site Inspection Report, the USEPA's Chief of Emergency Response for Illinois, Mr. Donald Bruce, determined that the Site did not require a time-critical or non time-critical removal action, and that the Site property does not pose an immediate threat to human health or the environment.

On May 22, 1998, Eagle Zinc entered into an Interim Consent Order with the Illinois Attorney General and IEPA, which required sampling of storm water discharges, residues, and ground water.

Pursuant to the Interim Consent Order, a monitoring well installation and ground water sampling program was conducted at the Site by GBI, which included the installation and sampling of nine shallow monitoring wells. IEPA representatives collected split samples from the monitoring wells. This investigation culminated in the submission of the March 1999 report entitled *Monitoring Well Installation and Ground Water Sampling Interim Report* to the IEPA. Sampling of residual piles and underlying soils was also conducted by GBI pursuant to the Interim Consent Order. The results of this investigation, which also included the collection of split samples by IEPA, were submitted to IEPA in a March 1999 report entitled *Interim Report of Residue Sampling and Analysis.*

A NPDES Notice of Intent (NOI) was prepared by Eagle Zinc and submitted to the IEPA. On June 20, 2000, IEPA issued NPDES Permit No. IL0074519. The NPDES permit requires: monthly monitoring of NPDES Outfall 002; preparation/implementation of a Storm Water Pollution Prevention Plan (SWPPP); and submission of an annual

inspection report to IEPA. A SWPPP was prepared for the Site in December 2000.

The removal of a 500-gallon gasoline UST in April 1998 resulted in the reporting of a Leaking UST (LUST) incident to IEPA, because a limited amount of impacted soil was observed in the tank excavation and a pin-size hole was observed in the tank itself. No free-phase gasoline or ground water was observed in the tank excavation. Site classification and assessment activities were subsequently performed by GBI and Philip, including screening of soil samples collected from soil borings using a photoionization detector (PID), collection of soil samples for particle size analysis, installation of four monitoring wells, sampling of five monitoring wells,[1] the completion of slug tests to estimate hydraulic conductivity, and the completion of a well search. Samples collected from the monitoring wells on July 10, 2001 contained no detectable concentrations of benzene, toluene, ethylbenzene and xylenes (BTEX). Based on these results and discussions with IEPA, the LUST incident was classified as "low priority" and ground water in the former tank area is being monitored periodically for three years, after which a no further action proposal is expected to be made, barring any detections of contaminants above applicable ground water standards. Subsequent monitoring is ongoing and has not detected any contaminants above applicable ground water standards to date.

ii. Current Status

An RI/FS is being completed pursuant to the Statement of Work (SOW) within the Administrative Order on Consent (AOC) between the

[1] Pre-existing well G-106 was designated MW-C and sampled as part of the UST investigation.

Eagle Zinc Site Parties and the U.S. Environmental Protection Agency (USEPA) dated December 31, 2001. The SOW requires the investigation of soil, sediment, residues, surface water and ground water and describes the general scope of these investigations.

b. Site/Facility Description

The Site is located in the Township of Hillsboro, Illinois. Hillsboro is located in central Montgomery County, Illinois, approximately 50 miles northeast of St. Louis, Missouri and 35 miles south of Springfield, Illinois. The Site is approximately 132 acres in size and is defined as the parcels of land currently owned by Eagle Zinc Company.

The Site is located in a mixed commercial/industrial/residential area located in the northeastern part of Hillsboro. The Site extends from Smith Road south to an unnamed tributary to the Middle Fork of Shoal Creek. Industrial Drive extends north and south along much of the eastern property boundary. North of the Site is Smith Road, a small facility called Hayes Abrasives, a golf course, and farm fields. Industrial Drive, an asphalt company, a railroad corridor, and the former Hillsboro Glass Company facility (now a steel warehouse) are located east of the Site. Some small commercial/industrial facilities (Fuller Brothers Construction/Ready Mix, Illinois Wood Preservers, Hillsboro Rental, Vogel Plumbing) are located south of the Site. Some undeveloped land and a residential area containing single- and multi-family dwellings are located west of the Site. The nearest residential properties are located approximately 200 feet west of the Site.

It is estimated that between 10 and 15 percent of the Site is covered by buildings. Approximately 23 buildings currently exist at the Site, many of which are currently unused. The types of buildings currently used for facility operations include the office/laboratory building, manufacturing/processing

buildings, equipment/raw material/finished product storage buildings, baghouses, and maintenance facilities. Other Site features include railroad spurs, raw material and residual material stockpiles, two storm water detention ponds, a small pond in the southeast corner of the property, and several paved and unpaved roadways.

4. Project Description

a. Project Objectives

The overall purpose of the RI is to investigate the Site's physical characteristics, identify sources of contamination, and determine the nature and extent of contamination at the Site. The results of the field sampling activities will provide input to the baseline risk assessment and the evaluation of potential response action alternatives in the Feasibility Study. The baseline risk assessment, to be completed as part of the RI, will quantify the potential risk to human health and the environment posed by contaminants potentially present in soil, sediment, surface water and ground water on-site, as well as sediment and surface water in drainageways located off-site. The evaluation of remedial alternatives for the site will include an assessment of risk. The results of the RI will be sufficient to provide a basis for the development and evaluation of potential remedial response alternatives for the site during the subsequent FS.

The Site activities will be conducted in two separate phases: Phase 1-Contaminant Characterization, and Phase 2-Contaminant Migration Assessment. Phase 1 will include the investigation and sampling of soil, sediment and plant residues. Phase 2 will include the investigation and sampling of surface water and ground water.

b. Project Target Parameters and Intended Data Usages

Sample matrices and analytical parameters are presented in Table B-1. The rationale for these parameters is described in the FSP.

i. Field Parameters

The field parameters to be measured consist of screening of all media samples using a PID and soil samples using an XRF analyzer. Field parameters to be measured on the ground water samples also include pH, temperature, specific conductivity, and dissolved oxygen.

ii. Laboratory Parameters

The analytical parameters and methods to be followed for this RI are presented in Table B-1. These parameters include volatile organic compounds (VOCs), semivolatile organic compounds (SVOCs), polychlorinated biphenyls (PCBs) and metals. The rationale for the selection of these parameters is discussed in the FSP.

c. Project Schedule

The schedule for the RI, which is current as of the date of this work plan, is presented in Figure II-15 of the RI/FS Work Plan. The actual time required to implement the investigation may vary, depending upon changes in scope, Agency review periods, or other factors. The project schedule will be revised accordingly throughout the RI. The actual date of project mobilization is dependent upon Agency approval of the various project plans comprising the RI/FS Work Plan.

5. Quality Objectives and Criteria for Measurement Data

The overall quality assurance (QA) objective is to develop and implement procedures for field sampling, chain-of-custody, laboratory analysis, and reporting that will provide high quality results, and which are legally defensible in a court of law. Specific procedures for sampling, chain-of-custody, laboratory instrument calibration, laboratory analysis, reporting of data, internal quality control, audits, preventive maintenance of field equipment, and corrective action are described in other sections of this QAPP. The purpose of this section is to address the specific objectives for accuracy, precision, completeness, representativeness, and comparability.

The fundamental QA objective with respect to accuracy, precision, and sensitivity of laboratory analytical data is to achieve the QC acceptance criteria of the analytical protocols. SOPs for field methods are provided in Attachment B-1. SOPs for laboratory analyses are provided in Attachment B-2. The SOPs address the required accuracy, precision, and sensitivity of the analyses.

a. Data Quality Objectives (DQOs) Process

DQOs are qualitative and quantitative statements that clearly state the objective of a proposed project, define the most appropriate type of data to collect, determine the most appropriate conditions for data collections, and specify acceptable decision error limits that establish the quantity and quality of data needed for decision making. The DQO process is described in a series of seven sequential steps: Step 1: Stating the Problem; Step 2: Identifying the Decision; Step 3: Identifying Inputs to the Decision; Step 4: Defining the Boundaries of the Study; Step 5: Developing a Decision Rule; Step 6: Specifying Limits on Decision Errors; and Step 7: Optimizing the Design. The DQO process is discussed below, including applicable aspects of the

Eagle Zinc Company Site RI/FS. Certain information contained in other portions of the RI/FS Work Plan are included by reference.

Step 1: Stating the Problem

As described in Section A.3 above, the project consists of the completion of a multi-media RI/FS at the site pursuant to the Statement of Work (SOW) contained in an Administrative Order on Consent (AOC) between the Eagle Zinc Site Parties and the USEPA dated December 31, 2001. The SOW requires the investigation of soil, sediment, surface water and ground water and describes the general scope of the investigation. The scope of the RI is described in detail in Section II of the RI/FS Work Plan and in the FSP (Appendix A of the RI/FS Work Plan).

As discussed above, the overall purpose of the RI is to investigate the Site's physical characteristics, identify sources of contamination, and determine the nature and extent of contamination at the Site. The results of the field sampling activities will provide input to the baseline risk assessment and the evaluation of potential response action alternatives in the Feasibility Study. The baseline risk assessment, to be completed as part of the RI, will quantify the potential risk to human health and the environment posed by contaminants potentially present in soil, sediment, surface water and ground water on-site, as well as sediment and surface water in drainageways located off-site. The evaluation of remedial alternatives for the site will include an assessment of risk. The results of the RI will be sufficient to provide a basis for the development and evaluation of potential remedial response alternatives for the site during the subsequent FS. The current schedule for completion of the RI/FS is included in the RI/FS Work Plan as Figure II-15.

The roles and responsibilities of all key management, quality assurance, laboratory and field personnel are provided in Section A.2 above. The DQOs for the project have been developed by ENVIRON personnel (the "scoping team") under the direction of the ENVIRON Project Manager and the Project Coordinator for the Eagle Zinc Parties. The scoping team has developed a preliminary conceptual site model (CSM), which is summarized as Section II.A of the RI/FS Work Plan. The CSM will be updated, as necessary, as additional data becomes available during the RI. The current understanding of the distribution of potential sources and locations of contamination is discussed in Section II.A of the RI/FS Work Plan. Potential Preliminary Response Objectives and Applicable or Relevant and Appropriate Requirements (ARARs) are discussed in Section II.B of the RI/FS Work Plan. These action levels will be established during the RI.

Step 2: Identifying the Decision

The principal decision is whether, and if applicable, to what extent, various environmental media at the site and in adjacent off-site areas have been impacted by contamination from the site. Alternative actions that may be taken based on the outcome of the study (i.e., following completion of the FS) include various remedial measures, including the no action alternative. The process for identifying and evaluating potential remedial alternatives during the FS is described in Section III of the RI/FS Work Plan. These alternative actions will be developed based on refinement of the CSM, determination of risk associated with each environmental medium/exposure pathway, and potentially through the completion of bench-scale studies.

Step 3: Identifying Inputs to the Decision

The types of information that will be used as input to the decision consists of : (1) historical information and data previously compiled by the scoping team and presented in Section II.A of the RI/FS Work Plan; (2) on-site data generated from field and laboratory analysis of soil, sediment, residues, and ground water; (3) off-site analytical data generated from laboratory analysis of sediment and surface water; (4) on-site stratigraphical and hydrogeological data developed from on-site subsurface investigations; and (5) bench-scale studies, if applicable. While they will be developed during completion of the RI, it is anticipated that the specific action levels that will provide the criteria for selecting alternative actions will consist of regulatory standards.

Step 4: Defining the Boundaries of the Study

Consistent with the RI/FS Work Plan, the general spatial boundaries of the RI include the site property boundaries and the storm water drainage channels and intermittent streams that receive storm water runoff from the site. Specifically, soil sampling will be conducted within seven on-site areas of potential concern described in the FSP. Sediment sampling will be conducted within two well-defined drainage channels that extend from the site to off-site areas. Residue samples will be collected from several residue stockpiles or groupings of stockpiles. Surface water samples will be collected from the aforementioned drainage channels. Ground water samples will be collected from temporary and permanent monitoring wells installed at various locations on the site property. Sample depths and sampling methods are described in detail in the FSP.

At this time, no temporal boundaries that could affect the decision are anticipated. With the possible exception of meteorological conditions, which

could affect the timing or methods of sample collection activities in the drainage channels, no practical constraints on data collection are envisioned.

Step 5: Developing a Decision Rule

The purpose of developing a decision rule is to integrate the output from previous steps of the DQO process into a statement that defines the parameter of interest; delineates the scale of decision making; specifies the action level; and describes the logical basis for choosing among alternative actions. The output for this step is develop an "if...then..." statement that defines the conditions that would cause the decision maker to choose among alternative courses of action. Decision rule statements will be developed during the later stages of the RI and during completion of the FS and will incorporate the results of the Baseline Risk Assessment. This process will be reflected in the data evaluation and remedial alternatives selection processes presented in the RI and FS Reports.

Step 6: Specifying the Limits on Decision Errors

The sampling and analysis program discussed in the FSP provides procedures by which a comprehensive RI will be completed at the site. Procedures such as random selection of soil sampling locations from a pre-established grid and multiple compositing of residue samples reduce the possibility of decision errors. Other procedures described in this QAPP, such as field QC samples, and validation of laboratory data, will further reduce sampling and measurement errors. Corrective actions may be considered during implementation of the RI if suspect or questionable laboratory or field data are identified through QA/QC reviews.

Step 7: Optimizing the Design

Several steps, such as random selection of sample locations and use of field screening, have been proposed, which optimize the design of the data collection program. Additional data collection may be conducted as part of the RI to fill in any significant data gaps. Documentation of the current design is presented in the RI/FS Work Plan. Any agreed upon alterations to the design will be documented in future report submissions.

b. Precision

i. Definition

Precision refers to how closely two or more measurements of the same parameter or property agree with each other. Chemical concentration data obtained from the analyses of field duplicate and matrix spike duplicate samples will be compared to evaluate analytic precision. Field precision will also be determined by the collection of duplicate PID, XRF, and other field screening readings.

ii. Field Precision Objectives

Duplicate samples and ground water samples will be collected in the field. The project goal is to collect one duplicate per 20 samples during the RI. Section B.5 of this Plan describes duplicate collection techniques and review.

iii. Laboratory Precision Objectives

Measurement of precision is mathematically defined for laboratory analysis in SectionB.5 of this Plan.

c. Accuracy

i. Definition

Accuracy is the degree of agreement between an observed value and an accepted reference value.

ii. Field Accuracy Objectives

The accuracy of the field data will be maintained by ensuring instruments are in good working condition and properly calibrated. Each piece of equipment will have a unique serial number for tracking during field use, calibration, and maintenance records. Team members will be familiar with the field calibration, operations, and maintenance of the equipment. They will maintain proficiency in equipment operation, perform the prescribed field operating and calibration procedures outlined in the equipment manuals for each respective instrument, and keep records of all field instrument calibrations and field checks in logbooks. The accuracy of the PID field screening, ground water field parameter measurements, and metals screening will be evaluated in conjunction with the instrument calibration records to ensure the highest possible accuracy. Accuracy in the field will also be assessed through the use of equipment rinse and trip blanks as discussed in SectionD.1 of this Plan and through adherence to all sample handling, preservation and holding time requirements.

iii. Laboratory Accuracy Objectives

Laboratory accuracy is mathematically defined in Section D.1 of this Plan. These are updated annually based on the laboratory

performance data from the previous year. Accuracy control limits are given in the laboratory SOPs in Attachment B-2.

d. Completeness

i. Definition

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

ii. Field Completeness Objectives

Field completeness is a measure of the amount of valid measurements obtained from all the measurements taken in the project. The equation for completeness is provided in Section D.1 of this Plan. Field completeness for this project will be greater than 90 percent.

iii. Laboratory Completeness Objectives

Laboratory completeness is a measure of the amount of valid measurements obtained from all the measurements planned for the project. The equation for completeness is presented in Section D.1 of this Plan. Laboratory completeness for this project will be greater than 90 percent.

e. Representativeness

i. Definition

Representativeness expresses the degree to which data accurately and precisely represent a characteristic of a population, parameter

variations at a sampling point, a process condition, or an environmental condition. Representativeness is a qualitative parameter, which is dependent upon the proper design of the sampling program and proper laboratory protocol. The sampling network was designed to provide data representative of facility conditions.

ii. Measures to Ensure Representativeness of Field Data

Representativeness is a qualitative parameter, which is dependent upon the proper design of the various monitoring programs and proper laboratory protocol. The monitoring plans are designed to provide data representative of site conditions. Representativeness will be satisfied by requiring that the procedures detailed in the FSP are followed, proper sampling techniques are used, proper analytical procedures are followed and holding times of the samples are not exceeded in the laboratory. Representativeness will be insured by the selection of proper well locations, boring locations, and samples. Decisions regarding wells, borings, and sample locations and the statistical design are documented in the FSP.

iii. Measures to Ensure Representativeness of Lab Data

Representativeness will be satisfied by insuring that proper analytical procedures are followed and holding times of the samples are not exceeded in the laboratory.

f. Comparability

i. Definition

Comparability expresses the confidence with which one data set can be compared with another. To the extent that the data collection objectives and analytical methods are similar, data generated during previous field investigations are expected to be comparable to data that will be generated during the RI.

ii. Measures to Ensure Comparability of Field Data

Comparability is dependent upon proper design of the sampling program and will be satisfied by ensuring that the FSP and this QAPP are followed. The methods used to collect field data will be consistent during the RI ensuring comparability of field data.

iii. Measures to Ensure Comparability of Lab Data

The extent to which existing and planned analytical data will be comparable depends on the similarity of the sampling and analytical methods as documented in this QAPP. Comparability is also dependent on similar QA objectives. The methods used to collect for analysis and the methods used to analyze samples will be consistent during the RI, ensuring comparability of laboratory data.

g. Level of Quality Control Effort

Trip blank, field duplicate and matrix spike samples will be analyzed to assess the quality of the data resulting from the various monitoring programs implemented for this project. Trip blanks consisting of deionized water will be submitted to the EnChem laboratory to provide the means to assess the

quality of the data resulting from the program. Trip blanks are used to assess the potential for contamination of samples due to contaminant migration during sample shipment and storage. Duplicate samples are analyzed to check for sampling and analytical reproducibility. Matrix spike/matrix spike duplicates (MS/MSD) provide information about the effect of the sample matrix on the digestion and measurement methodology. All matrix spikes are performed in duplicate and are hereinafter referred to as MS/MSD samples.

The general level of the QC effort will be one field duplicate and one MS/MSD for every 20 or fewer investigative samples per sample matrix (i.e., soil, ground water, etc.). One trip blank consisting of deionized organic-free water will be included with each cooler containing aqueous volatile organic compounds (VOCs) samples. The trip blank will be analyzed for VOCs only. MS/MSD samples are investigative samples. Soil MS/MSD samples require no extra volume for metals and extractable organics. In addition, soil MS/MSD samples require no extra volume for VOCs if samples exceed 200 ppb for any parameter, as a LCS and LCS Duplicate shall be substituted. However, aqueous MS/MSD samples must be collected at triple the volume for VOCs, metals, and other parameters.

The number of field duplicate samples, trip blank samples, and matrix spike samples to be collected is detailed in Table B-1.

h. Special Training/Certification

No specialized training or certification beyond that typically required for this type of investigation (e.g., OSHA health and safety training, etc.) will be needed for the completion of this project. ENVIRON personnel conducting field XRF analyses have been trained by the instrument manufacturer in the proper use of the instrument and safety precautions. All certificates and/or

documentation representing completion of specialized training will be maintained in personnel files.

i. Documents and Records

The ENVIRON Project Manager will be responsible for insuring the appropriate project personnel have the most current approved version of the QAPP. Any revisions to the QAPP will be circulated to the individuals listed in Section A.1.

Detailed information concerning the field and laboratory reporting and record keeping procedures is provided in Section B.10. The content of and reporting frequency for project QA reports is discussed in Section C.2.

The final evidence file will be the central repository for all documents that constitute evidence relevant to sampling and analysis activities as described in this QAPP. The ENVIRON Project Manager is custodian of the final evidence file and maintains the contents of evidence files for the RI, including all relevant records, reports, logs, field notebooks, pictures, subcontractor reports and data reviews in a secured, limited access area and under custody of the ENVIRON Project Manager. All evidence in the file will be retained for a period of six years from the date the Consent Order is terminated as provided in the AOC.

B. Group B: Data Generation and Acquistion

1. Sampling Process Design

The field sampling program is discussed in Section C of the FSP. Table B-1 presents a summary of this information.

2. Sampling Methods

a. Sample Collection/Preparation Procedures

The sample collection procedures are detailed in Section C of the FSP.

b. Sample Containers, Preservatives and Volume Requirements

Requirements for sample containers, preservation and volume are summarized in Table B-2.

c. Decontamination Procedures

Decontamination procedures are described in Section E of the FSP.

d. Sample Packaging and Shipment Procedures

As soon as sample labeling is completed, each sample will be placed in an insulated container (cooler) that contains sufficient ice to maintain the samples between 4° C and 6° C. During each sampling task, samples will be shipped to the fixed-base laboratory at least every other working day. The sample containers will be placed in ZipLoc™ bags and then individually wrapped with cushioning material (bubble wrap). The bottom of the insulated sample shipping container (cooler) will be lined with cushioning material (bubble wrap) to reduce the possibility of sample container breakage. Sufficient ice will be placed in each cooler to maintain the samples between

4° C and 6° C for the overnight shipment to the fixed-base laboratory. Chain-of-custody procedures will be followed as described in Section B.3. Once the required chain-of-custody efforts (forms, custody seals, etc.) have been performed, the shipping container will be additionally sealed with plastic shipping tape, and shipped via overnight courier for morning delivery to EnChem.

e. Field Corrective Action

Corrective action in the field may be needed when the sample network is changed (i.e. more/less samples, sampling locations other than those specified in the QAPP, etc.), when sampling procedures require modification, etc., due to unexpected conditions. Field and technical staff will be responsible for reporting all suspected technical or QA non-conformances or suspected deficiencies of any field activity or issued document by reporting the situation to the ENVIRON Field Activities Coordinators or designee. The ENVIRON Field Activities Coordinator or designee will be responsible for assessing the suspected problems in consultation with the ENVIRON Project Manager and/or QA Director. A decision will be made based on the potential for the situation to impact the quality of the data. The ENVIRON Field Activities Coordinator or designee will then be responsible for initiating corrective action for non-conformances by:

- Evaluating all reported non-conformances;
- Controlling additional work on non-conforming items;
- Determining disposition or action to be taken;
- Maintaining a log of non-conformances;
- Reviewing non-conformance reports and corrective actions taken; and

- Verifying that non-conformance documentation is included in the project files.

If appropriate, the ENVIRON Project Manager or designee will see that no additional work that is dependent on the non-conforming activity is performed until the corrective actions are completed.

The ENVIRON Field Activities Coordinator or designee is responsible for controlling, tracking and implementing the identified changes. All changes will be reported by the ENVIRON Project Manager in the monthly progress reports submitted to USEPA.

3. Sample Handling and Custody

Custody is one of several factors, which is necessary for the admissibility of environmental data as evidence in a court of law. Custody procedures help to satisfy the two major requirements for admissibility: relevance and authenticity. Sample custody is addressed in three parts: field sample collection, laboratory analysis and final evidence files. Final evidence files, including all originals of laboratory reports and purge files, are maintained under document control in a secure area.

A sample or evidence file is under your custody if it is:

- In your possession;
- In your view, after being in your possession;
- In your possession and you place it in a secured location; or
- In your designated secure area.

A chain-of-custody record is a record of all persons who have collected, relinquished and/or received samples and the dates and times when these activities occurred. Items that must be held under a chain-of-custody include samples, sample tags, air-bills and a chain-of-custody record form. The chain-of-custody will be initiated in the field and will be maintained until delivered to the laboratory. The laboratory is a restricted access facility with lockable cold storage. Sample containers that are returned to the field or subcontracted shall initiate the chain of custody record again.

a. Field Custody Procedures

The sample packaging and shipment procedures summarized below will ensure that the samples will arrive at the laboratory with the chain-of-custody intact. The protocol for specific sample labeling is included in Section D of the FSP.

i. Field Procedures

The field sampler is personally responsible for the care and custody of the samples until they are transferred or properly dispatched. As few people as possible should handle the samples. Following are basic sampling procedures:

- All containers will be labeled with sample numbers and locations, date/time of collection, and type of analysis. The sample numbering system is presented in Section D of the FSP
- A sample label will be attached to each individual sample aliquot for each investigative or quality control sample. The sample label will include the following information: the field sample number, date and time of collection, type of analysis, type of preservative

(if any), a space for the lab sample number, project identification, and the name of the person collecting the sample. The label may also include a space for comments. The sample label will be adhesive-backed and will be attached to the sample container. Sample labels will be completed for each sample using waterproof ink.

- The ENVIRON Field Activities Coordinator and Project Manager will review all field activities to determine whether proper custody procedures were followed during the field work and decide if additional samples are required.

ii. Transfer of Custody and Shipment Procedures

Samples collected for off-site analysis at the EnChem fixed base laboratory will be accompanied by properly completed chain-of-custody forms. The sample numbers and locations will be listed on the chain-of-custody form. When transferring the possession of samples, the individuals relinquishing and receiving will sign, date, and note the time on the record. This record documents transfer of custody of samples from the sampler to another person, to a mobile laboratory, to the permanent laboratory, or to/from a secure storage area.

Samples will be properly packaged on ice between 4°C and 6° C for shipment and dispatched to the appropriate laboratory for analysis, with a separate signed custody record secured to the inside top of each sample box or cooler. Shipping containers will be secured with strapping tape and custody seals for shipment to the laboratory. The preferred procedure includes use of a custody seal attached to the front right and back left of the cooler. The custody seals will be covered with

clear plastic tape. The cooler will be strapped shut with strapping tape in at least two locations.

The chain-of-custody record identifying the contents will accompany all shipments. The original record will accompany the shipment, and the sampler for return to the sampling office will retain copies.

Air bills or bills of lading will be used when samples are sent by Federal Express, UPS, Airborne Express or Express Mail. Receipts of air bills or bills of lading will be retained as part of the permanent documentation. If sent by mail, the package will be registered with return receipt requested. The custody forms will be sealed inside the sample cooler and coolers will be sealed intact; therefore, the commercial carriers will not be required to sign off on the custody form.

b. Laboratory Custody Procedures

All samples shipped off-site for analysis will be received at EnChem by the sample custodian (SC). It will be the responsibility of the SC to determine whether or not the samples are close to exceeding their holding time and require immediate attention and the manner in which those samples will be split, preserved and stored or routed.

The sample custodian is responsible for the receipt, log-in, and access controlled storage of all client samples at EnChem. Each sample is labeled with a unique number, which is entered into the sample receiving log. The samples are placed into appropriate storage within the access-controlled location. All samples are maintained under proper storage conditions for thirty days past the generation of the analytical report.

4. Analytical Methods

All soil, sediment, residue, surface water and ground water samples collected during the RI field sampling activities will be analyzed using SW846 methodologies by EnChem.

a. Field Analytical and Measurement Procedures

Field methodologies for the collection of field VOC and XRF screening data are provided in the FSP. Quality assurance objectives for field parameters are included in Table B-3.

b. Laboratory Analytical and Measurement Procedures

Standard operating procedures have been prepared for all methods used for analysis of samples for this project. These project specific laboratory SOPs are included in Attachment B-2.

Each of these SOPs is based on an analytical method published by the USEPA. Each specifies:

- Procedures for sample preparation;
- Instrument start up and performance check;
- Initial and continuing calibration check requirements;
- Specific methods for each sample matrix type; and
- Required analysis procedures.

Table B-4 provides a list of parameters and quantitation limits. Quality assurance objectives for laboratory parameters are included in Table B-5.

c. Laboratory Corrective Action

Corrective actions may be required for two classes of problems: analytical or equipment problems and noncompliance problems. Specific corrective actions are presented in the laboratory SOPs for each analytical method (Attachment B-2). Specific corrective actions are not repeated in the text of this QAPP to avoid redundancy, which could become burdensome during the RI phase of corrective action, where the possible use of numerous additional analytical methods may be required.

Analytical or equipment problems may occur during sample preparation, laboratory instrumental analysis, or data review. Corrective measures for these types of problems are discussed in the following sections.

For noncompliance problems, a formal corrective action program will be determined and implemented at the time the problem is identified. The person who identifies the problem is responsible for notifying the EnChem QA Officer or ENVIRON Project Manager. Implementation of corrective action for noncompliance problems will be confirmed in writing through the same channels.

Laboratory personnel are alerted that corrective actions may be necessary if:

- QC data are outside the warning or acceptable windows for precision and accuracy;
- Blanks contain target analyses above acceptable levels;
- Undesirable trends are detected in spike recoveries or Relative Percent Difference between duplicates;
- There are unusual changes in detection limits;

- Deficiencies are detected by the Laboratory QA Officer during internal or external audits or from the results of performance evaluation samples; or
- Inquiries concerning data quality are received.

In addition, the data for trip blanks, etc., will be monitored for contamination, and corrective actions will be taken as soon as a problem is identified. This will be accomplished either by discontinuing the use of a specific bottle lot, contacting the bottle supplier(s) for re-testing the representative bottle from a suspect lot, re-sampling the suspected samples, validating the data taking into account that the contaminants could be introduced by the laboratory (i.e., common lab solvents, sample handling artifacts, etc.) or could be bottle QC problem, so as to make an educated determination of whether the bottles and hence the data are still usable, etc., whichever is appropriate.

For this particular project, the corrective actions will be conducted in a comprehensive manner in order to avoid the use of identified contaminated lot(s), and to ensure that the bottle supplier(s) is deemed responsive and able to provide clean bottles as specified.

Corrective action procedures are often handled at the bench level by the analyst, who reviews the preparation or extraction procedure for possible errors, and checks the instrument calibration, spike and calibration mixes, instrument sensitivity, and so on. If the problem persists or cannot be identified, the matter is referred to the Laboratory Project Manager or Operations Manager. Once resolved, full documentation of the corrective action procedure is filed with the Laboratory QA Director.

5. Quality Control

The number of duplicate and blank samples to be collected is listed in Table B-1. Sample procedures are specified below. The evaluation protocols and acceptance criteria for field duplicate samples, split samples, and trip blanks are stated below.

a. Field Duplicate and Split Samples

Field duplicates or split samples should be identified. Reported results for each sample and duplicate will be compared by calculating the relative percent difference (%RPD), as defined in Section D.1. Positive results for a target compound will be flagged "J" in the sample and its duplicate or split if the following criteria are not met:

- A control limit of +/-20% for aqueous samples (40% for solid samples) for the %RPD will be used if both the sample and its duplicate or split results are greater than 5X the Contract Required Quantitation Limit (CRQL).

- If either the sample or its duplicate or split result is less than 5X the CRQL, then for aqueous samples, if the %RPD is less than the CRQL, and for solid samples, if the %RPD is less than 2X the CRQL.

b. Trip Blanks

The evaluation protocol and acceptance criteria for trip blanks are identical to those described for method blanks as discussed in SectionD.1.

c. Field Duplicate Collection

The general level of the QC effort will be one field duplicate for every 20 or fewer investigative samples. The field duplicate samples will be analyzed for the same list of parameters as the ground water or soil sample with which they are collected.

Duplicate samples will be collected in accordance with the general procedure described below:

1. The investigative sample location from which a duplicate sample will be collected will be identified.

2. The duplicate sample will be collected at the same time and location as one of the investigative samples using the same procedure outlined in the sampling procedure for investigative samples.

3. The investigative sample containers for VOC analyses will be completely filled and sealed. and then the duplicate sample container will be filled and sealed. For all other sample parameter containers, the duplicate and investigative sample containers will be alternately filled.

4. The field notebook, sample log sheet, labels, and chain-of-custody sheets will be filled out with the duplicate sample properly designated and logged.

5. The duplicate sample will be preserved, handled, and shipped following the same procedures as the investigative samples.

d. Matrix Spike/Matrix Spike Duplicate Collection

MS/MSD samples are investigative samples. Soil MS/MSD samples require no extra volume for VOCs or extractable organics. However, aqueous MS/MSD samples must be collected at triple the volume for VOCs. One MS/MSD sample will be collected/designated for every 20 or fewer investigative samples per sample matrix (i.e., ground water, soil). Samples designated for MS/MSD analysis will be collected following the same procedure as other investigative samples, except that additional volume for the aqueous samples will be collected, as necessary.

e. Trip Blank Preparation

Trip blanks are deionized organic free water samples in VOC vials provided by the analytical laboratory and stored with VOC sample containers before filling and during shipment. These samples will remain unopened. If these "blanks" show "detectable" for one or more compounds, the problem could be cross-contamination between sample and container via air in the storage or shipment containers, or lab contamination. One VOC trip blank consisting of deionized organic-free water will be prepared at the EnChem laboratory and included along with each shipment of aqueous VOC samples.

f. Laboratory QC Procedures

The laboratory SOPs include a QC section which addresses the minimum QC requirements for the analysis of specific analyte groups. For the RI, no specific compounds will be added to the spiking solution; rather standard QA/QC measures will be implemented.

If it cannot be demonstrated that the selected compounds are being analyzed in an accurate and precise manner, the analytical approach will be reviewed and changed as necessary. An example of a potential change is

modification of the matrix spike spiking solution to include the poor purging compounds. The list of VOC spiking compounds presented in this QAPP is consistent with the recommendations of the referenced analytical Method 8260.

A summary of the additional QC checks and samples analyzed by the laboratory, including the required frequency, acceptance criteria and corrective actions for those QC checks and samples, is included in Attachment B-3 to this QAPP. All field QC samples to be collected are described above and in the FSP.

6. Instrument/Equipment Testing, Inspection, and Maintenance

a. Field Instrument Preventive Maintenance

PIDs, an XRF analyzer, pH meters, conductivity meters, and dissolved oxygen meters will be used during the field investigation. Specific preventative maintenance procedures to be followed are those recommended by the manufacturer. These instruments will be checked daily and calibrated at an interval recommended by the manufacturer. Backup instruments and equipment will be available on-site or within one-day shipment to avoid delays in the field schedule.

b. Laboratory Instrument Preventive Maintenance

As part of their QA/QC program, a routine preventive maintenance program is conducted by EnChem to minimize the occurrence of instrument failure and other system malfunctions. EnChem staff will perform preventive maintenance and repair or coordinate with a vendor for the repair of all instruments. All laboratory instruments are maintained in accordance with manufacturer's specifications and the requirements of the specific method

employed. This maintenance is carried out on a regular, scheduled basis, and is documented in the laboratory instrument service logbook, for each instrument. Emergency repair or scheduled manufacturer's maintenance is provided under a repair and maintenance contract with factory representatives.

7. Instrument/Equipment Calibration and Frequency

This section describes procedures for maintaining the accuracy of all instruments and measuring equipment used for conducting field and laboratory analyses. The field analyst will calibrate these instruments and equipment prior to each use or on a scheduled, periodic basis.

a. Field Instrument Calibration

Photoionization detectors (PIDs), an XRF analyzer, pH meters, conductivity meters, and dissolved oxygen meters, will be used during the field investigation. All instruments will be calibrated at the interval specified in the manufacturer's instructions and noted below. Calibration data shall be recorded in dedicated calibration logbooks or field logbooks.

b. Laboratory Instrument Calibration

Calibration of laboratory equipment will be based on approved written procedures contained in the laboratory SOPs (Attachment B-2) for the appropriate analytical methods listed in Table B-1. Records of calibration, repairs, or replacement will be filed and maintained by the designated laboratory analyst. These records will be filed at the location where the work is performed-and will be subject to QA audit. For all instruments, the laboratory maintains in-house spare parts and/or service contracts with vendors.

8. Inspection/Acceptance of Supplies and Consumables

Consumable field items will be inspected by the ENVIRON Field Activities Coordinator prior to acceptance and use. Only calibration gases provided by the equipment supplier will be used. Deionized water will be obtained from commercial suppliers. Potable water will be obtained from the city water supply.

The sample containers used for this RI will be prepared in accordance with the USEPA, 1990 guidance document, Contaminant-Free Sample Containers. The bottles used for sampling will not contain contaminants exceeding the level specified in the above-mentioned document. The bottle supplier will issue a certificate of cleanliness for all bottles used. The certificate will be specific for the compounds of concern at the site VOCs and metals. Specifications for the bottles will be verified by checking the supplier's certified statement and analytical results for each bottle lot, and will be documented on a continuing basis. These data will be maintained in a central contractor file and will be available, if requested, for USEPA/IEPA review.

9. Non-Direct Measurements

No data needed to complete this project will be obtained from non-measurement sources.

10. Data Management Procedures

a. Field Data Reporting

Field logbooks will provide the means of recording data collection activities. As such, entries will be described in as much detail as possible so that persons going to the facility could reconstruct a particular situation without reliance on memory. Field logbooks will be bound field survey books or notebooks. Logbooks will be assigned to field personnel, but will be stored

in the document control center when not in use. Each logbook will be identified by the project-specific document number.

The title page of each logbook will contain the following:

- Person to whom the logbook is assigned;
- Logbook number;
- Project name;
- Project start date; and
- End date.

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

A record will be kept of field measurements and collected samples. All entries will be made in ink signed and dated and no erasures will be made. If an incorrect entry is made, the information will be crossed out with a single strike mark signed and dated by the sampler. The number of photographs taken of the station, if any, will also be noted. All equipment used to make measurements will be identified.

The equipment used to collect samples will be noted, along with the time of sampling, sample description, depth at which the sample was collected, volume and number of containers. The sample identification number will be assigned prior to sample collection. Field duplicate samples, which will receive an entirely separate sample identification number, will be noted under

sample description. The system for assigning a sample identification number is described in the FSP.

Field data reporting shall be conducted principally through the transmission of spreadsheets containing tabulated results of all measurements made in the field, and documentation of all field calibration activities. If field logbook information is to be used in the project reports, it will likely be presented in a tabular format.

b. Laboratory Data Reporting

Upon acceptance of the preliminary reports by the Laboratory QA Officer, final reports will be generated and signed by the Laboratory Project Manager. The laboratory package shall be presented in the same order in which the samples were received.

EnChem will prepare and retain full analytical and QC documentation similar to that required by the contract laboratory program. Such retained documentation need not be hard (paper) copy, but may be in other storage media (e.g., electronic). EnChem will supply a hard copy of the retained information on an as needed or as requested basis.

EnChem will report the data in the same chronological order in which analyses are conducted, along with QC data. Each analytical data package will include the following:

- cover sheets (Signature Page) listing the samples included in the report and narrative comments describing problems encountered in analysis;

- tabulated results of organic compounds identified and quantified for investigative and blank samples (Form I from LIMs);

- analytical results for sample spikes, sample duplicates, and laboratory control samples; and (Form III)

- tabulation of instrument detection limits determined in pure water.

For organic analyses, surrogate spike recoveries, chromatograms, GC/MS spectra, calibration verification of standards and blanks, GG/MS system tuning data, standard procedural blanks, and raw data system printouts (or legible photocopies) identifying data of analyses, analyst's name, and parameters determined, will be retained by EnChem. These data will be provided to Heartland for the performance of data validation.

All data generated for the RI will be computerized in a format organized to facilitate data review and evaluation. The RI data set will be available for controlled access by the ENVIRON Project Manager and by authorized personnel. The final data deliverables will be presented in a "CLP-like" format.

C. Group C: Assessment and Oversight

1. Assessments and Response Actions

Corrective action is the process of identifying, recommending, approving and implementing measures to counter unacceptable procedures or out of quality control performance, which can affect data quality. Corrective action can occur during field activities, laboratory analyses, data validation and data assessment. All corrective action proposed and implemented should be documented in the regular quality assurance reports to management. Corrective action should only be implemented after approval by the ENVIRON Project Manager, or his designee.

For noncompliance problems, a formal corrective action program will be determined and implemented at the time the problem is identified. The person who identifies the problem is responsible for notifying the EnChem QA Officer or ENVIRON Project Manager, as appropriate. Any non-conformance with the established quality control procedures in this QAPP or FSP will be identified and corrected in accordance with this QAPP.

Performance and system audits of both field and laboratory activities will be conducted to verify that sampling and analysis are performed in accordance with the procedures established in the FSP and the QAPP. The audits of field and laboratory activities include two separate independent parts: internal and external audits.

a. Field Performance and System Audits

i. Internal Field Audits

Field Sampling Technical System Audits (TSAs) of field activities will be conducted by the ENVIRON QA Director or a designated representative. An initial TSA will be conducted at the onset of the

Phase I field sampling activities to verify that all established procedures are followed. In addition, ENVIRON plans to perform one field TSA during the second week of the Phase I sampling program and one TSA during the first week of the Phase II sampling program. The Phase I TSA will include a Field Analytical Audit, including an audit of field XRF and PID measurement procedures. The need for additional internal audits will be determined by the ENVIRON QA Director. An example of a situation that would likely trigger an audit would be difficulties in obtaining reproducible field laboratory data that could not be attributed to laboratory procedures. In such a situation, an internal audit of field sampling procedures may be warranted to assist in identifying the cause of the difficulties. Another example of a situation where an internal audit may be conducted would be following the implementation of significant corrective action activities.

The field sampling TSAs will be conducted by the ENVIRON QA Manager and/or ENVIRON Field Activities Coordinator. The audits will include examination of field sampling and measurement records, field instrument calibration and operating records, and sample collection, handling, packaging, and documentation in compliance with the established procedures.

ii. External Field Audits

An external audit may be conducted at the discretion of the USEPA/IEPA Project Manager. External field audits may be conducted any time during the field operations. These audits may or may not be announced and are at the discretion of the USEPA/IEPA. External field audits will be conducted according to the field activity information presented in the QAPP.

b. Laboratory Performance and Systems Audits

i. Internal Laboratory Audits

The internal performance and system audits of EnChem will be conducted by the Respective Laboratory QA officers. The system audits, which will be done on an annual basis, will include examination of laboratory documentation on sample receiving, sample log-in, sample storage, chain-of-custody procedure, sample preparation and analysis, and instrument operating records.

The internal lab system audits will include an examination of laboratory documentation on sample receiving, sample log-in, sample storage, chain-of-custody procedures, sample preparation and analysis, instrument operating records, etc. The performance audits will involve preparing blind QC samples and submitting them along with project samples to the laboratory for analysis throughout the project. EnChem's QA Officer will evaluate the analytical results of these blind performance samples to ensure the laboratory maintains acceptable QC performance.

ii. External Laboratory Audits

Any external audits will be conducted at the discretion of the USEPA/IEPA Project Manager.

2. Reports to Management

TSA audit results will be provided in writing to the ENVIRON Project Manager within two weeks of completion of the audit. In addition, the QA manager and ENVIRON field personnel will report any field QA difficulties or lack of

compliance to the ENVIRON project manager as specified in Section C.1. Issues will include any problems regarding sampling, field measurement, sample handling, communication or documentation. The laboratory QA officer will report any laboratory QA problems to the ENVIRON project manager. The ENVIRON project manager will maintain a file of any checklists or corrective action letters during the project, to be compiled and included in the Final Report.

The Final RI Report will contain separate QA sections in which data quality information collected during the task is summarized. The final report will be the responsibility of the ENVIRON Project Manager and will include the appropriate EnChem QA Officer reports on the accuracy, precision, and completeness of the data as well as the results of the TSAs, and any corrective action needed or taken, provided analytical data were generated for the report.

The QA sections of the Final RI Report will contain a discussion of the issues noted above. It will also include a discussion of the data quality objectives any qualified data, and major project problems. The section will also provide justification for use of qualified and/or if necessary, recommend that supplemental data be collected to replace the affected data set. The project-specific issues to be included in the Final RI Report are outlined in Section II.D of the RI/FS Work Plan.

D. Group D: Data Validation and Usability

1. Data Review, Verification, and Validation

All chemical analysis data generated will be reviewed and validated based upon its accuracy, precision, and completeness as defined in the following sections. A Data Validation Report will be prepared by ENVIRON/Heartland at the completion of the RI activities. We anticipate two validation reviews by Heartland (for Phase I and Phase II data). However, the reviews may be separated into additional tasks as receipt of results allows.

a. Accuracy Assessment

Accuracy of laboratory results will be assessed for compliance with the established QC criteria that are described in Section B.5 using the analytical results of method blanks, reagent/preparation blanks, MS/MSD samples, equipment rinse blanks, and trip blanks. The percent recovery (%R) of matrix spikes will be calculated using:

$$\%R = [(A - B) / C] \times 100$$

Where:

A = the analyte concentration determined experimentally from the spiked sample;

B = the background level determined by a separate analysis of the unspiked sample; and

C = the amount of the spike added.

Blanks (method, equipment, and trip) will be evaluated according to the USEPA Contract Laboratory Program National Functional Guidelines for Organic (NFGO) Data Review and the USEPA National Functional Guidelines for Inorganic (NFGI) Data Review. The NFGO and NFGI state that if a contaminant is detected in a sample at a concentration less than five times the concentration detected in any associated blank (less than ten times for the common lab contaminants), the contaminant should be qualified as not detected.

b. Precision Assessment

Precision of laboratory analyses will be assessed by comparing the analytical results between duplicate samples (investigative and MS/MSD). The relative percent difference (%RPD) will be calculated for each pair of duplicate analyses using:

$$\%RPD = (S - D) / [(S + D) / 2] \times 100$$

Where:

S = first sample value (original or MS value)

D = second sample value (duplicate or MSD value)

c. Completeness Assessment

The data completeness of laboratory analyses results will be assessed for compliance with the amount of data required for decision-making. The completeness is calculated using:

Completeness = [(valid data obtained) / (total data planned)] x 100

2. Verification and Validation Methods

a. Procedures Used to Validate Field Data

Procedures to evaluate field data for this project primarily include checking for transcription errors and review of field logbooks, on the part of field crew members. This task will be the responsibility of the ENVIRON Field Activities Coordinator.

b. Procedures Used to Validate Lab Data

EnChem will perform in-house analytical data validation under the direction of the Laboratory QA Officer. The Laboratory QA Officer is responsible for assessing data quality and advising of any data which were rated "preliminary" or "unacceptable" or other notations which would caution the data user of possible unreliability.

Data validation by the laboratory will be conducted as follows:

- The Laboratory QA Officer will complete a thorough audit of preliminary reports at a frequency of one in ten.
- The Laboratory QA Officer and area supervisors will decide whether any sample re-analysis is required.

The EnChem assessment of laboratory data will be accomplished by the joint efforts of their respective Laboratory QA Officers and Laboratory Project Managers. The data assessment by the Laboratory Project Manager will be based on the assumption that the sample was properly collected and handled according to the FSP and Section D of this QAPP.

EnChem data reviewers will conduct a systematic review of the data for compliance with the established QC criteria based on the spike, duplicate and

blank results provided by the laboratory. An evaluation of data accuracy, precision, sensitivity and completeness based on criteria discussed in Section D.1 of this QAPP will be performed.

Data validation independent of EnChem will be performed at a frequency of one hundred percent. Independent data validation will be performed by Heartland Environmental Services, Inc. (Heartland) in accordance with the USEPA Contract Laboratory NFGO and NFGI Data Reviews.

The data reviewers will identify any out-of-control data points and data omissions and will interact with the laboratory to correct data deficiencies. Decisions to repeat sample collection and analyses may be made by the ENVIRON Project Manager based on the extent of the deficiencies and their importance in the overall context of the project. The ENVIRON Project Manager may seek USEPA/IEPA input and approval prior to repeating any sample collection and analysis, as appropriate.

3. Reconciliation with User Requirements

ENVIRON will review the data validation results to determine the data's accuracy, precision, and completeness objectives as defined in Section D.1 of this QAPP.

The response to address data that does not meet the QC objectives noted above will be based on whether the data is critical or non-critical. Critical data is defined as that which is required to achieve project objectives as follows:

- Complete the determination of the extent and magnitude of impact beyond the Site property limits;
- Characterize and quantify contaminant migration in the shallow aquifer;
- Perform the baseline risk assessment; and

- Evaluate response action alternatives.

Should critical data be found to not meet QC objectives, the ENVIRON Project Manager will take an appropriate course of action to obtain acceptable data. This may include collecting new investigative samples, re-analyzing existing samples, or other action that will result in obtaining acceptable data. The specific course of action will be determined on a case-by-case basis.

Non-critical data is defined as that which, while providing useful information, is not critical in completing the project objectives. Non-critical data that does not meet the QC objectives will be appropriately documented; however, re-sampling or re-analysis to address such data will not typically be performed.

E. References

USEPA (1999) USEPA Contract Laboratory Program, National Functional Guidelines for Inorganic Data Review.

USEPA (1999) EPA Requirements for Quality Assurance Project Plans, Interim Final, EPA QA/R-5, Washington, D.C.

USEPA (1999) USEPA Contract Laboratory Program, National Functional Guidelines for Organic Data Review.

USEPA (2000) Instructions on the Preparation of a Superfund Division Quality Assurance Project Plan- Region 5, Revision 0, based on EPA QA/R-5.

APPENDIX B
TABLES

Table B-1
Summary of Sampling and Analysis Program

Sampling Area	*Matrix*	*Field Parameters*	*Analyses*	*SW-846 Laboratory Methods*	*Number of Samples Investigative Samples (Estimated)*	*Duplicates*	*Trip Blanks*	*MS/MSD*
On-Site areas 1-4, undeveloped site areas, manufacturing area	Soil	Visual, PID Screening, XRF	XRF TAL metals TCL organics PCBs	6010/6020 8260B/8270C 8082	130 26 13 13	1/20		1/20
Residual Piles	Residue	Visual, PID	TCLP metals SPLP metals	1312/6010/6020 1311/6010	20 20	1/20		1/20
Eastern and Western Drainageways	Sediment	Visual, PID	TAL metals TCL organics PCBs	6010/6020 8260B/8270C 8082	16 4 4	1/20		1/20
	Surface Water	Visual	TAL metals TCL organics PCBs Sulfates	6010/6020 8260B/8270C 8082 9036	15 4 4 15	1/20	1/shipment	1/20
Site-wide	Ground Water (temporary wells)	Visual	TAL metals TCL organics PCBs	6010/6020 8260B, 8270C 8082	10 4 4	1/20	1/shipment	1/20
	Ground Water (permanent wells)	Visual, pH, conductivity, Temperature Dissolved Oxygen	TAL metals TCL organics PCBs Sulfates	6010/6020 8260B,8270C 8082 9036	23 4 4 23	1/20	1/shipment	1/20

Key:

PID = Photoionization Detector
XRF = X-Ray Fluorescence Analyzer
PCOCs = Potential Contaminants of Concern for Groundwater (Cadmium, Lead, Manganese, Zinc, Iron, and Sulfate)
MS/MSD = Matrix Spike/Matrix Spike Duplicate

Table B-2
Sampling Containers, Preservation, and Holding Times

Matrix	*Parameter*	*Container*	*Preservation*	*Hold Times*	*Sample Volume*
Water	Volatile Organics	Glass Vials	HC1 to pH <2; Cool to 4°C	14 days	3 x 40 mL
Soil	Volatile Organics	3 *En Core* Samplers and 1 poly jar	Cool to 4°C	14 days	Two 5-gram *En Core* samplers, one 25 gram *En Core* sampler and one 5-ounce size poly jar
Water	Semi Volatile Organics	Amber Glass	Cool to 4°C	7 days to extractions 40 days till analysis	2 X 1000 mL
Soil	Semi Volatile Organics	Amber Glass	Cool to 4°C	14 days to extractions 40 days till analysis	250 g (500 mL)
Water	Sulfate	Poly Bottle	Cool to 4°C	28 days	250 mL
Soil	TCLP/SPLP Metals	Poly/glass	Cool to 4°C	180 days to extraction 180 days till analysis	400 g
Soil	Metals*	Poly/glass	Cool to 4°C	6 months	100 g
Water	Metals*	Poly/Glass	HNO_3 to pH <2	6 months	500 mL
Soil	Chromium VI	Poly/glass	Cool to 4°C	24 hours	50 g
Water	Chromium VI	Poly/Glass	Cool to 4°C	24 hours	500 mL

*Including TAL metals and metals analyzed specifically as PCOCs

Table B-3

QA Objectives for Field Measurements

PARAMETER	METHOD[1] REFERENCE	PRECISION[2]	ACCURACY[3]	COMPLETENESS
WATER				
Standing Water Levels	Solinist Water Level Indicator	±0.01 ft.	0.005 ft.	95%
Temperature	E170, Mercury Thermometer or Electronic Temperature Probe	±0.5°C	1.0°C	95%
Conductivity	E120.1, Electrometric	±25	10 umho/cm^2	95%
pH	E150.1	±0.1 pH units	0.05 pH units	95%
Dissolved Oxygen	SM-A4500	±0.05 mg/L	10 mV ±0.1 mg/L	95%
SOIL				
Organic Vapor Screening	Photoionization Detector (PID)	±0.1-1.0 ppmV	±2.0 ppmV or 10% of reading	95%
Metals Field Analysis	XRF Analyzer	Instrument/Soil Specific	Instrument/Soil Specific	95%

NOTES:

1. Methods: E - *Method for Chemical Analysis for Water and Wastes* (U.S. EPA, 1983).
   SW - *Test for the Evaluation of Solid Waste*, SW-846, U.S. EPA, September 1986, Update III, June 1997.
   SM - *Standard Methods for Examination of the Water and Wastewater*, 18th ed. (APHA, 1992).
   ASTM - *Annual Book of ASTM Standards*, American Society of Testing and Materials, 1995.
2. Expressed as the acceptable deviation from the Scale.
3. Expected based on equipment manufacturer specifications.

Table B-4
Quantitation Limits for Target Compounds

* - Unpublished	VOCs Reporting Limits					
	Aqueous (ug/L)			Solid (ug/kg)		
COMPOUND	Project Quantitation Limit	Analytical Method MDL	En Chem RL	Project Quantitation Limit	Analytical Method MDL	En Chem RL
1,1-Dichloroethane	1.0	0.87	1.0	5.0	2.8	5
1,1-Dichloroethene	1.0	0.56	1.0	5.0	2.1	5
1,1,1-Trichloroethane	1.0	0.65	1.0	5.0	3.3	5
1,1,2-Trichloro-1,2,2-Trifluroethane	1.0	0.93	1.0	5.0	1.9	5
1,1,2-Trichloroethane	1.0	0.5	1.0	5.0	1.7	5
1,1,2,2-Tetrachloroethane	1.0	0.77	1.0	5.0	1.5	5
1,2-Dibromo-3-chloropropane	1.0	0.88	1.0	5.0	2.9	5
1,2-Dibromoethane	1.0	0.66	1.0	5.0	1.4	5
1,2-Dichlorobenzene	1.0	0.55	1.0	5.0	3.3	5
1,2-Dichloroethane	2.0	1.2	2.0	10.0	6.5	10
1,2-Dichloropropane	1.0	0.39	1.0	5.0	1.4	5
1,2,4-Trichlorobenzene	1.0	0.57	1.0	5.0	1.6	5
1,3-Dichlorobenzene	1.0	0.58	1.0	5.0	1.3	5
1,4-Dichlorobenzene	1.0	0.63	1.0	5.0	1.3	5
2-Butanone	5.0	4	5.0	5.0	4.4	5
2-Hexanone	5.0	1.2	5.0	5.0	1.7	5
4-Methyl-2-pentanone	5.0	0.91	5.0	5.0	1.5	5
Acetone	5.0	3.3	5.0	5.0	3.8	5
Benzene	1.0	0.25	1.0	5.0	1.5	5
Bromodichloromethane	1.0	0.23	1.0	5.0	1.3	5
Bromoform	1.0	0.45	1.0	5.0	1.4	5
Bromomethane	1.0	0.87	1.0	5.0	3.5	5
Carbon Disulfide	1.0	0.5	1.0	5.0	1.3	5
Carbon Tetrachloride	1.0	0.47	1.0	5.0	3.2	5
Chlorobenzene	1.0	0.58	1.0	5.0	1.5	5
Chlorodibromomethan/ Dibromchloromethane	1.0	0.84	1.0	5.0	1.8	5
Chloroethane	1.0	0.84	1.0	5.0	2.0	5
Chloroform	1.0	0.45	1.0	5.0	3.7	5
Chloromethane	1.0	0.27	1.0	5.0	2.3	5
cis-1,2-Dichloroethene	1.0	0.81	1.0	5.0	3.4	5
cis-1,3-Dichloropropene	1.0	0.57	1.0	5.0	1.4	5
Cyclohexane	5.0	1.2	5.0	5.0	2.7	5
Dichlorodifluoromethane	1.0	0.57	1.0	5.0	2.0	5
Ethylbenzene	1.0	0.53	1.0	5.0	1.1	5
Isopropylbenzene	5.0	0.66	5.0	5.0	1.4	5
Methyl tert-Butyl Ether	5.0	0.87	5.0	5.0	4.3	5
Methyl Acetate	1.0	1.9	1.0	5.0	4.4	5

Table B-4
Quantitation Limits for Target Compounds

* - Unpublished	VOCs Reporting Limits					
	Aqueous (ug/L)			Solid (ug/kg)		
COMPOUND	Project Quantitation Limit	Analytical Method MDL	En Chem RL	Project Quantitation Limit	Analytical Method MDL	En Chem RL
Methylcyclohexane	5.0	0.73	5.0	5.0	1.7	5
Methylene Chloride	5.0	0.47	5.0	5.0	1.5	5
Styrene	1.0	0.62	1.0	5.0	1.3	5
Tetrachloroethene	1.0	0.63	1.0	5.0	1.7	5
Toluene	1.0	0.84	1.0	5.0	1.4	5
trans-1,2-Dichloroethene	1.0	0.8	1.0	5.0	3.2	5
trans-1,3-Dichloropropene	1.0	0.64	1.0	5.0	1.6	5
Trichloroethene	1.0	0.39	1.0	5.0	1.4	5
Trichlorofluoromethane	1.0	0.85	1.0	5.0	2.0	5
Vinyl Chloride	1.0	0.11	1.0	5.0	2.4	5
Xylenes (Total)	3.0	1.9	3.0	15.0	3.9	15

Table B-4
Quantitation Limits for Target Compounds

COMPOUND	VOCs Reporting Limits High Level Extraction Solid (ug/kg)		
	Project Quantitation Limit	Analytical Method MDL	En Chem RL
1,1-Dichloroethane	50	15	50
1,1-Dichloroethene	50	17	50
1,1,1-Trichloroethane	50	20	50
1,1,2-Trichloro-1,2,2-Trifluroethane	50	25	50
1,1,2-Trichloroethane	50	23	50
1,1,2,2-Tetrachloroethane	50	23	50
1,2-Dibromo-3-chloropropane	100	32	100
1,2-Dibromoethane	50	21	50
1,2-Dichlorobenzene	50	25	50
1,2-Dichloroethane	100	25	100
1,2-Dichloropropane	50	23	50
1,2,4-Trichlorobenzene	50	23	50
1,3-Dichlorobenzene	50	18	50
1,4-Dichlorobenzene	50	16	50
2-Butanone	250	88	250
2-Hexanone	250	43	250
4-Methyl-2-pentanone	250	27	250
Acetone	250	79	250
Benzene	50	18	50
Bromodichloromethane	50	20	50
Bromoform	50	18	50
Bromomethane	50	20	50
Carbon Disulfide	50	23	50
Carbon Tetrachloride	50	17	50
Chlorobenzene	50	14	50
Chlorodibromomethan/ Dibromchloromethane	50	12	50
Chloroethane	50	19	50
Chloroform	50	18	50
Chloromethane	50	12	50
cis-1,2-Dichloroethene	50	13	50
cis-1,3-Dichloropropene	50	18	50
Cyclohexane	250	18	250
Dichlorodifluoromethane	50	20	50
Ethylbenzene	50	21	50
Isopropylbenzene	50	13	50
Methyl tert-Butyl Ether	50	18	50
Methyl Acetate	250	74	250
Methylcyclohexane	250	20	250
Methylene Chloride	50	17	50
Styrene	50	14	50

Table B-4
Quantitation Limits for Target Compounds

	VOCs Reporting Limits High Level Extraction		
	Solid (ug/kg)		
	Project Quantitation Limit	Analytical Method MDL	En Chem RL
Tetrachloroethene	500	14	500
Toluene	50	17	50
trans-1,2-Dichloroethene	50	21	50
trans-1,3-Dichloropropene	50	24	50
Trichloroethene	50	25	50
Trichlorofluoromethane	50	22	50
Vinyl Chloride	50	19	50
Xylenes (Total)	150	46	150

Table B-4
Quantitation Limits for Target Compounds

COMPOUND	SVOCs Reporting Limits					
	Aqueous (ug/L)			Solid (ug/kg)		
	Project Quantitation Limit	Analytical Method MDL	En Chem RL	Project Quantitation Limit	Analytical Method MDL	En Chem RL
1,1'-Biphenyl	10	5.3	10	830	124	830
2-Chloronapthalene	10	4.2	10	330	132	330
2-Chlorophenol	10	1.1	10	330	109	330
2-Methylnaphthalene	10	3.9	10	330	109	330
2-Methylphenol	10	2.3	10	330	149	330
2-Nitroaniline	25	4.2	25	330	122	330
2-Nitrophenol	10	3.7	10	330	121	330
2,2'-oxybis(1-Chloropropane)	10	3.6	10	330	96	330
2,4-Dichlorophenol	10	3.7	10	330	138	330
2,4-Dimethylphenol	10	2.9	10	330	124	330
2,4-Dinitrophenol	25	3.0	25	330	59	330
2,4-Dinitrotoluene	10	1.7	10	330	138	330
2,4,5-Trichlorophenol	25	4.6	25	330	130	330
2,4,6-Trichlorophenol	10	4.0	10	330	125	330
2,6-Dinitrotoluene	10	3.7	10	330	138	330
3-Nitroaniline	25	2.8	25	830	158	830
3,3'-Dichlorobenzidine	10	2.8	10	330	211	330
4-Bromophenyl-phenylether	10	3.6	10	330	145	330
4-Chloro-3-methylphenol	10	4.1	10	330	133	330
4-Chloroaniline	10	4.2	10	330	141	330
4-Chlorophenyl-phenylether	10	4.7	10	830	140	830
4-Methylphenol	10	2.0	10	330	128	330
4-Nitroaniline	25	1.8	25	830	160	830
4-Nitrophenol	25	1.8	25	330	107	330
4,6-Dinitro-2-methylphenol	25	1.7	25	330	113	330
Acenaphthene	10	4.6	10	830	124	830
Acenaphthylene	10	4.7	10	830	137	830
Acetaphenone	10	4.5	10	330	86	330
Anthracene	10	2.8	10	330	165	330
Atrazine	10	2.2	10	330	172	330
Benzaldehyde	10	8.2	10	830	354	830
Benzo(a)anthracene	10	1.7	10	330	110	330
Benzo(a)pyrene	10	1.5	10	330	52	330
Benzo(b)fluoranthene	10	2.2	10	330	143	330
Benzo(g,h,i)perylene	10	2.1	10	330	138	330
Benzo(k)fluoranthene	10	2.4	10	330	115	330
bis(2-Chloroethoxy) methane	10	4.5	10	330	112	330
bis(2-Chloroethyl) ether	10	0.87	10	330	96	330
bis-(2-Ethylhexyl)phthalate	10	1.4	10	330	157	330
Butylbenzylphthalate	10	1.8	10	330	121	330
Caprolactam	10	1.3	10	330	126	330

Table B-4
Quantitation Limits for Target Compounds

COMPOUND	SVOCs Reporting Limits					
	Aqueous (ug/L)			Solid (ug/kg)		
	Project Quantitation Limit	Analytical Method MDL	En Chem RL	Project Quantitation Limit	Analytical Method MDL	En Chem RL
Carbazole	10	1.4	10	330	135	330
Chrysene	10	1.8	10	330	110	330
Di-n-butylphthalate	10	1.4	10	330	139	330
Di-n-octylphthalate	10	1.3	10	330	134	330
Dibenzo(a,h)anthracene	10	2.4	10	330	58	330
Dibenzofuran	10	4.7	10	330	143	330
Diethylphthalate	10	2.7	10	330	142	330
Dimethylphthalate	10	3.5	10	330	130	330
Fluoranthene	10	1.6	10	330	130	330
Fluorene	10	4.7	10	330	148	330
Hexachlorobenzene	10	1.4	10	670	156	670
Hexachlorobutadiene	10	2.9	10	330	130	330
Hexachlorocyclopentadiene	10	1.4	10	330	142	330
Hexachloroethane	10	2.3	10	330	111	330
Indeno(1,2,3-cd)pyrene	10	1.6	10	330	203	330
Isophorone	10	4.5	10	330	93	330
N-Nitroso-di-n-propylamine	10	4.3	10	330	99	330
N-Nitrosodiphenylamine	10	2.2	10	330	179	330
Naphthalene	10	3.8	10	330	132	330
Nitrobenzene	10	3.5	10	330	101	330
Pentachlorophenol	25	0.78	25	330	125	330
Phenanthrene	10	2.1	10	330	153	330
Phenol	10	1.0	10	330	112	330
Pyrene	10	1.8	10	330	137	330

Table B-4
Quantitation Limits for Target Compounds

* - Unpublished	Metals Reporting Limits					
	Aqueous (ug/L)			Solid (mg/kg)		
ANALYTES	Project Quantitation Limit	Analytical Method MDL	En Chem RL	Project Quantitation Limit	Analytical Method MDL	En Chem RL
Aluminum	200	30	26	20	15	1.7
Antimony	10	21	4.9	1.0	11	0.54
Arsenic	10	35	5.4	1.0	18	0.59
Barium	5.0	0.87	0.45	0.50	0.44	0.024
Beryllium	1.0	0.18	0.58	0.10	0.09	0.062
Cadmium	1.0	2.3	0.37	0.10	1.2	0.058
Calcium	100	6.7	52	50	3.4	1.3
Chromium	3.0	4.7	1.2	0.30	2.4	0.20
Cobalt	3.0	4.7	0.71	0.50	2.4	0.073
Copper	10	3.6	3.5	2.0	1.8	0.13
Iron	50	4.1	51	10	2.1	1.3
Lead	5.0	28	2.6	0.50	14	0.26
Magnesium	30	20	7.7	30	10	1.3
Manganese	2.0	0.93	0.37	0.20	0.47	0.04
Mercury	0.20	0.20	0.044	0.033	0.10	0.0033
Nickel	5.0	10	3.2	0.50	5.0	0.22
Potassium	100	-	11	100	-	2.6
Selenium	10	50	5.2	1.0	25	0.38
Silver	5.0	4.7	1.3	0.50	2.4	0.06
Sodium	500	19	120	200	9.5	12
Thallium	10	27	4.2	1.0	14	0.53
Vanadium	5.0	5.0	0.86	0.50	2.5	0.14
Zinc	20	1.2	5.4	2.0	0.60	0.29
Cyanide	10	10	2.1	1.0	0.50	0.25

Table B-4
Quantitation Limits for Target Compounds

COMPOUND	PCBs Reporting Limits					
	Aqueous (ug/L)			Solid (ug/kg)		
	Project Quantitation Limit	Analytical Method MDL	En Chem RL	Project Quantitation Limit	Analytical Method MDL	En Chem RL
Aroclor-1016	1.0	0.21	1.0	50	13	50
Aroclor-1221	1.0	0.21	1.0	50	13	50
Aroclor-1232	1.0	0.21	1.0	50	13	50
Aroclor-1242	1.0	0.21	1.0	50	13	50
Aroclor-1248	1.0	0.21	1.0	50	13	50
Aroclor-1254	1.0	0.21	1.0	50	13	50
Aroclor-1260	1.0	0.21	1.0	50	13	50

Table B-5
QA Objectives for Laboratory Parameters

COMPOUND	VOCs					
	Aqueous (ug/L)			Solid (ug/kg)		
	LCS	MS	MS %RPD	LCS	MS	MS %RPD
1,1 - Dichloroethane	80-120	82-121	20	70-130	50-153	30
1,1 - Dichloroethene	82-127	83-125	20	70-130	53-156	30
1,1,1 - Trichloroethane	80-120	87-127	20	70-130	28-153	30
1,1,2 - Trichloroethane	80-120	70-130	30	70-130	35-157	30
1,1,2,2 - Tetrachloroethane	74-115	70-130	30	70-130	42-139	30
1,2 - Dichloroethane	80-120	70-130	30	70-130	36-145	30
1,2 - Dichloropropane	80-120	70-130	30	70-130	42-138	30
2 - Butanone	59-122	42-156	30	70-130	57-178	30
2 - Hexanone	60-123	32-174	43	70-130	60-168	30
4 - Methyl - 2 - pentanone	69-119	66-122	25	70-130	41-153	30
Acetone	42-120	38-139	30	70-130	48-178	30
Benzene	80-120	70-130	30	70-130	62-170	30
Bromodichloromethane	80-120	70-130	30	70-130	40-134	30
Bromoform	66-123	66-124	30	70-130	34-133	30
Bromomethane	53-137	57-135	23	70-130	57-150	30
Carbon disulfide	78-130	77-128	38	70-130	21-148	30
Carbon tetrachloride	85-128	84-132	30	70-130	27-149	30
Chlorobenzene	80-120	70-130	30	70-130	37-136	30
Chlorodibromomethane / Dibromochloromethane	80-120	79-119	20	70-130	36-142	30
Chloroethane	73-127	71-127	30	70-130	58-143	30
Chloroform	80-120	70-130	30	70-130	36-147	30
Chloromethane	80-120	70-130	30	70-130	35-136	30
cis - 1,2 - Dichloroethene	48-134	39-138	21	70-130	70-130	30
cis - 1,3 - Dichloropropene	78-120	70-119	20	70-130	38-125	30
Ethylbenzene	80-120	70-130	30	70-130	30-167	30
Methylene chloride	77-117	70-130	30	70-130	27-156	30
Styrene	80-120	70-130	30	70-130	32-139	30
Tetrachloroethene	80-120	88-121	13	70-130	46-142	30
Toluene	80-120	70-130	30	70-130	46-157	30
trans - 1,2 - Dichloroethene	80-120	70-130	30	70-130	35-136	30
trans - 1,3 - Dichloropropene	80-120	70-130	30	70-130	33-151	30
Trichloroethene	80-120	70-130	30	70-130	38-188	30
Vinyl chloride	61-134	62-137	30	70-130	39-148	30
Xylene, total	80-120	73-132	30	70-130	78-184	30
Dibrmofluoromethane (surrogate)	-	61-136	-	-	51-127	-
Toluene $_{d8}$ (surrogate)	-	63-140	-	-	62-126	-
4-Bromofluorobenzene (surrogate)	-	55-136	-	-	60-109	-

Table B-5
QA Objectives for Laboratory Parameters

COMPOUND	VOCs Reporting Limits High Level Extraction Solid (ug/kg)		
	LCS	MS	MS %RPD
1,1 - Dichloroethane	72-119	44-106	30
1,1 - Dichloroethene	62-123	44-117	30
1,1,1 - Trichloroethane	72-125	44-97	30
1,1,2 - Trichloroethane	80-120	44-107	30
1,1,2,2 - Tetrachloroethane	47-128	44-134	30
1,2 - Dichloroethane	73-125	44-105	30
1,2 - Dichloropropane	78-121	44-99	30
2 - Butanone	53-118	44-103	30
2 - Hexanone	61-130	44-105	30
4 - Methyl - 2 - pentanone	70-121	44-100	30
Acetone	58-126	44-102	30
Benzene	75-115	44-109	30
Bromodichloromethane	76-125	44-101	30
Bromoform	72-132	44-96	30
Bromomethane	33-144	44-118	30
Carbon disulfide	64-124	44-87	30
Carbon tetrachloride	72-125	44-94	30
Chlorobenzene	80-120	44-95	30
Chlorodibromomethane / Dibromochloromethane	78-125	44-97	30
Chloroethane	43-141	44-112	30
Chloroform	77-121	44-97	30
Chloromethane	71-119	44-96	30
cis - 1,2 - Dichloroethene	54-101	31-107	30
cis - 1,3 - Dichloropropene	68-112	44-121	30
Ethylbenzene	75-129	44-160	30
Methylene chloride	72-121	44-97	30
Styrene	80-120	44-93	30
Tetrachloroethene	80-120	44-103	30
Toluene	80-120	44-142	30
trans - 1,2 - Dichloroethene	71-119	44-96	30
trans - 1,3 - Dichloropropene	77-126	44-99	30
Trichloroethene	83-130	44-148	30
Vinyl chloride	41-110	44-89	30
Xylene, total	80-120	70-130	30
Dibrmofluoromethane (surrogate)		57-118	-
Toluene $_{d8}$ (surrogate)		72-115	-
4-Bromofluorobenzene (surrogate)		67-112	-

Table B-5
QA Objectives for Laboratory Parameters

COMPOUND	SVOCs					
	Aqueous			Solid		
	LCS	MS	MS %RPD	LCS	MS	MS %RPD
1,2 - Dichlorobenzene	46-104	30-108	30	62-113	49-104	33
1,2,4 - Trichlorobenzene	41-90	36-107	18	67-115	49-109	35
1,3 - Dichlorobenzene	39-87	56-106	30	60-110	44-103	35
1,4 - Dichlorobenzene	39-106	28-118	30	60-111	45-103	35
2 - Chloronaphthalene	56-456	47-124	30	76-119	57-113	24
2 - Chlorophenol	43-92	33-98	24	63-115	46-109	27
2 - Methylnaphthalene	52-114	42-116	30	67-116	53-110	25
2 - Methylphenol	40-86	22-92	28	57-123	40-120	34
2 - Nitroaniline	67-127	56-131	23	64-122	49-120	30
2 - Nitrophenol	50-115	40-118	27	68-118	45-119	33
2,2' - oxybis (1 Chloropropene)	49-109	37-115	21	58-125	41-120	32
2,4 - Dichlorophenol	53-116	43-119	29	72-120	48-116	29
2,4 - Dimethylphenol	42-124	38-126	34	49-119	45-126	38
2,4 - Dinitrophenol	24-145	26-146	34	42-131	23-127	28
2,4 - Dinitrotoluene	25-156	26-156	24	44-156	24-145	28
2,4,5 - Trichlorophenol	58-125	55-124	26	64-134	45-123	32
2,4,6 - Trichlorophenol	56-120	51-121	25	68-119	42-115	30
2,6 - Dinitrotoluene	68-127	65-130	22	70-128	54-121	29
3 - Nitroaniline	63-126	61-141	33	55-107	41-107	31
3,3 - Dichlorobenzidine	44-122	41-151	22	50-114	40-130	35
4 - Bromophenyl phenylether	64-124	58-120	22	67-122	54-113	35
4 - Chloro - 3 - methylphenol	53-116	45-119	30	72-128	43-127	31
4 - Chloroaniline	23-132	16-135	42	38-101	35-107	26
4 - Chlorophenyl phenylether	65-121	56-123	26	72-121	55-116	37
4 - Methylphenol	24-77	18-82	30	62-122	44-117	31
4 - Nitroaniline	38-196	35-198	38	58-147	43-168	27
4 - Nitrophenol	24-91	23-103	30	53-131	36-132	29
4,6 - Dinitro - 2 - Methylphenol	62-146	53-120	33	56-133	45-130	32
Acenaphthene	60-118	49-123	33	66-121	56-110	31
Acenaphthylene	57-120	49-125	32	71-116	54-110	28
Anthracene	74-117	49-125	32	68-115	52-110	31
Benzo(a)anthracene	76-127	75-131	39	68-128	54-118	31
Benzo(a)pyrene	68-131	66-134	27	64-125	48-122	31
Benzo(b)fluoranthene	61-144	55-150	31	65-133	46-135	30
Benzo(g,h,i)perylene	48-159	33-171	31	59-130	40-132	33
Benzo(k)fluoranthene	66-141	66-143	34	66-128	60-126	27
bis(2-Chloroethoxy)methane	52-118	42-119	30	68-116	60-113	32
bis(2-Chloroethyl)ether	50-108	38-112	25	59-116	53-112	36
bis(2-Ethylhexyl)phthalate	72-135	72-138	26	60-141	64-135	32
Butylbenzylphthalate	69-135	66-139	32	70-12	63-127	34
Carbazole	72-144	65-154	19	77-128	65-131	36

Table B-5
QA Objectives for Laboratory Parameters

COMPOUND	SVOCs					
	Aqueous			Solid		
	LCS	MS	MS %RPD	LCS	MS	MS %RPD
Chrysene	76-122	74-124	26	68-117	63-113	28
Dibenzo(a,h)anthracene	75-136	75-137	21	67-134	66-127	29
Dibenzofuran	54-160	45-155	36	63-142	63-148	37
Diethylphthalate	40-149	35-129	22	54-117	43-121	35
Dimethyphthalate	60-121	53-122	28	71-119	54-114	32
Di-n-butylphthalate	68-133	68-139	27	68-129	54-120	32
Di-n-octylphthalate	68-121	67-119	25	64-26	55-114	32
Fluoranthene	63-139	63-141	21	71-123	46-129	34
Fluorene	62-125	56-126	27	64-127	55-118	30
Hexachlorobenzene	67-123	65-122	30	65-121	53-112	32
Hexachlorobutadiene	38-104	31-105	30	62-118	50-110	27
Hexachlorocyclopentadiene	40-115	41-129	30	44-114	39-136	28
Hexachloroethane	58-128	36-118	30	59-118	45-107	27
Indeno(1,2,3-cd)pyrene	56-131	51-162	33	66-136	45-141	33
Isophorone	53-103	49-115	30	57-117	44-125	25
Naphthalene	57-107	43-125	30	64-120	69-137	32
Nitrobenzene	69-132	61-132	24	64-127	43-124	26
N-Nitrosodi-n-propylamine	51-113	38-118	30	68-119	50-114	21
N-Nitrosodiphenylamine	46-119	34-121	26	62-123	44-117	22
Pentachlorophenol	55-141	35-142	26	57-127	38-125	28
Phenanthrene	74-122	73-124	24	70-120	54-115	25
Phenol	40-110	21-114	30	64-119	43-117	24
Pyrene	60-136	59-140	24	66-129	46-118	21
2-Fluorophenol (surrogate)	-	13-70	-	-	35-113	-
Phenol $_{d5}$ (surrogate)	-	8-44	-	-	29-114	-
2-Chlorophenol $_{d4}$ (surrogate)	-	29-104	-	-	34-107	-
1,2-Dichlorobenzene $_{d4}$ (surrogate)	-	34-112	-	-	27-116	-
Nitrobenzene $_{d5}$ (surrogate)	-	34-126	-	-	32-118	-
2-Fluorobiphenyl (surrogate)	-	36-126	-	-	26-126	-
2,4,6-Tribromophenol (surrogate)		39-133			17-129	
Terphenyl $_{d14}$ (surrogate)	-	56-139	-	-	23-141	-

Table B-5
QA Objectives for Laboratory Parameters

ANALYTES	Metals					
	Aqueous			Solid		
	LCS	MS	MS %RPD	LCS	MS	MS %RPD
Aluminum	76-124	64-167	5.0	71-123	75-125	27
Antimony	87-118	90-119	5.0	85-113	72-110	11
Arsenic	88-110	87-121	5.0	78-110	56-106	11
Barium	89-110	88-110	5.0	84-110	34-151	24
Beryllium	90-110	65-115	5.0	83-110	61-106	8.0
Cadmium	86-110	61-112	5.0	82-110	57-110	11
Calcium	92-112	50-151	5.0	85-114	75-125	24
Chromium	88-110	81-110	5.0	83-110	60-111	11
Cobalt	88-110	70-112	5.0	83-110	57-110	7.0
Copper	90-110	79-147	5.0	81-110	75-125	18
Iron	83-116	57-129	6.0	88-116	75-125	25
Lead	88-110	60-114	5.0	75-110	75-125	23
Magnesium	83-116	82-119	5.0	80-110	32-161	22
Manganese	88-110	79-108	5.0	78-110	75-125	20
Mercury	85-117	72-123	6.0	90-113	63-131	15
Nickel	86-110	63-111	5.0	80-110	75-125	12
Potassium	82-110	79-117	5.0	71-110	75-125	8.0
Selenium	84-110	82-129	5.0	73-110	68-110	8.0
Silver	90-110	45-150	6.0	87-111	60-139	16
Sodium	86-110	72-131	5.0	78-110	70-110	8.0
Thallium	83-109	70-113	5.0	78-110	66-110	9.0
Vanadium	91-110	87-110	5.0	82-110	60-111	11
Zinc	86-110	57-114	5.0	83-110	56-105	17
Cyanide	90-110	56-138	20	80-120	43-134	31

Table B-5
QA Objectives for Laboratory Parameters

COMPOUND	PCBs					
	Aqueous			Solid		
	LCS	MS	MS %RPD	LCS	MS	MS %RPD
Aroclor-1016	38-105	27-117	21	45-121	24-124	24
Aroclor-1260	37-109	31-141	18	43-143	26-122	24
DCB (Surrogate)	-	22-133	-	-	11-142	-

ATTACHMENT B-1

Standard Operating Procedures – Field Methods

ATTACHMENT B-1

Standard Operating Procedures – Field Methods

1. Introduction

This Standard Operating Procedure (SOP) describes the protocols to be followed during the collection of soil, sediment, surface and ground water samples, and the decontamination of equipment. These activities will be performed as part of the Remedial Investigation (RI) at the Eagle Zinc Company Site. This SOP is included as part of the RI Quality Assurance Project Plan (QAPP).

2. Soil Sample Collection Procedures

This section describes the procedures that will be completed to collect soil samples. Soil borings will be completed using a direct-push drilling apparatus and macro-core samplers, which produce 4-foot long soil cores. An experienced geologist will prepare a detailed geological log for each soil boring. Initial field screening for volatile organic vapors will be conducted using a portable photoionization detector (PID) and XRF analyzer immediately after sample retrieval. Field measurement operating procedures are included in Section E.

A. Field Screening

The entire soil and sediment core recovered from the borehole will be field screened for VOCs using a PID and for metals using an XRF analyzer. The procedure for field screening the soil and sediment core is as follows:

- The soil will be field screened immediately after the sampling device is removed from the borehole and opened for inspection.
- The soil core will be screened with an organic vapor meter photoionization detector (PID) using a 11.8 eV bulb.
- For VOCs, the field screening will be performed by creating a headspace within the soil core using a stainless steel sampling spatula/spoon. The

probe of the PID will be inserted into this headspace for approximately 15 seconds, until the instrument has measured a maximum reading.

- For metals the field screening will be performed by making measurements at 6-inch increments along the soil core. To obtain measurements for the full range of metals of interest, a dual source analyzer will be used (i.e., requires two radioactive sources to measure the metals of interest).
- Field screening measurements will be collected at 6-inch intervals for the entire length of the recovered soil core. The measurements will be recorded on the boring log.

B. Soil Sampling

The procedure for collecting investigative soil samples is as follows:

- Soil samples will be collected for VOCs using SW-846 USEPA Method 5035 (methanol field preservation method or equivalent methods). Samples for all other parameters will be collected by filling the sample containers to obtain the required sample volumes.
- The investigative samples will be collected first, followed by the QA/QC samples (Field Duplicate and MS/MSD).

All samples will be preserved, handled, and shipped following the procedures described in Section B.2 of the QAPP.

C. Decontamination

All equipment inserted into a soil boring will be decontaminated prior to their use and before leaving the site. More specifically, the sampling equipment will be decontaminated as follows:

Step 1 Steam clean or scrub equipment with a non-phosphate detergent mixed with potable water.

Step 2 Rinse with potable water.

Step 3 Rinse twice with distilled water.

Step 4 Air dry.

Step 5 Place in a clean polyethylene bag or wrap in aluminum foil with shiny-side out when not in use and during transport.

In addition, all down hole drilling equipment (augers, drill bit, and rods) will be decontaminated with a pressurized steam cleaner.

3. Surface Water/Sediment Sample Collection Procedures

This section describes the procedure that will be completed to collect surface water and sediment samples.

A. Surface Water

The procedure for collecting surface water samples is as follows:

- Surface water samples will be taken from the horizontal and vertical center of the channel. Surface water will be collected by submerging the sampling container in the creek facing upstream. Care will be taken not to displace preservative from pre-preserved sample containers. Precautions to be exercised to minimize the risk of contamination from sediment include: (1) not allowing the sample container to contact the sediment during sampling, and (2) filling the sample container in a manner that prevents localized eddy phenomena from stirring up sediment.
- The surface water sampling effort will progress in the upstream direction to minimize cross-contamination of samples.
- Upon completion of sampling, a stake or flag will be placed on the stream bank parallel to the sample location for future resampling and/or surveying.

- The sample location, time, and other observations will be recorded in the field notebook.
- The field notebook, sample log sheets, labels, and chain-of-custody sheets will be filled out with the sample identification properly designated and logged.

The investigative samples will be preserved, handled, and shipped following the procedures described in Section B.2 of the QAPP.

B. Sediment

The procedure for collecting sediment samples is as follows:

- Sediment samples will be collected by submerging a soft sediment dredge such as a Peterson grab sampler into the surface water and carefully lowering the device to the base of the creek. The device will then be closed and raised to the surface where the sample will be will be immediately placed into the appropriate sample containers. A stainless steel spatula/spoon may be used to assist in filling the sample containers.
- Care will be taken to lower and raise the sampling device in a manner to minimize the loss of fine sediment particles.
- If the portion of stream to be sampled is dry the upper inch of sediment will be removed prior to sampling using a stainless steel spatula/spoon. A separate stainless steel spatula/spoon will be used to fill the sample containers.
- Upon completion of sampling, a stake or flag will be placed on the stream bank parallel to the sample location for future resampling and/or surveying.
- The sample location, time, and other observations will be recorded in the field notebook.

- The field notebook, sample log sheets, labels, and chain-of-custody sheets will be filled out with the sample identification properly designated and logged.

C. Decontamination

All surface water and sediment sampling equipment will be decontaminated prior to their use. More specifically, the sampling equipment will be decontaminated as follows:

Step 1 Steam clean or scrub equipment with a nonphosphate detergent mixed with potable water.

Step 2 Rinse with potable water.

Step 3 Rinse twice with distilled water.

Step 4 Air dry.

Step 5 Place in a clean polyethylene bag or wrap in aluminum foil with shiny-side out when not in use and during transport.

4. Ground Water Sample Collection Procedures

This section describes the procedures that will be completed to:

- Determine the ground water level and elevation at each well,
- Obtain ground water samples in open boreholes,
- Develop newly installed monitoring wells,
- Purge monitoring wells prior to sampling, and obtain ground water samples.

All equipment used for ground water level measurement and ground water sampling will be decontaminated prior to sampling and at the completion of the sampling activities by following the procedures described in Section 4.F of this Attachment.

A. Ground Water Elevation Determination

Prior to purging and sampling the monitoring wells, the depth to water and the total well depth will be measured using an electronic water level meter or equivalent. The instrument must have been calibrated by the manufacturer prior to its purchase and therefore should not require calibration in the field. The water levels will be determined to the nearest 0.01 of a foot with an accuracy of ±0.02 feet and the total well depth will be determined to the nearest 0.1 of a foot with an accuracy of ±0.2 feet. The water level meter probe will be decontaminated prior to their use as outlined in Section 4.F.

The depth to water and the total depth of the monitoring well will be used to determine the volume of ground water to be removed from the monitoring well as part of the purging activities.

Ground water level elevations will be calculated by subtracting the measured depth to water from the surveyed top of casing elevation. The field measurements will be recorded in a field notebook and used to prepare piezometric surface maps of the site. An Illinois-licensed professional land surveyor will measure the top of casing elevation of all monitoring wells.

B. Monitoring Well Development

Monitoring well development will be performed to allow the collection of water samples representative of the formation to be monitored. Well development will be conducted as follows:

- The water level and total depth will be measured, as described in section 4.A above, and be used to calculate casing volume. Any accumulation of sediment will be noted.
- Bailers or pumps may be used to remove water/sediment during the development
- At least three times the measured casing volume of water plus three times the saturated volume of the monitoring well sand pack will be removed. If any water is used during drilling activities three times the

quantity of water lost to the formation during drilling will be removed during development. Field-measured parameters of pH, specific conductance, dissolved oxygen, and temperature will be recorded at each casing volume. Both visual clarity and stabilization of field-measured parameters will be used to evaluate effectiveness of well development.

- A detailed description of each well's response during development and all of the field measurements will be recorded.

C. Monitoring Well Purging

Prior to collecting samples, each well will be purged of standing water using a submersible pump if adequate water is present. If adequate water is not available, a bailer will be used to purge monitoring wells. Monitoring wells screened in the overburden will be purged and sampled using low flow (low stress) techniques. Monitoring wells screened in bedrock intervals will be purged and sampled using low flow (low stress)/low volume techniques.

Low Flow Purging

Low flow purging of overburden wells will be conducted as follows:

- The water level and total depth will be measured, as described in section 4.1, and be used to calculate casing volume. Any accumulation of sediment will be noted. Use of a submersible pump or bailer for purging/sampling will be determined.
- If a bailer is used, field parameters will be measured at least every casing volume. The well will be considered purged when a minimum of three times the measured casing volume has been removed, and the field-measured parameters of pH, specific conductance, oxygen reduction potential, dissolved oxygen, turbidity, and temperature have stabilized. Field parameters are considered stabilized when consecutive readings are within 10% or, in the case of pH, 0.1 standard units (SU). If a well bails dry during purging, it will

be considered purged and sampling will commence once a sufficient amount of water has recharged in the well.

- If a submersible pump is used, the pump intake will be placed at the approximate center of the screened interval. The water level in the well should be monitored during pumping, and ideally the pumping rate should equal the well recharge rate with little or no water level draw down in the well (the water level should stabilize at the appropriate pumping rate). If possible, draw down will be minimized (less than 0.3 feet). There should be at least 1 foot of water over the pump intake so there is no risk of the pump suction being broken, or entrainment of air in the sample. Record the pumping rate adjustments and depth(s) to water in the logbook.
- During purging, field parameters will be measured continuously using a flow through cell and recorded at least every casing volume until the parameters stabilize. The well will be considered purged when a minimum of three times the measured casing volume has been removed, and the field-measured parameters of pH, specific conductance, oxygen reduction potential, dissolved oxygen, turbidity, and temperature have stabilized. Field parameters are considered stabilized when consecutive readings are within 10% or, in the case of pH, 0.1 standard units (SU). If a well pumps dry during purging, it will be considered purged and sampling will commence once a sufficient amount of water has recharged in the well.
- A submersible pump intake will be placed at the approximate center of the screened interval. If the length of the screened interval is greater than 10 feet the pump intake will be placed approximately 5 feet from the bottom of the well. The water level in the well should be monitored during pumping, and ideally the pumping rate should equal the well recharge rate with little or no water level draw down in the well (the water level should stabilize at the appropriate pumping rate). If possible, draw down will be minimized (less than 0.3 feet). There should be at least 1 foot of water over the pump intake so there is no risk of the pump suction being broken, or entrainment of air in

the sample. Record the pumping rate adjustments and depth(s) to water in the logbook.

- During purging, field parameters will be measured continuously using a flow through cell until all field parameters stabilize. The field parameters will be recorded at least every half casing volume or every 5 minutes which ever is less. The well will be considered purged when the field-measured parameters of pH, specific conductance, dissolved oxygen, and temperature have stabilized. Field parameters are considered stabilized when 3 consecutive readings are within the following limits: pH within 0.1 SU, specific conductance within 3%, dissolved oxygen (DO) within 10%, and temperature within 3%. Consecutive readings will not taken at less than 3-minute intervals. If a well pumps dry during purging, it will be considered purged and sampling will commence once a sufficient amount of water has recharged in the well.

D. Ground Water Sampling

After each monitoring well has been adequately purged, one round of ground water samples will be collected for laboratory analysis. If sufficient water is present samples will be collected immediate following purging using the bailer/pump used during purging. If using a pump with a flow through cell the samples will be collected directly from the pump discharge prior to discharge to the flow through cell. The pumping rate decreased as low a possible to limit sample disturbance. The ground water sample will be obtained by directly filling the sampling bottles from the bailer or pump discharge in the following order: VOCs, SVOCs, PCBs, metals, sulfate. VOC samples will be collected in 40-milliliter vials, ensuring that there is zero headspace. QA/QC samples will be collected by alternately filling bottles for each sample parameter. VOC sample bottles for both the investigative and the QA/QC sample will be completely filled, purged of headspace, and sealed.

E. Field Instrument Operation, Calibration, and Maintenance

The soil and ground water sampling equipment, including the photoionization detector (PID), XRF analyzer, electric water level meter, and water quality meter (containing the pH, specific conductivity, temperature, and dissolved oxygen probes) will be operated, calibrated, and maintained in the field in accordance to the manufacturer's specifications. A summary of the operating procedures for these instruments is provided below.

Photoionization Detector Operating Procedures:

- Equipment: OVM/DataLogger Model 580B, Thermo Environmental Instruments, Inc. A 11.8 eV lamp will be used with Argon gas and a wavelength of approximately 105 nm
- The OVM 580B is an Organic Vapor Meter that detects and quantitates most organic vapors with a sensitive photoionization detector (PID). It is used to detect volatile organics in the field and in field samples.
- Set up includes: 1) Attach power plug from the back of the unit to the RUN/CHG plug-in and depress the ON/Off switch to turn the unit on. 2) Attach extension to sample inlet at front of unit, 3) Calibrate each day as detailed below, 4) Check battery by observing if low battery signal is on LCD display and charge if needed.
- ENVIRON uses rental PID equipment, which is calibrated by the factory and by the rental company. Field calibration will be performed using the cylinder method at least daily. Calibration by cylinder is described as follows: Isobutylene is chosen as a reference standard (250 ppm) for calibration. A constant flow pressure regulator sets the flow needed for calibration of the 580B unit. The inlet to the unit is connected to the "T" tube that is provided with the calibration cylinder. The other end of the "T" is connected to the regulator on the Standard Cylinder. It is important that a length of tubing is attached to the "T" as this prevents diffusion of ambient air

into the sample line. "Zero" air is assumed to be background air unless field conditions are such that air without hydrocarbons is not available. The calibration steps are:

1. Power up instrument
2. Depress on/off key to ignite lamp and initiate sample pump
3. Depress Mode/Store Key
4. Depress -/CRSR key in response to Log this Value?
5. Depress 1/CRSR key to select parameters mode from the main menu
6. Depress +/INC key to advance thru the Run Mode selection parameter prompt
7. Depress +/INC key to advance thru the Auto Logging Mode selection parameter prompt
8. Depress +/INC key to advance thru the Average Time selection parameter prompt
9. Depress +/INC key to advance thru the Alarm Setting parameter prompt
10. Depress +/INC key to advance thru the Lamp Selection Prompt
11. Depress the +/INC key to advance thru Response Factor Setting parameter prompt.
12. Depress RESET key to initiate calibration sequence
13. Depress the -/CRSR key to decline restoration of the backup calibration
14. Connect the outlet of the calibration tubing assembly to the Model 580 Detector Inlet as describe above
15. Introduce Zero Air to Model 580 by opening the flow regulator
16. Depress RESET Key to zero the unit
17. Close the flow regulator

18. Simultaneously depress RESET and -/CRSR keys to activate the movable cursor
19. Repeat step 18 until cursor is at the ones place
20. Simultaneously depress RESET and +/INC keys to increment the ones place value
21. Repeat step 20 until the ones place value reads 0
22. Repeat step 18 to move cursor to the tens place
23. Repeat step 20 until the tens place value reads 5
24. Repeat step 18 to move the cursor to the hundreds place
25. Repeat step 20 until the hundreds place value reads 2
26. Repeat step 18 to move the cursor to the thousands place
27. Repeat step 20 until the thousands place value reads 0
28. The LCD should now read *SPAN PPM=0250 "+" to continue*
29. Depress the +/INC to accept the span concentration value
30. Connect isobutylene cylinder (250 ppm) to calibration tubing assembly
31. Connect outlet of the calibration tubing assembly to the Model 580B detector Inlet
32. Introduce isobutylene standard to unit by opening flow regulator
33. Reset key and unit will calibrate
34. Close flow regulator
35. Depress +/inc key in response to *Reset to Calibrate* message
36. Depress MODE/STORE to return to Run mode
37. For other calibration gases, and procedures, see operation manual

- Routine Maintenance: Routine maintenance such as lamp replacement, lamp cleaning is performed by the instrument rental service.

- Normal Measurement Procedures include: 1) Turn on equipment as described in Set up procedures above, 2) Once the lamp is lit, the display will show the PPM on the bottom line, 3) set RUN MODE to Max Hold mode and the top line of the display will indicate the maximum reading, 4) Set alarm to project action limit by simultaneously pressing the RESET switch the with either the +/INC switch or the -/CRSR switch, 5) Place unit inlet in area (headspace of soil sample, well, jar or bag sample, breathing zone as appropriate) to be measured and wait for concentration to stabilize, 6) Record reading in field book or field data sheet.
- Data logging and printing features are not expected to be used however instructions are included in the operations manual.
- Instruction manual should be kept with unit at all times to allow for troubleshooting and use of other calibration gases.

XRF Analyzer Operating Procedures

- Equipment: Niton XL-722 X-ray Fluorescence Analyzer
- XRF is used to determine concentration of metals in bulk soil samples.
- Setup Includes: 1) Select SETUP on the menu and verify that XRF is in Bulk Sampling Mode.
- Measurement includes: 1) Turn power on 15 minutes prior to use, 2) Push safety slide out from under the shutter release, 3) Place the unit on the test guard so that the rectangular opening on the test guard is under the window pf the NITON, squeeze the shutter release and press the unit flat against the surface of the test guard, 4) Wait until reading has stabilized (Approximately 30-60 seconds)
- After measurement: 1) turn off the power, 2) wash test guard thoroughly with tap water.
- The XRF is calibrated at the factory but needs to be self-calibrated several prior to use and several times daily to ensure the highest

possible accuracy: 1) Select CALIBRATE AND TEST from the menu and press clear/enter to start self-calibration, 2) Immediately following self-calibration check the unit using the provided standard samples.

- Instruction Manual should accompany the unit at all time to allow troubleshooting using error codes and for instructions regarding replacement of faulty probes.

Electric Water Level Meter Operating Procedures:

- Equipment: Solinst Water Level Meter, Model 101
- Water Level Meter is used to determine depth to water in wells and other locations
- Set up includes: 1) make sure downhole cable and sensor is clean, 2) turn unit on by turn Sensitivity knob clockwise, 3) check battery power by depressing Battery Test button, 4) insert sensor in container of water and test for beep tone.
- Measurement includes: 1) Unit should be on, set Sensitivity knob to approximately the midpoint of the range, 2) Lower sensor into well or measurement location until beep tone is heard. Do not allow the sensor to fall unrestricted to the bottom, 3) Pull up slightly on the cable until tone stops, 4) If tone lingers or is erratic, adjust sensitivity as appropriate, 5) Re-lower sensor until beep tone is heard and read depth on cable markings. The cable is marked with in 0.1 foot divisions, 6) Record depth measurement in field notebook or field form, 7) Use unit handle to reel in the cable and sensor after measurement, 8) Place sensor in metal cylinder-holder located on back side of unit.
- After measurement: 1) Decontaminate cable and sensor as they are retracted from the well or measurement location using soap and water. If necessary they can be soaked in a bucket of soapy water. 2) Rinse cable and sensor with potable water, 3) Turn off unit.

- The sensor and battery should be checked each day. There is no calibration needed for this unit

Water Quality Meter Operating Procedures:

- Equipment: Horiba U-10 Water Quality Checker
- U-10 is used to monitor water parameters during well purge
- Set up includes: 1) Removal of protective rubber cap from pH sensor, 2) check for pH sensor, DO sensor, temperature sensor, conductivity sensor and condition of the probe guard.
- Measurement includes: 1) Turn power on (check battery if no power or if error code *Er1* shows in readout), 2) Gently place the probe into the water sample. Water should be above the level of the DO probe for measurement, 3) when the power is turned on, the U-10 will be in measurement mode. All parameters are measured at once. Use the Select key to toggle the upper cursor on the readout to the parameter to be read, 4) To get a uniform reading, slowly move the probe up and down to circulate the water through it then wait for the readout to stabilize.
- Expanded Readout methods: Use EXP key to see the results with one additional decimal place of accuracy.
- After measurement: 1) turn off the power, 2) wash the probe thoroughly with tap water. 3) Fill the small rubber cap with tap water and use it to cover the pH sensor, 4) Place vinyl tape around the O-ring portion of the reference sensor to prevent the KCl internal solution.
- The U-10 is to be calibrated daily prior to use: 1) Fill the calibration beaker to about 2/3 with the standard solution, to the line on the beaker. 2) Fit the probe over the beaker noting that the DO sensor is not to be immersed in the standard solution, 3) Turn the power on, 4) press the Mode key until cursor shows MAINT for maintenance mode; the lower cursor should be on the AutoSub-Mode, 5) Press the

ENT key and the upper cursor will gradually move across the four auto calibration parameters one by one. When the auto-calibration has stabilized, the upper cursor will stop blinking and automatically move to MEAS mode. 6) If the unit does not move to MEAS mode and the readout shows *Er3* or *Er4*, press the CLR key to cancel the error code and redo the calibration. See the operation manual for procedures if the unit is to be calibrated manually or if the unit will not calibrate automatically.

- Instruction Manual should accompany the unit at all time to allow troubleshooting using error codes and for instructions regarding replacement of faulty probes.

F. Decontamination

All equipment inserted into a monitoring well will be decontaminated prior to their use. More specifically, the sampling equipment will be decontaminated as follows:

Step 1 Scrub equipment with a non-phosphate detergent mixed with potable water.

Step 2 Rinse with potable water.

Step 3 Rinse twice with distilled water.

Step 4 Air dry.

Step 5 Place in a clean polyethylene bag or wrap in aluminum foil with shiny-side out when not in use and during transport.

Non-dedicated pump will be decontaminated as follows:

Step 1 The outside of the pump will be scrubbed with a non-phosphate detergent mixed with potable water.

Step 2 A volume of non-phosphate detergent mixed with potable water solution equal to at least three times the volume of the discharge tubing will be pumped through the pump.

Step 3 Deionized water will be pumped through the pump until specific conductance readings approach zero.

Disposable sampling equipment and sampling gloves will be discarded after sampling each well. All investigation-derived wastes will be handled as indicated in Section F.4 of the FSP.

ATTACHMENT B-2

Standard Operating Procedures – Laboratory Analyses

TITLE: Solids Sample Preparation for the Analysis of Semivolatile Base/Neutral/Acids-Full Scan

MATRICES:

This SOP pertains to Solid Matrices.

DETECTION LIMITS:

Reporting Limits are used in the absence of Project or State Specific Required Detection Limits. The determinative method contains the current method detection limits (MDL). The Reporting Limits.are found in Appendix B Table A)

The method detection limits were obtained by multiplying 3.143 by the standard deviation of seven spikes. The spikes went through this digestion procedure and contain one gram of clean Ottawa sand, spiked at a concentration were the resulting MDL is suspected to be within a factor of ten less than the spike. Cited from 40CFR part 136 and the WI Department of Natural Resources.

1.0 **SCOPE AND APPLICATION:**

1.1 Accelerated Solvent Extraction (ASE) is a procedure for isolating nonvolatile and semi-volatile organic compounds from solids such as sludges, soils and wastes. The process ensures intimate contact of the sample with the extraction solvent.

2.0 **SUMMARY OF THE TEST METHOD:**

2.1 A measured mass of sample, nominally 20 grams, is dried by mixing with sodium sulfate until it is free flowing. The sample mixture is then transferred into an extraction cell. The cells are then loaded on the carousel of the Accelerated Solvent Extractor. The samples in the cells are then subjected to a pre-programmed heat and pressure extraction cycle which isolates the organic components from the sample mixture by contact with the solvent. The extract is collected and dried with sodium sulfate and then concentrated to a final volume. The concentrated extract is then transferred to crimp top vial and sealed.

3.0 **DEFINITIONS:**

3.1 Terms used throughout this document are defined in Appendix A.

4.0 INTERFERENCES

4.1 Solvents, reagents and glassware can interfere with sample analysis. En Chem purchases high quality solvents and reagents to eliminate this potential of contamination and require vendors to stand behind claims made on the solvents purchased. En Chem has adopted and implemented SOPs addressing proper glassware cleaning techniques, tailored to the department using the apparatus.

4.2 Solvents, reagents, glassware, and other sample processing may yield artifacts and /or interferences to sample analysis. All these materials are demonstrated to be free from interferences under the conditions of the analysis by analyzing method blanks.

4.3 Interferences coextracted from samples will vary considerably from source to source. If analysis of an extracted sample is prevented due to interferences, further clean-up of the sample may be necessary. Phthalate esters contaminate many types of products commonly found in laboratories. Plastics, in particular, must be avoided because phthalates are commonly used as plasticicizers and are easily extracted from plastic materials.

4.4 Because of the concern of interferences affecting the quality of an analysis, all glassware and other materials that come into contact with the sample, directly or indirectly, are first rinsed with the extraction solvent.

5.0 SAFETY:

5.1 Safety is everyone's business at En Chem, Inc. and everyone is responsible for assisting in reducing unsafe and unhealthy working conditions or potential hazards. The company provides a safe place to work, but employee cooperation is needed to keep it safe. When you see something that does not look safe, or you see someone working in an unsafe manner, inform your supervisor.

5.2 The toxicity or carcinogenicity of each reagent used in this method has not been precisely defined. However, each chemical compound should be treated as a potential health hazard. Exposure to these chemicals must be reduced to the lowest possible level by whatever means possible.

5.3 All department personnel shall be familiar with company safety policies as covered in the employee manual, section 5.

5.4 Due to the high volatility of methylene chloride extreme pressure can be generated quickly; use caution when using methylene chloride in closed containers.

5.5 Care should be taken not to use cracked or broken glassware.

5.6 Work with all chemicals and solvents should take place within a hood.

5.7 MSDS sheets are kept by the department supervisor and are located in the sample receiving area on the bookshelf behind the sample log in computer. They should be consulted whenever there is a question related to material handling and safety. Also, *read all chemical labels for special handling requirements, precautions, and warnings.*

5.8 Required safety clothing/equipment to be worn while performing this method can be found in Appendix B Table B.

6.0 **EQUIPMENT AND SUPPLIES:**

6.1 See Appendix B Table C and Table D for a summary.

7.0 **REAGENTS AND STANDARDS:**

7.1 Where the purity of reagents and standards are not specified analytical reagent grade will be used. A grade no less than those specified by the references will not be used.

7.2 Internal standard solutions - The internal standards are listed in Table !:

Table 1

1,4-Dichlorobenzene-$d_{(4)}$,
Naphthalene-$d_{(8)}$
Acenaphlhene-$d_{(10)}$
Chrysene-$d_{(12)}$,
Phenanthrene-$d_{(10)}$
Perylene-$d_{(12)}$

Stock solutions obtained from Restek (Catalog # 31006) have each compound at 4000 ug/ml. Each 1 mL sample extract undergoing analysis should be spiked with 10 uL of this stock standard, resulting in a concentration of 40 ng/uL of each internal standard. Store at 4°C or less when not being used.

7.3 Surrogate Standards - The surrogate standards listed inTable2

Table 2

2-Fluorophenol	Acid Surrogate
Phenol-$d_{(5)}$,	Acid Surrogate
2-Cholorphenol-$d_{(4)}$	Acid Surrogate
1,2 Dichlorobenzene-$d_{(4)}$,	B/N Surrogate
Nitrobenzene-$d_{(5)}$	B/N Surrogate
2-Fluorobiphenyl	B/N Surrogate
2,4,6-Tribromophenol	Acid Surrogate
p-Terphenyl-$d_{(14)}$	B/N Surrogate

The intermediate surrogate solution contains base/neutral and acid based compounds. TheB/N-based surrogate compounds are spiked at an on column concentration of 50 ng, while the acid-based surrogate compounds are spiked on column at a concentration of 75 ng. Soils and waters are spiked with 100 uL at a concentration of 500/750 mg/mL. Surrogate standards must be added to all blanks, spikes, and sample extracts. Take into account all dilutions of sample extracts. These stock standards are obtained from Restek (Catalog #'s 31082 and 31083) at a concentrations of 5000 ppm and 7500 ppm respectively.

7.4 8270 Surrogate Standard, 500/750 ppm intermediate
-1.0 mL of 5000 ppm B/N surrogate + 1.0 mL of 7500 ppm acid surrogate diluted to 10 mL in CH_2Cl_2.

7.5 Matrix spike standards - A sample and a duplicate are spiked with the entire compound list at a on column concentration of 50 ng. 250 ul of a 200 ug/ml stock custom mix standard (Supelco # 20084376), along with 10 ul of a 5000ppm n-nitrosodiphenylamine (Supelco Cat# 46702-U) stock solution are injected into 20.0 grams of soil. The concentration in the soil is at 2500 ug/kg for each compound.

7.6 Blank spike standards - A 20 gram portion of sand and a duplicate are spiked with the entire compound list at a on column concentration of 50 ng. 250 ul of a 200 ug/ml stock custom mix standard (Supelco # 20084376), along with 10 ul of a 5000ppm n-nitrosodiphenylamine (Supelco Cat# 46702-U) stock solution are injected into 20.0 grams of sand. The concentration in the sand is at 2500 ug/kg for each compound.

7.7 See Appendix B Table E and Table F for a summary.

8.0 **SAMPLE COLLECTION, PRESERVATION, SHIPMENT AND STORAGE:**

8.1 Samples are to be collected in glass jars with Teflon lined lids, demonstrated to be free of contamination at or below the reporting limit.

8.2 Samples are to be chilled to 4.0° C immediately after collection or to a specified temperature range governed by accredit ting authorities to preserve integrity. Samples that are hand delivered to the laboratory immediately after collection may be accepted if there is evidence, such as arrival on ice, that the chilling process has begun.

8.3 Shipments to the laboratory must be timely to avoid preparation holding time issues. Shipment to the laboratory may be accomplished by hand delivery, courier services or common carrier during normal business hours (8:00 am – 5:00 pm central time). Arrangements to accept samples outside normal business hours must be made with personnel prior to shipment.

8.4 Upon receipt and after processing requests for analysis, samples are to be refrigerated at 4.0° C until preparation. After preparation the extracts are held at 4.0° C until analysis.

8.5 See Appendix B Table G for a summary.

9.0 **QUALITY CONTROL:**

9.1 All Quality Control measures shall be subjected to exactly the same preparation procedures as those used on actual samples.

9.2 Quality Control measures shall be assessed on an on going basis to determine the usability of the data. The controls measure contamination and statistical measures of accuracy and bias.

9.3 Method blanks shall be performed at a frequency of one per extraction batch, not to exceed 20 environmental samples. This is a negative control used to assess contamination during the preparation process. The investigation of what the potential sources are encompass all the reagents (chemicals and solvents), equipment and supplies, along with the adherence to proper procedures. In most cases this is rarely qualified unless there is no sample left to prepare.

9.4 Laboratory Control Spikes (LCS) and duplicates (LCSD) shall be performed at a frequency of one pair per extraction batch, not to exceed 20 environmental samples. These are positive controls used to assess the manner in which the samples were prepared. This is accomplished by evaluating the statistical measures of accuracy (percent recovery) and bias (relative percent difference). If low recoveries are observed with the instrument in control there is only a slim chance that the samples, a more complex matrix, had adequate sample preparation to produce results truly characterizing the extent or absence of environmental contamination that may exist. If high recoveries are observed with the instrument in control can indicate a potential for off-specification neat standards, poor dilution or spiking technique. That latter casts doubt on the accuracy of sample preparation.

9.5 Matrix Spikes (MS) and duplicates (MSD) shall be performed at a frequency of one pair per extraction batch, not to exceed 20 environmental samples. These are positive controls used to assess extraction performance within the sample composition. This is accomplished by evaluating the statistical measures of accuracy (percent recovery) and bias (relative percent difference). If low recoveries are observed with the instrument and LCS data in control the sample exhibits a matrix effect of low accuracy. If the matrix spike duplicate recoveries are similar to the matrix spike the matrix effect has been confirmed. If high recoveries are observed with the instrument and LCS data in control, the sample may not have been spiked at a high enough concentration to been recovered over the background concentration present in the parent sample. Poor performance of the matrix spike shall be reported to the client whose sample was spiked.

9.6 Surrogate compounds are added to all samples and control measures. These are positive controls. This is done by spiking 100 ul of a 150 ug/ml surrogate solution for a final concentration of 0.75 mg/ kg. The compounds chosen are to mimic the properties of the targeted compounds but are not themselves found in environmental samples. Low recovery can indicate a matrix effect that bias results low. This is confirmed either by re-analysis of the sample or by observing similar recoveries of samples from the same project site. High recovery can indicate the presence of co-eluting compounds with similar properties as the surrogate. Control sample surrogate recovery in control, such as a blank, confirm the ability of both the instrument and staff to accurately spike and recover analytes through sample preparation.

9.7 See Appendix B Table H for a summary.

10.0 CALIBRATION AND STANDARDIZATION:

10.1 The top loading balance calibration is verified on a daily basis, if used. Using Class S weights. The control limits for the 1, 20, and 100 g weights is +/- 1%.

11.0 **PROCEDURE:**

11.1 Verify sample Station IDs and LIMS numbers on containers against LIMS backlog lists.

11.2 Lay out and record sample LIMs numberes as a batch with quality control measures at a frequency as described in Section 9 and summerized in Table H. Headings at the top of the page identify the sampling date, date of extraction, sample mass extracted, final volume of the sample extract, and a comments column concerning special circumstances regarding the sample.

11.3 If present, decant and discard any aqueous layer on sediment or sludge samples. If present, discard and foreign objects, such as sticks, rocks or leaves in soil samples.

11.4 Mix sample thouroughly, especially when compositing samples.

11.5 Using the top loading balance calibrated as described in Section 10 weigh out a mass of sample (or sand for certain quality control measures) as close to 20 grams as possible into a clean 250 mL beaker. Record the sample mass into the extraction logbook to the nearest tenth of a gram. Repeat process for all samples and quality control measures.

NOTE: **Method blank MB, LCS and LCSD are prepared by weighing out 20 grams of organic free ottawa sand in place of soil. and adding appropriate spiking solution to the LCS and LCSD.**

11.6 Add enough sodium sulfate to the sample in the beaker to create a dry free flowing mixture.

11.7 Using a clean powder funnel transfer the mixture from the beaker to an ASE extraction cell. Transer all samples and quality control measeures using the same process. Label each cell with the LIMs number or quality control measure identifier.

NOTE: **Clean extraction cells are prepared to receive the mixture by rinsing the cell and end caps with methylene chloride. An end cap is added to one end of the cell body.**

11.8 Spike each labelled extraction cell with 100 µL of a 500 µg/m L BN / 750 µg/mL A surrogate solution (BNA Surrogate Standard).

NOTE: **After surrogate spiking the extraction cells with the identified LCS, LCSD, MS and MSD are spiked with the appropriate spiking solution to the LCS and LCSD at the amount specified in Section 7.**

11.9 Cap the extraction cell and place them into the AES carousel.

11.10 Load the extract collection tray with labelled pre-cleaned 60 mL glass vials with septa tops corresponding to the labeled extraction cells in the caarousel.

11.11 Verify the ASE extraction settings as summerized in Table 3 below.

Table 3

Oven temperature	100 C
Pressure	1500 psi
Static time	5.0 minutes
Flush volume	0.6 times the cell volume (20.0 ml)
Nitrogen purge Solvent	180 seconds at 150 psi 2:1 Methylene Chloride/ Acetone

11.12 Start the ASE extraction process. The process will produce approximately 40 mL of extract that will be concetrated to a final volume.

NOTE: A rinse cycle is performed between each sample extract to prevent contamination.

11.13 Transfer the extracts from the 60 mL vials to a labeled Kontes tube ebullators.

11.14 Place the tubes into a Kontes tube heater.

11.15 Set the heater dial to 6.5. Place one stainless steel tube connected to the nitrogen regulator into each of the tubes. A high purity nitrogen stream sufficient ot create continuous agitation is used to assist in the concetration process.

11.16 Stop the concentration process when the extract is concetrated to approximately 0.75 mL.

NOTE: Do not over concentrate. If the sample is completely dried, the preparation must start again from the begining.

11.17 The tube and ground glass joints are rinsed with Methylene Chloride.

11.18 Adjust the final volume to 1.0 mL.

11.19 Transfer the extract to a 2.0 mL crimp top vial and seal with a crimp cap.

11.20 Store at appropriate temperature until analysis.

12.0 CALCULATIONS:

12.1 The concentrations determined are to be reported on the basis of the actual weight of the sample. If a dry weight analysis is desired, then the percent solids of the sample must also be provided.

12.2 See EnChem SOP G3-SVO-19 Section 12.0 for analysis calculations.

13.0 METHOD PERFORMANCE:

13.1 The method performance is validated through the Method Detection Limit Study, Initial Validations and the Performance Testing Samples taken through this procedure.

13.2 See Appendix B Table I for a Initial Demonstration of Capability summary.

14.0 **POLLUTION PREVENTION:**

14.1 Pollution prevention encompasses any technique or procedure that reduces or eliminates the quantity or toxicity of waste at the point of generation. Laboratory staff should order where possible acceptable non-toxic alternative supplies and prepare only those quantities of reagents or standards that will be used prior to the expiration date. Other appropriate measures to minimize waste generation should be brought to the attention of laboratory management. All laboratory waste shall be handled as directed by the Laboratory Waste Management Plan and Hazardous Waste Contingency Plan.

15.0 **DATA ASSESSMENT AND ACCEPTANCE CRITERIA FOR QUALITY CONTROL MEASURES:**

15.1 At a minimum there are three levels of data review:

15.2 Analyst/Technician verification at the bench top are looking at instrument performance as it relates to initial calibration, calibration verification, cleanliness.

15.3 Supervisor/Analyst verification after analysis are looking for sample concentration versus linear range of the instrument, typical patterns resulting from the compounds or elements in question, and quality control measure criteria. Data is either accepted without qualification, accepted with qualification, or rejected with samples reprocessed.

15.4 Report Reviewer/Project Management personnel look at the presentation of the data on final reports. The reports are verified for holding time compliance, receipt conditions, method citation and reasonableness, which may include field duplicate analysis, analytical comments and qualifications presented by the previous reviews, and comparing results of similar analytical techniques as it relates to the project site information when available.

15.5 A fourth level of review, data validation is determined from a project or program scope of laboratory services and encompasses the generation Quality Control summaries in the media of "Form Generation."

15.6 See En Chem SOP G3-SVO-19 Appendix B Table K through Table M for a summary of specific items cross checked.

16.0 **CORRECTIVE ACTIONS FOR OUT-OF-CONTROL DATA:**

16.1 Assessment of Quality Control measures is done to provide a level of confidence in the data generated. The measures provide documentation that the instrument conditions were reliable during the analysis. See Table N for corrective actions taken in cases where statistical goals were not achieved.

16.2 See En Chem SOP G3-SVO-19 Appendix B Table N

17.0 CONTINGENCIES FOR HANDLING OUT-OF-CONTROL OR UNACCEPTABLE DATA:

17.1 During analysis, events occur specific to the physical and chemical characteristics of the environmental sample. When possible, with received sample volumes, data generated along with measures that do not meet statistical goals are re-analyzed again to see if the statistical goal can be achieved. When environmental samples do not meet statistical unacceptable data is generated. These events are different from those pertaining to instrument operating conditions. These events occur when the instruments are operating under ideal conditions.

17.2 Contingencies for handling out-of-control or unacceptable data apply to the determinative SOP.

17.3 See En Chem SOP G3-SVO-19 Table O

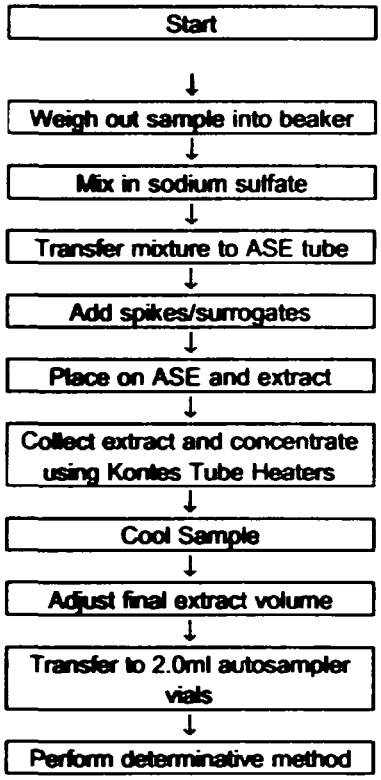
18.0 WASTE MANAGEMENT:

18.1 To minimize waste during sample preparation has two benefits. The first benefit is a cost savings to the lab in materials and supplies. The second is a benefit to the environment as less materials need to be disposed.

REFERENCES:

40CFR Part 136
American Society for Quality Control (ASQC), Definitions of Environmental Quality Assurance Terms, 1996
American National Standards Institute (ANSI), Style Manual for Preparation of Proposed American National Standards, Eighth Edition, March 1991
ANSI/ASQC E4, 1994
ANSI N42.23-1995, Measurement and Associated Instrument Quality Assurance for
National Institute of Standards and Technology (NIST)
National Environmental Laboratory Accreditation Conference (NELAC), July 1998 Standards
Random House College Dictionary
US EPA Quality Assurance Division (QAD)
Webster's New World Dictionary of the American Language
SW846 Method 8000B, December 1996
SW-846 3545 Pressurized Fluid Extraction, Revision 2, December 1996
U.S. EPA 40 CFR Part 136, "Guidelines Establishing Test Procedures for the Analysis of Pollutants Under the Clean Water Act; Final Rule and Interim Final Rule and Proposed Rule," October 26, 1984.
EPA 625

FLOWCHARTS


Start
Weigh out sample into beaker
Mix in sodium sulfate
Transfer mixture to ASE tube
Add spikes/surrogates
Place on ASE and extract
Collect extract and concentrate using Kontes Tube Heaters
Cool Sample
Adjust final extract volume
Transfer to 2.0ml autosampler vials
Perform determinative method

VALIDATION DATA:

The laboratory must demonstrate initial proficiency with each combination of sample preparation and determinative methods utilized, by generating data of acceptable accuracy and precision for a reference sample containing the target parameters in a clean matrix. The laboratory must also repeat this demonstration whenever new staff are trained or significant changes in instrumentation are made. Analysts previously trained shall perform a continued demonstration of proficiency annually by performing "Yearly Demonstration of Capability" (YDC).

The values from the determinative method are presented as guidance only and are not intended as absolute acceptance criteria. En Chem generates data for method performance and are presented along with the performance values in Table I

MANAGEMENT APPROVAL AND REVIEW OF SOPS - POLICY AND DOCUMENTATION

APPROVED BY: Nick J Severin 3/15/01
Section Supervisor Date

M. Suhr 03.15.01
Quality Assurance Officer Date

Nick Melly 3/15/01
Laboratory Manager Date

Periodic Review Record

Review Date					
Initials					

APPENDICES

Appendix A

Acceptance Criteria: specified limits placed on characteristics of an item, process, or service defined in requirement documents. (ASQC)
Accuracy: the degree of agreement between an observed value and an accepted reference value. Accuracy includes a combination of random error (precision) and systematic error (bias) components which are due to sampling and analytical operations; a data quality indicator. (QAMS)
Analytical Detection Limit: the smallest amount of an analyte that can be distinguished in a sample by a given measurement procedure throughout a given (e.g., 0.95) confidence interval. (applicable only to radiochemistry)
Analyst: the designated individual who performs the "hands-on" analytical methods and associated techniques and who is the one responsible for applying required laboratory practices and other pertinent quality controls to meet the required level of quality. (NELAC)
Assessment Criteria: the measures established by NELAC and applied in establishing the extent to which an applicant is in conformance with NELAC requirements. (NELAC)
Data Reduction: the process of transforming raw data by arithmetic or statistical calculations, standard curves, concentration factors, etc., and collation into a more useable form. (EPA-QAD)
Deficiency: an unauthorized deviation from acceptable procedures or practices, or a defect in an item. (ASQC)
Demonstration of Capability: a procedure to establish the ability of the analyst to generate acceptable accuracy. (NELAC)
Detection Limit: the lowest concentration or amount of the target analyte that can be determined to be different from zero by a single measurement at a stated degree of confidence.
Document Control: the act of ensuring that documents (and revisions thereto) are proposed, reviewed for accuracy, approved for release by authorized personnel, distributed properly and controlled to ensure use of the correct version at the location where the prescribed activity is performed. (ASQC)
Duplicate Analyses: the analyses or measurements of the variable of interest performed identically on two sub-samples of the same sample. The results from duplicate analyses are used to evaluate analytical or measurement precision but not the precision of sampling, preservation or storage internal to the laboratory. (EPA-QAD)
Equipment Blank: a sample of analyte-free media which has been used to rinse common sampling equipment to check effectiveness of decontamination procedures. (NELAC)
Matrix Spike Duplicate (spiked sample or fortified sample duplicate): a second replicate matrix spike prepared in the laboratory and analyzed to obtain a measure of the precision of the recovery for each analyte. (QAMS)
May: denotes permitted action, but not required action. (NELAC)
Method Blank: a sample of a matrix similar to the batch of associated samples (when available) that is free from the analytes of interest, which is processed simultaneously with and under the same conditions as samples through all steps of the analytical procedures, and in which no target analytes or interferences are present at concentrations that impact the analytical results for sample analyses. (NELAC)
Method Detection Limit: the minimum concentration of a substance (an analyte) that can be measured and reported with 99% confidence that the analyte concentration is greater than zero and is determined from analysis of a sample in a given matrix containing the analyte. (40 CFR Part 136, Appendix B)
Must: denotes a requirement that must be met. (Random House College Dictionary)
National Institute of Standards and Technology (NIST): an agency of the US Department of Commerce's Technology Administration that is working with EPA, States, NELAC, and other public

and commercial entities to establish a system under which private sector companies and interested States can be accredited by NIST to provide NIST-traceable proficiency testing, (PT), to those laboratories testing drinking water and wastewater. (NIST)

Negative Control: measures taken to ensure that a test, its components, or the environment do not cause undesired effects, or produce incorrect test results. (NELAC)

Quantitation Limits: the maximum or minimum levels, concentrations, or quantities of a target variable (e.g., target analyte) that can be quantified with the confidence level required by the data user. (NELAC)

Range: the difference between the minimum and the maximum of a set of values. (EPA-QAD)

Raw Data: any original factual information from a measurement activity or study recorded in a laboratory notebook, worksheets, records, memoranda, notes, or exact copies thereof that are necessary for the reconstruction and evaluation of the report of the activity or study. Raw data may include photography, microfilm or microfiche copies, computer printouts, magnetic media, including dictated observations, and recorded data from automated instruments. If exact copies of raw data have been prepared (e.g., tapes which have been transcribed verbatim, data and verified accurate by signature), the exact copy or exact transcript may be submitted. (EPA-QAD)

Reagent Blank (method blank): a sample consisting of reagent(s), without the target analyte or sample matrix, introduced into the analytical procedure at the appropriate point and carried through all subsequent steps to determine the contribution of the reagents and of the involved analytical steps. (QAMS)

Record Retention: The systematic collection, indexing and storing of documented information under secure conditions. (EPA-QAD)

Reference Material: a material or substance one or more properties of which are sufficiently well established to be used for the calibration of an apparatus, the assessment of a measurement method, or for assigning values to materials. (ISO Guide 30-2.1)

Reference Method: a method of known and documented accuracy and precision issued by an organization recognized as competent to do so. (NELAC)

Reference Standard: a standard, generally of the highest metrological quality available at a given location, from which measurements made at that location are derived. (VIM-6.08)

Replicate Analyses: the measurements of the variable of interest performed identically on two or more sub-samples of the same sample within a short time interval. (NELAC)

Requirement: denotes a mandatory specification; often designated by the term "shall". (NELAC)

Resource Conservation and Recovery Act (RCRA): the enabling legislation under 42 USC 321 *et seq.* (1976), that gives EPA the authority to control hazardous waste from the "cradle-to-grave", including its generation, transportation, treatment, storage, and disposal. (NELAC)

Sample Duplicate: two samples taken from and representative of the same population and carried through all steps of the sampling and analytical procedures in an identical manner. Duplicate samples are used to assess variance of the total method including sampling and analysis. (EPA-QAD)

Selectivity: (Analytical chemistry) the capability of a test method or instrument to respond to a target substance or constituent in the presence of non-target substances. (EPA-QAD)

Sensitivity: the capability of a method or instrument to discriminate between measurement responses *representing different levels (e.g., concentrations) of a variable of interest.* (NELAC)

Shall: denotes a requirement that is mandatory whenever the criterion for conformance with the specification requires that there be no deviation. This does not prohibit the use of alternative approaches or methods for implementing the specification so long as the requirement is fulfilled. (ANSI)

Should: denotes a guideline or recommendation whenever noncompliance with the specification is permissible. (ANSI)

Spike: a known mass of target analyte added to a blank sample or sub-sample; used to determine recovery efficiency or for other quality control purposes. (NELAC)

Standard: the document describing the elements of laboratory accreditation that has been developed and established within the consensus principles of NELAC and meets the approval requirements of NELAC procedures and policies. (ASQC)

Standard Operating Procedures (SOPs): a written document which details the method of an operation, analysis or action whose techniques and procedures are thoroughly prescribed and which is accepted as the method for performing certain routine or repetitive tasks. (QAMS)

Standardized Reference Material (SRM): a certified reference material produced by the U.S. National Institute of Standards and Technology or other equivalent organization and characterized for absolute content, independent of analytical method. (EPA-QAD)

Supervisor (however named): the individual(s) designated as being responsible for a particular area or category of scientific analysis. This responsibility includes direct day-to-day supervision of technical employees, supply and instrument adequacy and upkeep, quality assurance/quality control duties and ascertaining that technical employees have the required balance of education, training and experience to perform the required analyses. (NELAC)

Surrogate: a substance with properties that mimic the analyte of interest. It is unlikely to be found in environment samples and is added to them for quality control purposes. (QAMS)

Systems Audit (also Technical Systems Audit): a thorough, systematic, qualitative on-site assessment of the facilities, equipment, personnel, training, procedures, record keeping, data validation, data management, and reporting aspects of a total measurement system. (EPA-QAD)

Test: a technical operation that consists of the determination of one or more characteristics or performance of a given product, material, equipment, organism, physical phenomenon, process or service according to a specified procedure. The result of a test is normally recorded in a document sometimes called a test report or a test certificate. (ISO/IEC Guide 2-12.1, amended)

Test Method: an adoption of a scientific technique for a specific measurement problem, as documented in a laboratory SOP. (NELAC)

Tolerance Chart: A chart in which the plotted quality control data is assessed via a tolerance level (e.g. +/- 10% of a mean) based on the precision level judged acceptable to meet overall quality/data use requirements instead of a statistical acceptance criteria (e.g. +/- 3 sigma) (applies to radiobioassay laboratories). (ANSI)

Toxic Substances Control Act (TSCA): the enabling legislation in 15 USC 2601 *et seq.*, (1976), that provides for testing, regulating, and screening all chemicals produced or imported into the United States for possible toxic effects prior to commercial manufacture. (NELAC)

Traceability: the property of a result of a measurement whereby it can be related to appropriate standards, generally international or national standards, through an unbroken chain of comparisons. (VIM-6.12)

United States Environmental Protection Agency (EPA): the federal governmental agency with responsibility for protecting public health and safeguarding and improving the natural environment (i.e., the air, water, and land) upon which human life depends. (US-EPA)

Validation: the process of substantiating specified performance criteria. (EPA-QAD)

Verification: confirmation by examination and provision of evidence that specified requirements have been met. (NELAC) NOTE: In connection with the management of measuring equipment, verification provides a means for checking that the deviations between values indicated by a measuring instrument and corresponding known values of a measured quantity are consistently smaller than the maximum allowable error defined in a standard, regulation or specification peculiar to the management of the measuring equipment. The result of verification leads to a decision either to restore

in service, to perform adjustment, to repair, to downgrade, or to declare obsolete. In all cases, it is required that a written trace of the verification performed shall be kept on the measuring instrument's individual record.

Work Cell: a well-defined group of analysts that together perform the method analysis. The members of the group and their specific functions within the work cell must be fully documented. (NELAC)

Appendix B

Tables

Table A	**Reporting Limits**
Table B	**Required Safety Equipment**
Table C	**Equipment**
Table D	**Supplies**
Table E	**Reagents**
Table F	**Standards**
Table G	**Sample Collection**
Table H	**Quality Control**
Table I	**Initial Demonstration of Capability**

Table A

REPORTING LIMITS

SEMIVOLATILE TARGET COMPOUND LIST SOIL - GC/MS

Compound	Reporting Limit	Compound	Reporting Limit
Phenol	250	Acenaphthene	250
bis(2-Chloroethyl)ether	250	2,4-Dinitrophenol	500
2-Chlorophenol	250	Dibenzofuran	250
1,3-Dichlorobenzene	250	4-Nitrophenol	500
1,4-Dichlorobenzene	250	2,4-Dinitrotoluene	250
1,2-Dichlorobenzene	250	Fluorene	250
2-Methylphenol	250	Diethylphthalate	250
2,2-oxybis(1-Chloropropene)	250	4-Chlorophenyl phenyl ether	250
N-Nitroso-di-n-propylamide	250	4-Nitroaniline	500
4-Methylphenol	250	4,6-Dinitro-2-methylphalate	250
Hexachloroethane	250	N-Nitrosodiphenylamine	250
Nitrobenzene	250	4-Bromophenyl phenyl ether	250
Isophorone	250	Hexachlorobenzene	250
2-Nitrophenol	250	Pentachlorophenol	500
2,4-Dimethylphenol	500	Phenanthrene	250
bis(2-Chloroethoxy)methane	250	Anthracene	250
2,4-Dichlorophenol	250	Carbazole	250
1,2,4-Trichlorobenzene	250	di-n-Butylphthalate	250
Naphthalene	250	Fluoranthene	250
4-Chloroaniline	500	Pyrene	250
Hexachlorobutadiene	250	Butylbenzylphthalate	250
4-Chloro-3-methylphenol	250	Benzo(a)anthracene	250
2-Methylnaphthalene	250	3,3'-Dichlorobenzidine	250
Hexachlorocyclopentadiene	250	Chrysene	250
2,4,6-Trichlorophenol	250	bis(2-Ethylhexyl)phthalate	250
2,4,5-Trichlorophenol	250	di-n-Octylphthalate	500
2-Chloronaphthalene	250	Benzo(b)fluoranthene	250
2-Nitroaniline	500	Benzo(k)fluoranthene	250
Dimethylphthalate	250	Benzo(a)pyrene	250
Acenaphthylene	250	Indeno(1,2,3-cd)pyrene	250
2,6-Dinitrotoluene	250	Dibenzo(a,h)anthracene	250
3-Nitroaniline	500	Benzo(g,h,i)perylene	250

Units = ug/kg

Table B

REQUIRED SAFETY EQUIPMENT

Item:	Safety Apparel Description	Mandatory for Procedure	Optional
1	Safety Goggles or Glasses	X	
2	Lab Coat	X	
3	Gloves	X	
4	Face Shield		X

Table C

EQUIPMENT

Equipment	Manufacturer	Model(s)	Serial #	Date In Service
Accelerated Solvent Extractor #1	Dionex	200	96030214	May 1996
Accelerated Solvent Extractor #2	Dionex	200	98110378	May 2000
Blue M Oven	Lindberg	51794	927000	March 1992
Scientific Products Oven	Scientific Products	N8620-10A	01920328	March 1995
Kontes Tube Heater Blocks #1	Kontes		1776	March 1995
Kontes Tube Heater Blocks #2	Kontes		1860	March 1995
Kontes Tube Heater Blocks #3	Kontes		2023	March 1998
Kontes Tube Ebullator	Kontes	569401	——	——

Table D

SUPPLIES

Supplies	Manufacturer	Vendor	Catalog #
2 mL autosampler vials	VWR	VWR	66020-953
Aluminum crimp seals	VWR	VWR	66010-847
Rubber pipette bulb	NJ Rubber	VWR	R5002-2
N-Dex Nitrile Glove Powder free (medium)	Best	Fisher Scientific	6005 PFM
N-Dex Nitrile Glove Powder free (large)	Best	Fisher Scientific	7005L
N-Dex Nitrile Glove (extra large)	Best	Fisher Scientific	7005XL
Labtone glassware detergent	VWR	VWR	21851-006
Tube and bottle brush	VWR	VWR	PK-5-17041-002
5.75" disposable pipets	Fisher Scientific	Fisher Scientific	13-678-20-A
Disposable culture tubes	VWR	VWR	60825-618
25 mL adjustable repipetor	VWR	VWR	40000-066
Lab notebooks	VWR	VWR	51280-108
600 mL beakers	Kimble	Fisher Scientific	13910-145
250 mL beakers	Kimble	Fisher Scientific	14005-250
150 mL beakers	Kimble	Fisher Scientific	02539J
60 degree powder funnel	Pyrex	VWR	30240-047
2.0 mL class A Volumetric Flask	Pyrex	Fisher Scientific	20-814-B
5.0 mL class A Volumetric Flask	Pyrex	Fisher Scientific	29623-300
25.0 mL class A Volumetric Flask	Pyrex	Fisher Scientific	10-210-A
50.0 mL class A Volumetric Flask	Pyrex	Fisher Scientific	10-210-B
9 oz Amber jars	QEC	QEC	2124-0009
33 ml stainless steel cells	Dionex	Dionex	049562
Cellulose filters	Dionex	Dionex	049458
60 mL collection vials	I-Chem	VWR	405531
Kontes Concentrator Tubes	Kontes	VWR	570051-2526
Micro distilling columns	Kontes	VWR	569261-0324
Stainless Steel Spatula	VWR	VWR	
25 uL gastight syringe	Hamilton	Fisher Scientific	24559
50 uL gastight syringe	Hamilton	Fisher Scientific	111111
100 uL gastight syringe	Hamilton	Fisher Scientific	13-684-100
250 uL gastight syringe	Hamilton	Fisher Scientific	81100
500 uL gastight syringe	Hamilton	Fisher Scientific	81217
1000 uL gastight syringe	Hamilton	Fisher Scientific	1482425
Pyrex baking dish	Corning	—	6001078

Table E

REAGENTS

Reagent	Purity	Manufacturer	Vendor	Catalog #
Methylene Chloride	Pesticide Quality	Burdick & Jackson	Fisher Scientific	CS300-200
Acetone	Pesticide Quality	Burdick & Jackson	Fisher Scientific	010-4
Di-ionized Water	Type I ASTM	—	—	—
Ottawa Sand	—	Fisher Scientific	Fisher Scientific	S23-3
Sodium sulfate (anhydrous; 10-60 mesh)	ASC grade	Fisher Scientific	Fisher Scientific	MK802406
Nitrogen	99.99%	Whatman	—	—

Table F

STANDARDS

Standard	Acronym	Concentration	Direction found in Section:	Alias
Equity 8270 GC/MS Tuning Solution II	Tune Stock	1000 ug/ml	Stock Standard	Tune Stock
Custom LCS Spike Mix	Spk Mix	200 ug/ml	Stock Standard	Spk Mix
Equity n-Nitrosodiphenylamine	n-NDPA	5000 ug/ml	Stock Standard	n-NDPA
EPA CLP SOW OLMO4 Semivolatile Mix	Spk Mix	2000 ug/ml	Stock Standard	Spk Mix
Custom Mix	Cal Mix	400 ug/ml	Stock Standard	Calibration mix
B/N Surrogate	B/N Surr	5000 ug/ml	Stock Standard	B/N Surr
Acid Surrogate	Acid Surr	7500 ug/ml	Stock Standard	Acid Surr
SV Internal Standard Mix	INT STD	4000 ug/ml	Stock Standard	INT STD
8270 Tune/Degrad check standard	DFTPP	50 ug/ml	Reagents & Standards	DFTPP/Degrad
8270 Surrogate	Surrogate spike	500/750 ug/ml	Reagents & Standards	Surrogate spike
Continuing Calibration check standard	CCV	50 ug/ml	Reagents & Standards	Continuing Cal
Initial Calibration check standard	ICV	50 ug/ml	Reagents & Standards	Initial Cal
8270 Calibration Spike Mix		200 ug/ml	Reagents & Standards	Mix #1
8270 Spike Mix		200 ug/ml	Reagents & Standards	Mix #2

Table G

SAMPLE COLLECTION, PRESERVATION, SHIPMENT AND STORAGE

Matrix	Prep Method	Container(s)	Preservation	Shipment Conditions	Lab Storage Conditions
SOIL	3545	Amber glass jars with teflon-lined caps	none	On ice 4+/- 2° Celsius	Refrigerate 4+/- 2° Celsius

Table H

QUALITY CONTROL

Preparation Method ⇨ Quality Control Measure ⇩	**SW846 8270C**	**EPA 625**
Initial Calibration	Minimum of five levels; lowest level near but above MDL.	Minimum of five levels; lowest level near but above MDL.
Method Blank	One per batch of samples, up to 20 enviromental samples, whichever is more frequent.	One per batch of samples, up to 20 enviromental samples, whichever is more frequent.
DFTPP Tune Check	One at the beginning of a 12 hour time clock or more frequent.	One at the beginning of a 12 hour time clock or more frequent.
Calibration Verification Standard (CVS)	One at the beginning of a 12 hour time clock or more frequent.	One at the beginning of a 12 hour time clock or more frequent.
Independent Calibration Verification	Indepenent source run immediately after the calibration curve.	Indepenent source run immediately after the calibration curve.
Laboratory Control Spike and Duplicate	One pair per batch of samples, up to 20 environmental samples, whichever is more frequent.	One pair per batch of samples, up to 20 environmental samples, whichever is more frequent.
Matrix Spike and Duplicate	One pair per batch of samples, up to 20 environmental samples, whichever is more frequent.	One pair per batch of samples, up to 20 environmental samples, whichever is more frequent.
Internal Standards	Added to every sample.	Added to every sample.
Method Validation	Annually	Annually
MDL	Annually	Annually
Surrogate Standards	Added to every sample.	Added to every sample.

Table I

En Chem Initial Demonstration of Capability

Compound	Recovery Range EPA 625	En Chem Inital Demonstration Recovery
Acenaphthene	47-145	88%
Acenaphthylene	33-145	90%
Anthracene	27-133	93%
Benz(a)anthracene	33-143	97%
Benzo(b)fluoranthene	24-159	98%
Benzo(k)fluoranthene	11-162	102%
Benzo(a)pyrene	17-163	97%
Benzo(g,h,i)perylene	D-219	96%
Benzyl butyl phthalate	D-152	101%
Bis(2-chloroethyl) ether	12-158	84%
Bis(2-chloroethoxy)methane	33-184	86%
Bis(2-chloroisopropyl) ether	36-166	85%
Bis(2-ethylhexyl) phthalate	8-158	103%
4-Bromophenyl phenyl ether	53-127	91%
2-Chloronaphthalene	60-118	87%
4-Chlorophenyl phenyl ether	25-158	92%
Chrysene	17-168	96%
Dibenzo(a,h)anthracene	D-227	93%
Di-n-butyl phthalate	1-118	100%
1,2-Dichlorobenzene	32-129	85%
1,3-Dichlorobenzene	D-172	82%
1,4-Dichlorobenzene	20-124	84%
3,3'-Dichlorobenzidine	D-262	113%
Diethyl phthalate	D-114	95%
Dimethyl Phthalate	D-112	93%
2,4-Dinitrotoluene	39-139	94%
2,6-Dinitrotoluene	50-158	91%
Di-n-octyl phthalate	4-146	101%
Fluoranthene	26-137	99%
Fluorene	59-121	93%
Hexachlorobenzene	D-152	91%
Hexachlorobutadiene	24-116	83%
Hexachloroethane	40-113	84%

Compound	Recovery Range EPA 625	En Chem Inital Demonstration Recovery
Indeno(1,2,3-cd)Pyrene	D-171	95%
Isophorone	21-196	86%
Naphthalene	21-133	86%
Nitrobenzene	35-180	82%
N-Nitroso-Di-n-Propylamine	D-230	86%
Phenanthrene	54-120	93%
Pyrene	52-115	100%
1,2,4-Trichlorobenzene	44-142	83%
4-Chloro-3-Methylphenol	22-147	87%
2-Chlorophenol	23-134	80%
2,4-Dichlorophenol	39-135	85%
2,4-Dimethylphenol	32-119	86%
2,4-Dinitrophenol	D-191	89%
4,6-Dinitro-2-Methylphenol	D-181	96%
2-Nitrophenol	29-182	85%
4-Nitrophenol	D-132	74%
Pentachlorophenol	14-176	98%
Phenol	5-112	34%
2,4,6-Trichlorophenol	37-144	89%
Additional Compounds		
2,4,5-Trichlorophenol		87%
2-Methylnaphthalene		88%
2-Methylphenol		74%
2-Nitroaniline		88%
3-Nitroaniline		102%
4-Chloroaniline		86%
4-Methylphenol		69%
4-Nitroaniline		126%
Benzo(a)Anthracene		97%
Benzoic Acid		36%
Benzyl Alcohol		77%
Carbazole		113%
Dibenz(a,h)Anthracene		93%
Hexachlorocyclopentadiene		88%
N-Nitrosodimethylamine		53%
N-Nitrosodiphenylamine		91%
Pyridine		25%
Aniline		51%
Benzaldehyde		103%

Compound	**Recovery Range EPA 625**	**En Chem Inital Demonstration Recovery**
Additional Compounds		
Acetophenone		84%
Caprolactum		27%
Azobenzene		92%
Atrazine		103%
Biphenyl		90%
1-Methylnaphthalene		84%
Dibenzofuran		91%

Standard Operating Procedure

TITLE: Soil and sediment Preparation for the Analysis of Polychlorinated Biphenyls by GC ECD

MATRICES

This SOP pertains to Solids Matrices

DETECTION LIMITS

Reporting Limits are used in the absence of Project or State Specific Required Detection Limits. The determinative method contains the current method detection limits (MDL). The Reporting Limits.are found in Appendix B Table A.

The method detection limits were obtained by multiplying 3.143 by the standard deviation of seven spikes. The spikes went through this digestion procedure and contain one gram of clean Ottawa sand, spiked at a concentration were the resulting MDL is suspected to be within a factor of ten less than the spike. Cited from 40CFR part 136 and the WI Department of Natural Resources.

1.0 SCOPE AND APPLICATION:

1.1 Accelerated Solvent Extraction (ASE) is a procedure for isolating nonvolatile and semi-volatile organic compounds from solids such as sludges, soils and wastes. The process ensures intimate contact of the sample with the extraction solvent.

2.0 SUMMARY OF THE TEST METHOD:

2.1 A measured mass of sample, nominally 20 grams, is dried by mixing with sodium sulfate until it is free flowing. The sample mixture is then transferred into an extraction cell. The cells are then loaded on the carousel of the Accelerated Solvent Extractor. The samples in the cells are then subjected to a pre-programmed heat and pressure extraction cycle which isolates the organic components from the sample mixture by contact with the solvent. The extract is collected and dried with sodium sulfate and then concentrated to a final volume. The concentrated extract is then transferred to crimp top vial and sealed.

3.0 DEFINITIONS

3.1 Terms used throughout this document are defined in Appendix A.

4.0 INTERFERENCES

4.1 Solvents, reagents and glassware can interfere with sample analysis. En Chem purchases high quality solvents and reagents to eliminate this potential of contamination and require vendors to stand behind claims made on the solvents purchased. En Chem has adopted and implemented SOPs addressing proper glassware cleaning techniques, tailored to the department using the apparatus.

4.2 Solvents, reagents, glassware, and other sample processing may yield artifacts and /or interferences to sample analysis. All these materials are demonstrated to be free from interferences under the conditions of the analysis by analyzing method blanks.

4.3 Interferences coextracted from samples will vary considerably from source to source. If analysis of an extracted sample is prevented due to interferences, further clean-up of the sample may be necessary. Phthalate esters contaminate many types of products commonly found in laboratories. Plastics, in particular, must be avoided because phthalates are commonly used as plasticicizers and are easily extracted from plastic materials.

4.4 Because of the concern of interferences affecting the quality of an analysis, all glassware and other materials that come into contact with the sample, directly or indirectly, are first rinsed with the extraction solvent.

5.0 SAFETY:

5.1 Safety is everyone's business at En Chem, Inc. and everyone is responsible for assisting in reducing unsafe and unhealthy working conditions or potential hazards. The company provides a safe place to work, but employee cooperation is needed to keep it safe. When you see something that does not look safe, or you see someone working in an unsafe manner, inform your supervisor.

5.2 The toxicity or carcinogenicity of each reagent used in this method has not been precisely defined. However, each chemical compound should be treated as a potential health hazard. Exposure to these chemicals must be reduced to the lowest possible level by whatever means possible.

5.3 All department personnel shall be familiar with company safety policies as covered in the employee manual, section 5.

5.4 Due to the high volatility of methylene chloride extreme pressure can be generated quickly; use caution when using methylene chloride in closed containers.

5.5 Care should be taken not to use cracked or broken glassware.

5.6 Work with all chemicals and solvents should take place within a hood.

5.7 MSDS sheets are kept by the department supervisor and are located in the sample receiving area on the bookshelf behind the sample log in computer. They should be consulted whenever there is a question related to material handling and safety. Also, read all chemical labels for special handling requirements, precautions, and warnings.

5.8 Required safety clothing/equipment to be worn while performing this method can be found in Appendix B Table B.

6.0 **EQUIPMENT AND SUPPLIES:**

6.1 See Appendix B Table C and Table D for a summary.

7.0 **REAGENTS AND STANDARDS:**

7.1 Where the purity of reagents and standards are not specified analytical reagent grade will be used. A grade no less than those specified by the references will not be used.

7.2 Laboraotry Control and Matrix Spiking Solution:

Dilute 50 µL of the stock standard (Restek aroclor 1016/1260 mix catalog number 32012 at 1000 µg/ml) to 5.0 mL with methylene chloride to prepare spike intermediate #1. Spike 500 µl of spike intermediate #1 is added to LCS, LCSD, MS and MSD.

Note: This solution is stored in a refrigerator at 4° C.

7.3 Surrogate Spiking Solution:

Dilute 250 µL of the stock standard surrogate (Restek pesticide surrogate mix catalog number 3200 at 200 µg/ml), to 10 mL in a volumetric flask with methylene chloride to prepare surrogate intermediate #1. This solution is stored in a refrigerator at 4 C until needed. Spike 100 uL of surrogate intermediate #1 into all QC and samples.

Note: This solution is stored in a refrigerator at 4° C.

7.4 See Appendix B Table E and Table F for a summary.

8.0 **SAMPLE COLLECTION, PRESERVATION, SHIPMENT AND STORAGE:**

8.1 Samples are to be collected in amber glass jars with Teflon lined lids, demonstrated to be free of contamination at or below the reporting limit.

8.2 Samples are to be chilled to 4.0^0 C immediately after collection or to a specified temperature range governed by accredit ting authorities to preserve integrity. Samples that are hand delivered to the laboratory immediately after collection may be accepted if there is evidence, such as arrival on ice, that the chilling process has begun.

8.3 Shipments to the laboratory must be timely to avoid preparation holding time issues. Shipment to the laboratory may be accomplished by hand delivery, courier services or common carrier during normal business hours (8:00 am – 5:00 pm central time). Arrangements to accept samples outside normal business hours must be made with personnel prior to shipment.

8.4 Upon receipt and after processing requests for analysis, samples are to be refrigerated at 4.0^0 C until preparation. After preparation the extracts are held at 4.0^0 C until analysis.

8.5 See Appendix B Table G for a summary.

9.0 **QUALITY CONTROL:**

9.1 All Quality Control measures shall be subjected to exactly the same preparation procedures as those used on actual samples.

9.2 Quality Control measures shall be assessed on an on going basis to determine the usability of the data. The controls measure contamination and statistical measures of accuracy and bias.

9.3 Method blanks shall be performed at a frequency of one per extraction batch, not to exceed 20 environmental samples. This is a negative control used to assess contamination during the preparation process. The investigation of what the potential sources are encompass all the reagents (chemicals and solvents), equipment and supplies, along with the adherence to proper procedures. In most cases this is rarely qualified unless there is no sample left to prepare.

9.4 Laboratory Control Spikes (LCS) and duplicates (LCSD) shall be performed at a frequency of one pair per extraction batch, not to exceed 20 environmental samples. These are positive controls used to assess the manner in which the samples were prepared. This is accomplished by evaluating the statistical measures of accuracy (percent recovery) and bias (relative percent difference). If low recoveries are observed with the instrument in control there is only a slim chance that the samples, a more complex matrix, had adequate sample preparation to produce results truly characterizing the extent or absence of environmental contamination that may exist. If high recoveries are observed with the instrument in control can indicate a potential for off-specification neat standards, poor dilution or spiking technique. That latter casts doubt on the accuracy of sample preparation.

9.5 Matrix Spikes (MS) and duplicates (MSD) shall be performed at a frequency of one pair per extraction batch, not to exceed 20 environmental samples. These are positive controls used to assess extraction performance within the sample composition. This is accomplished by evaluating the statistical measures of accuracy (percent recovery) and bias (relative percent difference). If low recoveries are observed with the instrument and LCS data in control the sample exhibits a matrix effect of low accuracy. If the matrix spike duplicate recoveries are similar to the matrix spike the matrix effect has been confirmed. If high recoveries are observed with the instrument and LCS data in control, the sample may not have been spiked at a high enough concentration to been recovered over the background concentration present in the parent sample. Poor performance of the matrix spike shall be reported to the client whose sample was spiked.

9.6 Surrogate compounds are added to all samples and control measures. These are positive controls. This is done by spiking 100 ul of a 150 ug/ml surrogate solution for a final concentration of 0.75 mg/ kg. The compounds chosen are to mimic the properties of the targeted compounds but are not themselves found in environmental samples. Low recovery can indicate a matrix effect that bias results low. This is confirmed either by re-analysis of the sample or by observing similar recoveries of samples from the same project site. High recovery can indicate the presence of co-eluting compounds with similar properties as the surrogate. Control sample surrogate recovery in control, such as a blank, confirm the ability of both the instrument and staff to accurately spike and recover analytes through sample preparation.

9.7 See Appendix B Table H for a summary.

10.0 **CALIBRATION AND STANDARDIZATION**:

10.1 The top loading balance calibration is verified on a daily basis, if used. Using Class S weights. The control limits for the 1, 20, and 100 g weights is +/- 1%.

11.0 PROCEDURE:

11.1 Verify sample Station IDs and LIMS numbers on containers against LIMS backlog lists.

11.2 Lay out and record sample LIMs numberes as a batch with quality control measures at a frequency as described in Section 9 and summerized in Table H. Headings at the top of the page identify the sampling date, date of extraction, sample mass extracted, final volume of the sample extract, and a comments column concerning special circumstances regarding the sample.

11.3 If present, decant and discard any aqueous layer on sediment or sludge samples. If present, discard and foreign objects, such as sticks, rocks or leaves in soil samples.

11.4 Mix sample thouroughly, especially when compositing samples.

11.5 Using the top loading balance calibrated as described in Section 10 weigh out a mass of sample (or sand for certain quality control measures) as close to 20 grams as possible into a clean 250 mL beaker. Record the sample mass into the extraction logbook to the nearest tenth of a gram. Repeat process for all samples and quality control measures.

NOTE: Method blank MB, LCS and LCSD are prepared by weighing out 20 grams of organic free ottawa sand in place of soil. and adding appropriate spiking solution to the LCS and LCSD.

11.6 Add enough sodium sulfate to the sample in the beaker to create a dry free flowing mixture.

11.7 Using a clean powder funnel transfer the mixture from the beaker to an ASE extraction cell. Transer all samples and quality control measeures using the same process. Label each cell with the LIMs number or quality control measure identifier.

NOTE: Clean extraction cells are prepared to receive the mixture by rinsing the cell and end caps with methylene chloride. An end cap is added to one end of the cell body.

11.8 Spike each labelled extraction cell with 100 µL of a 50 µg/mL surrogate solution (surrogate intermediate #1).

NOTE: After surrogate spiking the extraction cells with the identified LCS, LCSD, MS and MSD are spiked with the appropriate spiking solution to the LCS and LCSD at the amount specified in Section 7.

11.9 Cap the extraction cell and place them into the AES carousel.

11.10 Load the extract collection tray with labelled pre-cleaned 60 mL glass vials with septa tops corresponding to the labeled extraction cells in the caarousel.

11.11 Verify the ASE extraction settings as summerized in Table 3 below.

Table 3

Oven temperature	100 C
Pressure	1500 psi
Static time	5.0 minutes
Flush volume	0.6 times the cell volume (20.0 ml)
Nitrogen purge Solvent	180 seconds at 150 psi 2:1 Methylene Chloride/ Acetone

11.12 Start the ASE extraction process. The process will produce approximately 40 mL of extract that will be concetrated to a final volume.

NOTE: A rinse cycle is performed between each sample extract to prevent contamination.

11.13 Transfer the extracts from the 60 mL vials to a labeled Kontes tube ebullators.

11.14 Place the tubes into a Kontes tube heater.

11.15 Set the heater dial to 6.5. Place one stainless steel tube connected to the nitrogen regulator into each of the tubes. A high purity nitrogen stream sufficient ot create continuous agitation is used to assist in the concetration process.

11.16 Stop the concentration process when the extract is concetrated to approximately 0.75 mL.

NOTE: Do not over concentrate. If the sample is completely dried, the preparation must start again from the begining.

11.17 The tube and ground glass joints are rinsed with Methylene Chloride.

11.18 Add approximately 8.0 mL of Hexane to the tube and concentrate again to 0.75 mL. Add a second aliquot of Hexane and again concentrate to 0.75 mL. Through this process a solvent exchange to Hexane is accomplished due to the higher volatility of the Acetone/Methylene Chloride mixture.

11.19 After the second Hexane concentration the extract is brought to a final volume of 10.0 mL.

11.20 Transfer the extract to labeled 12.0 mL amber screw top vials and seal with a teflon lined caps.

NOTE: Samples that require additional clean up are processed through a Sulfuric Acid Clean Up and/or a Mercury Clean Up as described in SOPs G3-SVOA-17 and G3-SVO-22 respectively.

11.21 Store extracts at an appropriate temperature until analysis.

12.0 CALCULATIONS:

12.1 The concentrations determined are to be reported on the basis of the actual weight of the sample. If a dry weight analysis is desired, then the percent solids of the sample must also be provided.

12.2 See EnChem SOP G3-SVOA-18 Section 12.0 for analysis calculations.

13.0 METHOD PERFORMANCE:

13.1 The method performance is validated through the Method Detection Limit Study, Initial Validations and the Performance Testing Samples taken through this procedure.

13.2 See Appendix B Table I for a Initial Demonstration of Capability summary.

14.0 POLLUTION PREVENTION:

14.1 Pollution prevention encompasses any technique or procedure that reduces or eliminates the quantity or toxicity of waste at the point of generation. Laboratory staff should order where possible acceptable non-toxic alternative supplies and prepare only those quantities of reagents or standards that will be used prior to the expiration date. Other appropriate measures to minimize waste generation should be brought to the attention of laboratory management. All laboratory waste shall be handled as directed by the Laboratory Waste Management Plan and Hazardous Waste Contingency Plan.

15.0 DATA ASSESSMENT AND ACCEPTANCE CRITERIA FOR QUALITY CONTROL MEASURES:

15.1 At a minimum there are three levels of data review:

15.2 Analyst/Technician verification at the bench top are looking at instrument performance as it relates to initial calibration, calibration verification, cleanliness.

15.3 Supervisor/Analyst verification after analysis are looking for sample concentration versus linear range of the instrument, typical patterns resulting from the compounds or elements in question, and quality control measure criteria. Data is either accepted without qualification, accepted with qualification, or rejected with samples reprocessed.

15.4 Report Reviewer/Project Management personnel look at the presentation of the data on final reports. The reports are verified for holding time compliance, receipt conditions, method citation and reasonableness, which may include field duplicate analysis, analytical comments and qualifications presented by the previous reviews, and comparing results of similar analytical techniques as it relates to the project site information when available.

15.5 A fourth level of review , data validation is determined from a project or program scope of laboratory services and encompasses the generation Quality Control summaries in the media of "Form Generation."

15.6 See En Chem SOP G3-SVOA-18 Appendix B Table K through Table M for a summary of specific items cross checked.

16.0 CORRECTIVE ACTIONS FOR OUT-OF-CONTROL DATA:

16.1 *Assessment of Quality Control measures is done to provide a level of confidence in the* data generated. The measures provide documentation that the instrument conditions were reliable during the analysis. See Table N for corrective actions taken in cases where statistical goals were not achieved.

16.2 See En Chem SOP G3-SVOA-18 Appendix B Table N

17.0 CONTINGENCIES FOR HANDLING OUT-OF-CONTROL OR UNACCEPTABLE DATA:

17.1 During analysis, events occur specific to the physical and chemical characteristics of the environmental sample. When possible, with received sample volumes, data generated along with measures that do not meet statistical goals are re-analyzed again to see if the statistical goal can be achieved. When environmental samples do not meet statistical unacceptable data is generated. These events are different from those pertaining to instrument operating conditions. These events occur when the instruments are operating under ideal conditions.

17.2 Contingencies for handling out-of-control or unacceptable data apply to the determinative SOP.

17.3 See En Chem SOP G3-SVOA-18 Table O

18.0 WASTE MANAGEMENT:

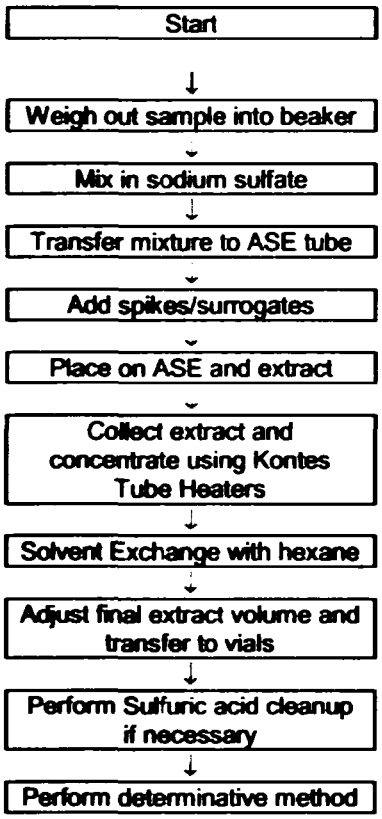
18.1 To minimize waste during sample preparation has two benefits. The first benefit is a cost savings to the lab in materials and supplies. The second is a benefit to the environment as less materials need to be disposed.

18.2 During the extraction of samples Methylene Chloride is used. In accordance with waste minimization Methylene Chloride is purchased in 55 gallon quantities reducing the amount of containers supplied to the lab. The 55 gallon container is equipped with a product delivery system with valves that eliminate open containers of product evaporating. When the container is empty, it is simply returned to the vendor to be recycled. The methylene Chloride used in the extraction of samples is re-captured and bulk containerized for recycling by the waste removal service.

REFERENCES:

40CFR Part 136
American Society for Quality Control (ASQC), Definitions of Environmental Quality Assurance Terms, 1996
American National Standards Institute (ANSI), Style Manual for Preparation of Proposed American National Standards, Eighth Edition, March 1991
ANSI/ASQC E4, 1994
ANSI N42.23-1995, Measurement and Associated Instrument Quality Assurance for
National Institute of Standards and Technology (NIST)
National Environmental Laboratory Accreditation Conference (NELAC), July 1998 Standards
Random House College Dictionary
US EPA Quality Assurance Division (QAD)
Webster's New World Dictionary of the American Language
SW846 Method 3545
USEPA 608

FLOWCHARTS


Start
Weigh out sample into beaker
Mix in sodium sulfate
Transfer mixture to ASE tube
Add spikes/surrogates
Place on ASE and extract
Collect extract and concentrate using Kontes Tube Heaters
Solvent Exchange with hexane
Adjust final extract volume and transfer to vials
Perform Sulfuric acid cleanup if necessary
Perform determinative method

VALIDATION DATA:

The laboratory must demonstrate initial proficiency with each combination of sample preparation and determinative methods utilized, by generating data of acceptable accuracy and precision for a reference sample containing the target parameters in a clean matrix. The laboratory must also repeat this demonstration whenever new staff are trained or significant changes in instrumentation are made. Analysts previously trained shall perform a continued demonstration of proficiency annually by performing "Yearly Demonstration of Capability" (YDC).

The values from the determinative method are presented as guidance only and are not intended as absolute acceptance criteria. En Chem generates data for method performance and are presented along with the performance values in Table I.

MANAGEMENT APPROVAL AND REVIEW OF SOPS - POLICY AND DOCUMENTATION

APPROVED BY: _____ 03/15/01
Section Supervisor — Date

M. Sutha — 03.15.01
Quality Assurance Officer — Date

_____ 3/15/01
Laboratory Manager — Date

Periodic Review Record

Review Date	5/24/02				
initials	MCA / ACC				

APPENDICES

Appendix A

Acceptance Criteria: specified limits placed on characteristics of an item, process, or service defined in requirement documents. (ASQC)
Accuracy: the degree of agreement between an observed value and an accepted reference value. Accuracy includes a combination of random error (precision) and systematic error (bias) components which are due to sampling and analytical operations; a data quality indicator. (QAMS)
Analytical Detection Limit: the smallest amount of an analyte that can be distinguished in a sample by a given measurement procedure throughout a given (e.g., 0.95) confidence interval. (applicable only to radiochemistry)
Analyst: the designated individual who performs the "hands-on" analytical methods and associated techniques and who is the one responsible for applying required laboratory practices and other pertinent quality controls to meet the required level of quality. (NELAC)
Assessment Criteria: the measures established by NELAC and applied in establishing the extent to which an applicant is in conformance with NELAC requirements. (NELAC)
Data Reduction: the process of transforming raw data by arithmetic or statistical calculations, standard curves, concentration factors, etc., and collation into a more useable form. (EPA-QAD)
Deficiency: an unauthorized deviation from acceptable procedures or practices, or a defect in an item. (ASQC)
Demonstration of Capability: a procedure to establish the ability of the analyst to generate acceptable accuracy. (NELAC)
Detection Limit: the lowest concentration or amount of the target analyte that can be determined to be different from zero by a single measurement at a stated degree of confidence.
Document Control: the act of ensuring that documents (and revisions thereto) are proposed, reviewed for accuracy, approved for release by authorized personnel, distributed properly and controlled to ensure use of the correct version at the location where the prescribed activity is performed. (ASQC)
Duplicate Analyses: the analyses or measurements of the variable of interest performed identically on two sub-samples of the same sample. The results from duplicate analyses are used to evaluate analytical or measurement precision but not the precision of sampling, preservation or storage internal to the laboratory. (EPA-QAD)
Equipment Blank: a sample of analyte-free media which has been used to rinse common sampling equipment to check effectiveness of decontamination procedures. (NELAC)
estimate of Target analyte concentration is available. Matrix spikes are used, for example, to determine the effect of the matrix on a method's recovery efficiency. (QAMS)
Matrix Spike Duplicate (spiked sample or fortified sample duplicate): a second replicate matrix spike prepared in the laboratory and analyzed to obtain a measure of the precision of the recovery for each analyte. (QAMS)
May: denotes permitted action, but not required action. (NELAC)
Method Blank: a sample of a matrix similar to the batch of associated samples (when available) that is free from the analytes of interest, which is processed simultaneously with and under the same conditions as samples through all steps of the analytical procedures, and in which no target analytes or interferences are present at concentrations that impact the analytical results for sample analyses. (NELAC)
Method Detection Limit: the minimum concentration of a substance (an analyte) that can be measured and reported with 99% confidence that the analyte concentration is greater than zero and is determined from analysis of a sample in a given matrix containing the analyte. (40 CFR Part 136, Appendix B)
Must: denotes a requirement that must be met. (Random House College Dictionary)
National Institute of Standards and Technology (NIST): an agency of the US Department of

Commerce's Technology Administration that is working with EPA, States, NELAC, and other public and commercial entities to establish a system under which private sector companies and interested States can be accredited by NIST to provide NIST-traceable proficiency testing (PT) to those laboratories testing drinking water and wastewater. (NIST)

Negative Control: measures taken to ensure that a test, its components, or the environment do not cause undesired effects, or produce incorrect test results. (NELAC)

(items), and services. The quality system provides the framework for planning, implementing, and assessing work performed by the organization and for carrying out required QA and QC. (ANSI/ASQC E-41994)

Quantitation Limits: the maximum or minimum levels, concentrations, or quantities of a target variable (e.g., target analyte) that can be quantified with the confidence level required by the data user. (NELAC)

Range: the difference between the minimum and the maximum of a set of values. (EPA-QAD)

Raw Data: any original factual information from a measurement activity or study recorded in a laboratory notebook, worksheets, records, memoranda, notes, or exact copies thereof that are necessary for the reconstruction and evaluation of the report of the activity or study. Raw data may include photography, microfilm or microfiche copies, computer printouts, magnetic media, including dictated observations, and recorded data from automated instruments. If exact copies of raw data have been prepared (e.g., tapes which have been transcribed verbatim, data and verified accurate by signature), the exact copy or exact transcript may be submitted. (EPA-QAD)

Reagent Blank (method blank): a sample consisting of reagent(s), without the target analyte or sample matrix, introduced into the analytical procedure at the appropriate point and carried through all subsequent steps to determine the contribution of the reagents and of the involved analytical steps. (QAMS)

Record Retention: The systematic collection, indexing and storing of documented information under secure conditions. (EPA-QAD)

Reference Material: a material or substance one or more properties of which are sufficiently well established to be used for the calibration of an apparatus, the assessment of a measurement method, or for assigning values to materials. (ISO Guide 30-2.1)

Reference Method: a method of known and documented accuracy and precision issued by an organization recognized as competent to do so. (NELAC)

Reference Standard: a standard, generally of the highest metrological quality available at a given location, from which measurements made at that location are derived. (VIM-6.08)

Replicate Analyses: the measurements of the variable of interest performed identically on two or more sub-samples of the same sample within a short time interval. (NELAC)

Requirement:. denotes a mandatory specification; often designated by the term "shall". (NELAC)

Resource Conservation and Recovery Act (RCRA): the enabling legislation under 42 USC 321 *et seq.* (1976), that gives EPA the authority to control hazardous waste from the "cradle-to-grave", including its generation, transportation, treatment, storage, and disposal. (NELAC)

Sample Duplicate: two samples taken from and representative of the same population and carried through all steps of the sampling and analytical procedures in an identical manner. Duplicate samples are used to assess variance of the total method including sampling and analysis. (EPA-QAD)

Selectivity: (Analytical chemistry) the capability of a test method or instrument to respond to a target substance or constituent in the presence of non-target substances. (EPA-QAD)

Sensitivity: the capability of a method or instrument to discriminate between measurement responses representing different levels (e.g., concentrations) of a variable of interest. (NELAC)

Shall: denotes a requirement that is mandatory whenever the criterion for conformance with the specification requires that there be no deviation. This does not prohibit the use of alternative

approaches or methods for implementing the specification so long as the requirement is fulfilled. (ANSI)
Should: denotes a guideline or recommendation whenever noncompliance with the specification is permissible. (ANSI)
Spike: a known mass of target analyte added to a blank sample or sub-sample; used to determine recovery efficiency or for other quality control purposes. (NELAC)
Standard: the document describing the elements of laboratory accreditation that has been developed and established within the consensus principles of NELAC and meets the approval requirements of NELAC procedures and policies. (ASQC)
Standard Operating Procedures (SOPs): a written document which details the method of an operation, analysis or action whose techniques and procedures are thoroughly prescribed and which is accepted as the method for performing certain routine or repetitive tasks. (QAMS)
Standardized Reference Material (SRM): a certified reference material produced by the U.S. National Institute of Standards and Technology or other equivalent organization and characterized for absolute content, independent of analytical method. (EPA-QAD)
Supervisor (however named): the individual(s) designated as being responsible for a particular area or category of scientific analysis. This responsibility includes direct day-to-day supervision of technical employees, supply and instrument adequacy and upkeep, quality assurance/quality control duties and ascertaining that technical employees have the required balance of education, training and experience to perform the required analyses. (NELAC)
Surrogate: a substance with properties that mimic the analyte of interest. It is unlikely to be found in environment samples and is added to them for quality control purposes. (QAMS)
Systems Audit (also Technical Systems Audit): a thorough, systematic, qualitative on-site assessment of the facilities, equipment, personnel, training, procedures, record keeping, data validation, data management, and reporting aspects of a total measurement system. (EPA-QAD)
Test: a technical operation that consists of the determination of one or more characteristics or performance of a given product, material, equipment, organism, physical phenomenon, process or service according to a specified procedure. The result of a test is normally recorded in a document sometimes called a test report or a test certificate. (ISO/IEC Guide 2-12.1, amended)
Test Method: an adoption of a scientific technique for a specific measurement problem, as documented in a laboratory SOP. (NELAC)
Tolerance Chart: A chart in which the plotted quality control data is assessed via a tolerance level (e.g. +/- 10% of a mean) based on the precision level judged acceptable to meet overall quality/data use requirements instead of a statistical acceptance criteria (e.g. +/- 3 sigma) (applies to radiobioassay laboratories). (ANSI)
Toxic Substances Control Act (TSCA): the enabling legislation in 15 USC 2601 *et seq.*, (1976), that provides for testing, regulating, and screening all chemicals produced or imported into the United States for possible toxic effects prior to commercial manufacture. (NELAC)
Traceability: the property of a result of a measurement whereby it can be related to appropriate standards, generally international or national standards, through an unbroken chain of comparisons. (VIM-6.12)
United States Environmental Protection Agency (EPA): the federal governmental agency with responsibility for protecting public health and safeguarding and improving the natural environment (i.e., the air, water, and land) upon which human life depends. (US-EPA)
Validation: the process of substantiating specified performance criteria. (EPA-QAD)

Verification: confirmation by examination and provision of evidence that specified requirements have been met. (NELAC)NOTE: In connection with the management of measuring equipment, verification provides a means for checking that the deviations between values indicated by a measuring instrument and corresponding known values of a measured quantity are consistently smaller than the maximum allowable error defined in a standard, regulation or

specification peculiar to the management of the measuring equipment. The result of verification leads to a decision either to restore in service, to perform adjustment, to repair, to downgrade, or to declare obsolete. In all cases, it is required that a written trace of the verification performed shall be kept on the measuring instrument's individual record.

Work Cell: a well-defined group of analysts that together perform the method analysis. The members of the group and their specific functions within the work cell must be fully documented. (NELAC)

Appendix B

Tables

Table A Reporting Limits

Table B Required Safety Equipment

Table C Equipment

Table D Required Safety Equipment

Table E Reagents

Table F Standards

Table G Sample Collection

Table H Quality Control

Table I Initial Demonstration of Capability

Table A

REPORTING LIMITS

PCB LIST SOLID - GC

Parameter	Reporting Limit
AROCLOR 1016	50ug/kg
AROCLOR 1221	50ug/kg
AROCLOR 1232	50ug/kg
AROCLOR 1242	50ug/kg
AROCLOR 1248	50ug/kg
AROCLOR 1254	50ug/kg
AROCLOR 1260	50ug/kg

Table B

REQUIRED SAFETY EQUIPMENT

Item:	Safety Apparel Description	Mandatory for Procedure	Optional
1	Safety Goggles or Glasses	x	
2	Lab Coat		x
3	Gloves	x	
4	Face Shield		x

Table C

EQUIPMENT

Equipment	Manufacturer	Model(s)	Serial #	Date In Service
Accelerated solvent extractor (ASE) # 1	Dionex	200	96030214,	6/96
Accelerated solvent extractor (ASE) # 2	Dionex	200	98110378	5/00
Kontes heater tube blocks # 1	Kontes	NA	1776	
Kontes heater tube blocks # 2	Kontes	NA	1860	6/96
Kontes heater tube blocks # 3	Kontes	NA	2023	6/97

Table D
Supplies

Supplies	Manufacturer	Vendor	Catalog #
evaporative column	Kontes	VWR	569261
Kontes concentrator tube	Kontes	VWR	570051-2526
heater block	Kontes	VWR	720000-0000
ebullator	Kontes	VWR	569401
filters	Dionex	Dionex	049458
collection vials	I-Chem		405531
2 mL autosampler vials	VWR	VWR	66020-953
12 mL screw cap vials with caps		Fisher Scientific	03-391-7D
Aluminum crimp seals	VWR	VWR	66010-847
Rubber pipette bulb	NJ Rubber	VWR	R5002-2
N-Dex Nitrile Glove Powder free (medium)	Best	Fisher Scientific	6005 PFM
N-Dex Nitrile Glove Powder free	Best	Fisher Scientific	7005L
Labtone glassware detergent	VWR	VWR	21851-006
Tube and bottle brush	VWR	VWR	PK-5-17041-002
5 3/4" disposable pipets	Fisher Scientific	Fisher Scientific	13-678-20-A
Amber jars 9 oz.	QEC	VWR	2124-0009
25 mL adjustable repipetor	Brinkman	VWR	
Lab notebooks	VWR	VWR	51280-108
250 mL beakers	Kimble	Fisher Scientific	14005-250
150 mL beakers	Kimble	Fisher Scientific	02539J
60 degree powder funnel	Pyrex	VWR	30240-047
2.0 mL class A Volumetric Flask	Pyrex	Fisher Scientific	20-814-B
5.0 mL class A Volumetric Flask	Pyrex	Fisher Scientific	29623-300
25.0 mL class A Volumetric Flask	Pyrex	Fisher Scientific	10-210-A
50.0 mL class A Volumetric Flask	Pyrex	Fisher Scientific	10-210-B
25 uL gastight syringe	Hamilton	Fisher Scientific	24559
50 uL gastight syringe	Hamilton	Fisher Scientific	111111
100 uL gastight syringe	Hamilton	Fisher Scientific	13-684-100
250 uL gastight syringe	Hamilton	Fisher Scientific	81100
500 uL gastight syringe	Hamilton	Fisher Scientific	81217
1000 uL gastight syringe	Hamilton	Fisher Scientific	1482425
PCB 1016	Restek	Restek	32006
PCB 1221	Restek	Restek	32007
PCB 1232	Restek	Restek	32008
PCB 1242	Restek	Restek	32009
PCB 1248	Restek	Restek	32010
PCB 1254	Restek	Restek	32011
PCB 1260	Restek	Restek	32012
PCB surrogate (TCMX; DCB)	Restek	Restek	32000

Table E
Reagents

Reagent	Purity	Manufacturer	Vendor	Catalog #
Methylene Chloride	Pesticide Quality	Burdick & Jackson	Fisher Scientific	CS300-200
Nitrogen	99.99%	Whatman	----	----
Di-ionized Water	Type I ASTM	----	----	----
Sodium sulfate (anhydrous; 10-60 mesh)	ASC grade	Fisher Scientific	Fisher Scientific	MK802406
Acetone	Pesticide Quality	Burdick & Jackson	Fisher Scientific	010-4
Hexane	Pesticide Quality	Burdick & Jackson	Fisher Scientific	217-4

Table F

STANDARDS

Standard	Acronym	Concentration	Direction found in Section:	Alias
Laboratory control spike and duplicate	LCS/LCSD	500 ppb	Procedure	BS/BSD
Matrix spike and duplicate	MS/MSD	500 ppb	Procedure	MS/MSD
Surrogate	DCB	50 ppb	Procedure	NA

Table G

SAMPLE COLLECTION, PRESERVATION, SHIPMENT AND STORAGE

Matrix	Prep Method	Container(s)	Preservation	Shipment Conditions	Lab Storage Conditions
water	SW-846 3510	Amber bottle 1 liter	none	On ice 4+/- 2° Celsius	Refrigerate 4+/- 2° Celsius
soil	SW-846 3545	Amber jars 9 oz	none	On ice 4+/- 2° Celsius	Refrigerate 4+/- 2° Celsius

Table H

QUALITY CONTROL

Preparation Method ⇨ Quality Control Measure ⇩	**SW846 8082C**	**EPA 608**
Initial Calibration	Minimum of five levels; lowest level near but above MDL.	Minimum of five levels; lowest level near but above MDL.
Method Blank	One per batch of samples, up to 20 enviromental samples, whichever is more frequent.	One per batch of samples, up to 10 enviromental samples, whichever is more frequent.
Calibration Verification Standard (CVS)	One at the beginning of a 12 hour time clock or more frequent.	One at the beginning of a 12 hour time clock or more frequent.
Independent Calibration Verification	Indepenent source run immediately after the calibration curve.	Indepenent source run immediately after the calibration curve.
Laboratory Control Spike and Duplicate	One pair per batch of samples, up to 20 environmental samples, whichever is more frequent.	One pair per batch of samples, up to 10 environmental samples, whichever is more frequent.
Matrix Spike and Duplicate	One pair per batch of samples, up to 20 environmental samples, whichever is more frequent.	One pair per batch of samples, up to 10 environmental samples, whichever is more frequent.
Method Validation	Annually	Annually
MDL	Annually	Annually
Surrogate Standards	Added to every sample.	Added to every sample.

Table I

En Chem Initial Demonstration of Capability

- PCB1 INSTRUMENT

Compound/Element	Acceptable Recov. from Authoritative Method	En Chem Initial Demonstration Recovery
1016 WATER	70%-130%	93%
1016 SOIL	70%-130%	112%
1260 WATER	70%-130%	92%
1260 SOIL	70%-130%	114%

En Chem Initial Demonstration of Capability

EASY FLASH TDX - PCB2 SCREENING INSTRUMENT

Compound/Element	Acceptable Recov. from Authoritative Method	En Chem Initial Demonstration Recovery
1016 WATER TDX	70%-130%	77%
1016 SOIL TDX	70%-130%	106%
1260 WATER TDX	70%-130%	91%
1260 SOIL TDX	70%-130%	114%

TITLE: Analysis of Base/Neutral and Acid (BNA) Compounds by GC/MS - Full Scan

MATRICES:

This SOP pertains to Aqueous and Solids Matrices

DETECTION LIMITS:

The Reporting Limits used in the absence of Project or State Specific Required Detection Limits are found Appendix B Table A.

1.0 **SCOPE AND APPLICATION:**

1.1 This method is used to determine the concentration of various BNA compounds in water soils samples. Appendix B Table A contains the compounds that may be determined by this method and the detection limits for each compound in reagent water and soils.

2.0 **SUMMARY OF THE TEST METHOD:**

2.1 This method provides the gas chromatographic conditions for the separation of the compounds in the extract for the quantitative analysis by mass spectrometry. A volume of a sample extract is injected into a gas chromatograph (GC) and compounds in the GC effluent are analyzed by mass spectrometry (MS).

3.0 **DEFINITIONS:**

3.1 Terms used throughout this document are defined in Appendix A.

4.0 **INTERFERENCES:**

4.1 Method interferences may be caused by contaminants (primarily phthalate esters) in solvents, reagents, glassware and other sample processing hardware that lead to discrete artifacts and/or elevated baselines in the total ion current profiles. All of these materials must be routinely demonstrated to be free from interferences under the conditions of the analysis by running laboratory reagent blanks. Contact with common plastics or rubber products must be avoided.

4.1 Matrix interference's may be caused by contaminants that are co-extracted from the sample. The extent of matrix interference's will vary considerably from source to source.

5.0 **SAFETY:**

5.1 Safety is everyone's business at En Chem, Inc. and everyone is responsible for assisting in reducing unsafe and unhealthy working conditions or potential hazards. The company provides you with a safe place to work, but we need your cooperation to keep it safe. When you see something that does not look safe, or you see someone working in an unsafe manner, mention it to your Supervisor.

5.2 All samples should be treated as hazardous. Safety glasses, gloves, and lab coats are to be worn. The toxicity or carcinogenicity of each reagent used in this method has not been precisely defined. However, each chemical compound should be treated as a potential health hazard. Exposure to these chemicals must be reduced to the lowest possible level by a safe technique. A reference file of material safety data sheets should also be made

available to all personnel involved in the chemical analysis.

5.3 Required Safety Equipment is listed in Appendix B Table B

6.0 **EQUIPMENT AND SUPPLIES:**

6.1 See Appendix B Table C and Table D for a summary.

7.0 **REAGENTS AND STANDARDS:**

7.1 Internal standard solutions - The internal standards recommended are 1,4 Dichlorobenzene-d_4, Napthalene-d_8, Acenapthalene-d_{10}, Phenanthrene-d_{10}, Chrysene-d_{12}, and Perylene-d_{12}. Stock solutions obtained from Restek (Catalog # 31006) have each compound at 4000 ug/ml. Each 1 mL sample extract undergoing analysis should be spiked with 10 uL of this stock standard, resulting in a concentration of 40 ng/uL of each internal standard. Store at 4°C or less when not being used.

7.2 GC/MS tuning standard - A methylene chloride solution containing 50 ng/uL of Decafluorotriphenylphosphine (DFTPP) is prepared. A stock solution of 1000 ug/ml is obtained from Supleco (Catalog # 46700-U). Store at 4°C or less when not being used.

DFTPP/Degrade Tune Check Standard, 50 ppm
250 ul of 1000 ppm 8270 GC/MS Tuning Solution II diluted to 5.0ml in CH_2Cl_2.

7.3 Surrogate Standards - The surrogate standards are 2-Fluorophenol, Phenol-d_5, 2-Cholorphenol-d_4, 1,2 Dichlorobenzene-d_4, Nitrobenzene-d_5, 2-Fluorobiphenyl, 2,4,6-Tribromophenol, and p-Terphenyl-d_{14}. The intermediate surrogate solution contains base/neutral and acid based compounds. The B/N-based surrogate compounds are spiked at an on column concentration of 50 ng, while the acid-based surrogate compounds are spiked on column at a concentration of 75 ng. Samples, aqueous and soils are spiked with 100ul at a concentration of 500/750 mg/ml. Surrogate standards must be added to all blanks, spikes, and sample extracts. Take into account all dilutions of sample extracts. These stock standards are obtained from Restek (Catalog #'s 31082 and 31083) at a concentrations of 5000 ppm and 7500 ppm respectively.

8270 Surrogate Standard, 500/750 ppm
1.0 ml of 5000 ppm B/N surrogate + 1.0 ml of 7500 ppm acid surrogate diluted to 10 ml in CH_2Cl_2.

7.4 Matrix Spike standards - A sample and a duplicate are spiked with the target compound list at a on column concentration of 50 ng. 250 ul of a 200 ug/ml stock custom mix standard, along with 10 ul of a 5000ppm N-Nitrosodiphenylamine stock solution are injected into 20.0 grams of soil or 1.0 L of water. The concentration for a soil sample is at 2500 ug/kg for each compound, while the aqueous sample concentration is at 50ug/L.

7.5 Laboratory Control Standards - same as matrix spikes except 20 grams of sand is used for soil samples and 1.0 L of de-ionized water is used for aqueous samples instead of the sample.

7.6 See Appendix B Table E and Table F for a summary.

8.0 **SAMPLE COLLECTION, PRESERVATION, SHIPMENT AND STORAGE:**

8.1 See Appendix B Table G for a summary.

9.0 **QUALITY CONTROL:**

9.1 En Chem operates a formal quality control program. The minimum requirements of this *program consist of an initial demonstration of laboratory capability and an ongoing* analysis of spiked samples to evaluate and document quality data. En Chem maintains records to document the quality of the data generated. Ongoing data quality checks are compared with established performance criteria to determine if the results of analyses meet the performance characteristics of the method. When results of sample spikes indicate atypical method performance, a quality control reference sample must be analyzed to confirm that the measurements were performed in an in-control mode of operation.

9.2 Before processing any samples, the analysts demonstrates, through the analysis of a method blank, that interferences from the analytical system, glassware, and reagents are under control. Each time a set of samples is extracted or there is a change in reagents, a reagent blank is processed as a safeguard against chronic laboratory contamination. The blanks are carried through all stages of sample preparation and measurement.

9.3 The experience of the analyst performing GC/MS analyses is invaluable to the success of the methods. Each day that analysis is performed, the daily calibration standard should be evaluated to determine if the chromatographic system is operating properly. Questions that are asked: Do the peaks look normal?; Is the response obtained comparable to the response from previous calibrations? Careful examination of the standard chromatogram can indicate whether the column is still good, the injector is leaking, the injector septum needs replacing, etc. If any changes are made to the system (e.g. column changed), recalibration of the system must take place.

9.4 . Required instrument QC is as follows:

9.5 The GC/MS system must be tuned to meet the DFTPP specification. See Table Q for DFTPP Key Ions And Ion Abundance Criteria

9.6 There must be an initial calibration of the GC/MS system as specified.

9.7 The GC/MS system must meet the above criteria every 12 hours or when a new time clock is begun.

9.8 When one or more of the analytes tested fail at least one of the acceptance criteria, the analyst must proceed according to the following corrective factors.

Locate and correct the source of the problem and repeat the test for all analytes of interest beginning with the daily GC/MS calibration section.

Beginning with the daily GC/MS calibration section, repeat the test only for those analytes that failed to meet criteria. Repeated failure, however, will confirm a general problem with the measurement system. If this occurs, locate and correct the source of the problem and repeat the test for all compounds of interest beginning with the daily GC/MS calibration section.

9.9 The laboratory must, on an ongoing basis, analyze a reagent blank, a laboratory control spike, a laboratory control spike duplicate, a matrix spike, and a replicate matrix spike for each analytical batch (up to maximum of 20 samples/batch) to assess accuracy. For soil and waste samples where detectable amounts of organics are present, replicate samples may be appropriate in place of matrix spiked samples. If analyzing only one to ten samples per month, at least one spiked sample per month is required.

9.10 As part of the QC program for the laboratory, method accuracy for each matrix studied must be assessed and records must be maintained. After the analysis of twenty spiked samples (of the same matrix) calculate the average percent recovery, (p), and the standard deviation of the percent recovery, (s(p)). Express the accuracy assessment as a percent recovery interval from average of (p) - 3s(p) to average of (p) + 3s(p). If average of (p) = 90% and s(p) = 10%, for example, the accuracy interval is expressed as 60-120%. Update the accuracy assessment for each analyte once a year. The same process should be done for laboratory control spikes and laboratory control spike duplicates.

9.11 To determine acceptable accuracy and precision limits for surrogate standards the following procedure should be performed:

For each sample analyzed, calculate the percent recovery of each surrogate in the sample.

Once a minimum of forty samples of the same matrix have been analyzed, calculate the average percent recovery (P) and standard deviation of the percent recovery(s) for each of the surrogates. (Forty random samples are chosen yearly for limit assessment).

For a given matrix, calculate the upper and lower control limit for method performance for each surrogate standard. This should be done as follows:

Upper Control Limit (UCL) = p + 3s
Lower Control Limit (LCL) = p - 3s

If recovery is not within limits, the following procedures are required:

Check to be sure there are no errors in calculations, surrogate solutions and internal standards. Also, check instrument performance.

Recalculate the data and/or reanalyze the extract if any of the above checks reveal a problem.

Re-extract and reanalyze the sample if none of the above are a problem or flag the data as "estimated concentration" if insufficient sample.

At a minimum, each laboratory should update surrogate recovery limits on a matrix-by matrix basis, annually. See Appendix B Table J''' for a summary.

See Appendix B Table H for a summary.

10.0 CALIBRATION AND STANDARDIZATION:

10.1 Calibration standards

A minimum of six calibration standards should be prepared. One of the calibration standards should be at a concentration near, but above, the method detection limit; the others should correspond to the range of concentrations found in real samples but should not exceed the working range of the GC/MS system. Each standard should contain each analyte for detection by this method (e.g. some or all of the compounds listed in Table S may be included). Each 1 mL aliquot of calibration standard should be spiked with 10 uL of the 4000 ppm internal stock standard solution prior to analysis. All standards should be stored at 4°C or less and should be freshly prepared once a year, or sooner if check standards indicate a problem. The daily calibration standard should be prepared bimonthly and stored at 4°C. The calibration standards are made from a stock custom mix solution of 400 ug/ml compounds obtained from Supleco (Catalog # 20114121). The daily calibration standards are made from the 200 ug/ml intermediate solution (Mix #1).

8270 Continuing Calibration Check Standard, 50 ppm
-250ul of 200 ppm 8270 spike mix #1 diluted to 1.0ml in CH_2Cl_2.

8270 Initial Calibration Check Standard, 50 ppm
-250ul of 200 ppm 8270 spike mix #2 + 10ul of 5000 ppm B/N surrogate + 6.66 ul of 7500 ppm acid surrogate diluted to 1.0ml in CH_2Cl_2.

Calibration Curve Standard, 200 ppm
-2.5ml of 400 ppm 8270 custom mix stock standard + 500ul of 2000 ppm EPA CLP SOW OL MO4 semi volatile mix + 200ul of 5000 ppm N-Nitrosodiphenylamine diluted to 5.0ml in CH_2Cl_2 (Mix #1)

Calibration Curve Pt. 1, 5 ppm
-25ul of 200 ppm calibration standard diluted to 1.0ml in CH_2Cl_2.
Calibration Curve Pt. 2, 10 ppm
-50ul of 200 ppm calibration standard diluted to 1.0ml in CH_2Cl_2.
Calibration Curve Pt. 3, 25 ppm
-125ul of 200 ppm calibration standard diluted to 1.0ml in CH_2Cl_2.
Calibration Curve Pt. 4, 50 ppm
-250ul of 200 ppm calibration standard diluted to 1.0ml in CH_2Cl_2.
Calibration Curve Pt. 5, 80 ppm
-400ul of 200 ppm calibration standard diluted to 1.0ml in CH_2Cl_2.
Calibration Curve Pt. 6, 100 ppm
-500ul of 200 ppm calibration standard diluted to 1.0ml in CH_2Cl_2.
Calibration Curve Pt. 7, 120 ppm
-600ul of 200 ppm calibration standard diluted to 1.0ml in CH_2Cl_2.

See Appendix B Table I for a summary.

Initial calibration:

Inject 1 ul of 50ng of Decafluorotriphenylphosphine (DFTPP). The average of three scans (the apex and the scan before and after the apex) may be used. Background subtraction is required and must be accomplished using a single scan acquired no more than 20 scans prior to the elution of DFTPP. Compare the mass listing to the tuning criteria in Appendix B. The tuning criteria must be met in order to continue calibration or sample analysis.

NOTE: All subsequent standards, samples, MS/MSDs, and blanks associated with a DFTPP analysis must use the identical mass spectrometer instrument conditions.

The DFTPP tuning standard includes 50 ng each of the following additional compounds, Pentachlorophenol, Benzidine, and DDT in order to assess GC column performance and injection port inertness. The responses for Benzidine and Pentachlorophenol should be normal with no peak tailing. The degradation of DDT to DDE and DDD should not exceed 20%. If degradation of DDT is excessive and/or the chromatography for Benzidine or Pentachlorophenol is poor the injection port may require cleaning and replacement of the glass liner, liner insert, and the gold seal. Also 6-12 inches of the column should be cut off. This preventative must be performed prior to sample analysis. If the response for Pentachlorophenol continues to be very poor or absent then the column may need to be replaced.

$$\text{Total peak area of \% DDT Breakdown} = \frac{(DDD + DDE)}{(DDD + DDE + DDT)} \times 100\%$$

Inject 1 µL of each of the calibration standard solutions, SSTD005, SSTD010, SSTD025, SSTD050, SSTD080, SSTD100, and SSTD120. Determine the response factors (RF), the average RF and percent relative standard deviation (%RSD) for each compound.

$$RF = (A_x C_{is})/(A_{is} C_x) \quad \text{(Equation 1)}$$

where:

A_x = Area of the characteristic ion for the compound being measured

A_{is} = Area of the characteristic ion for the specific internal standard

C_{is} = Concentration of the specific internal standard

C_x = Concentration of the compound being measured

$$\%RSD = 100[SD/RF] \qquad \text{(Equation 2)}$$

The %RSD should be less than or equal to 15% for each compound. The %RSD must not exceed 30% for the Calibration Check Compounds (CCC)

See Appendix B Table I for a summary and Appendix B Table Q for the compound list.

Linearity - If the %RSD is less than or equal to 15%, then the average RF is used for calculating the concentration of the compound being measured. If the %RSD is greater than 15% for compounds that have not demonstrated linearity then the analyst should select the regression order which introduces the least error into the quantitation.

South Carolina requires the use of a linear calibration model. Either the %RSD is less than 15% and the average RF is used for quantitation or a linear regression with a correlation coefficient greater than or equal to 0.99 must be used for all analytes listed in SW 846 Method 8270C.

The System Performance Check Compounds (SPCC) must have a minimum RF of 0.050. See Appendix B Table Q for the compound list.

If these criteria are not met, corrective action is required such as cleaning or replacing the injection port liner and/or capillary column.

See Appendix B Table I for a summary.

If the CCC's are not included in the list of analytes for a project, then all required analytes must meet the 30% RSD criterion.

See Appendix B Table I for a summary.

ANALYSIS OF INITIAL CALIBRATION VERIFICATION STANDARD

(Criteria may be based on Project Requirements approved by the Accrediting Authority)

In order to consider the initial calibration acceptable, an Initial Calibration Verification Standard (ICV) must be analyzed within the same time clock as the calibration curve. The ICV standard must be from a second source stock and meet the same criteria as the Continuing Calibration Verification (CCV) standard before the initial calibration may be considered valid.

See Appendix B Table I for a summary.

Daily GC/MS calibration:

Inject 1 µL of 50 ng DFTPP and compare the mass listing to the acceptance criteria in Table Q. The tuning standard must precede each 12 hours or per analysis sequence.

See Appendix B Table Q for a summary.

A midpoint calibration standard (50 ppm) must precede sample analysis. The calibration check response factors are compared to average response factors from the initial calibration.

The SPCCs must meet the minimum RF criteria of 0.050.

The CCCs must meet the percent difference (%D) criteria.

Calculation for %D: (Equation 3)

$$\%D = \frac{RF_{ave} - RF}{RF_{ave}} X100$$

If the percent difference for any compound is greater than 20%, this is considered a warning limit. If the percent difference for each of the CCCs is less than or equal to 20%, then the initial calibration is assumed valid and analysis of samples may proceed. If the 20% criterion is not met, corrective action must be taken in either instrument maintanence or re-calibration. If the CCCs are not analytes required, then all required analytes must meet the 20% drift criterion.

See Appendix B Table I for a summary.

If the retention time for any internal standard in the continuing calibration check standard changes by more than 30 seconds from the mid-point standard of the initial calibration, then the chromatographic system must be inspected for any malfunction and corrective action must be made.

If the area for any of the internal standards in the calibration verification standard changes by a factor of two (-50% to +100%) from that in the mid-point standard level of the most recent initial calibration sequence, the mass spectrometer must be inspected for malfunctions and corrections must be made, as appropriate. When corrections are made, reanalysis of samples analyzed while the system was malfunctioning is required.

See Appendix B Table I for a summary.

PROCEDURE:

Sample preparation: - Samples must be prepared by the following method prior to GC/MS analysis.

Matrix	Methods	
Soil/sediment	SW846 3545	EPA625
Aqueous	SW846 3510C	EPA625

Initial settings: - The recommended GC/MS operating conditions:

Mass Range:	35-500 amu
Initial temperature:	40°C, hold for 1 minute
Temperature program:	40-100°C at 18°C/min. 100-290°C at 15°C/min. hold for 5.95 minutes 290-320°C at 40°C/min.
Final temperature:	320°, hold for 1 minute
Injector temperature: 280°C	
Transfer line temperatue:	300°C
Injector:	Splitless for first 0.10 minutes
Injection pressure:	15 psi for 0.10 minutes and then constant flow of 1.2 mls/min.
Sample volume:	1.0 ul
Carrier gas:	Helium
Solvent delay:	2.5 minutes

Each GC/MS system must be hardware-tuned to meet the criteria in Table Q for a 50 ng injection of DFTPP. Analyses should not begin until all these criteria are met. The GC/MS tuning standard should also be used to assess GC column performance and injection port inertness.

The internal standard areas of the samples, method blanks, laboratory control samples, and matrix spikes must fall within a factor of two (-50% to +100%) range from the preceding midpoint calibration check standard. In addition, the relative retention times of the internal standards for each sample analysis must fall within a ± 30 second window defined by the midpoint calibration check standard.

GC/MS analysis:

All samples, method blanks, laboratory control samples and matrix spikes must be analyzed within 12 hours of a valid DFTPP tuning standard.

All samples, method blanks, laboratory control samples, and matrix spike extracts are spiked with 40 ng of internal standard solution mix just prior to analysis.

Surrogate recoveries are calculated using the following equation:

$$\text{Surrogate \% Recovery} = (C_{ex} / C_s) \times 100 \qquad \text{(Equation 4)}$$

where:

C_{ex} = Concentration of analyte in the extract (mg/L).
C_s = Calculated concentration of analyte spiked into extract based on amount spiked (mg/L).

Compare the surrogate recoveries according to the specific matrix to the recovery limits in Table J***. These limits are updated annually.

Qualitative sample analysis:

The relative retention time (RRT) for the sample component must compare within ± 0.06 RRT units of the standard component.

The mass spectrum for a sample component should compare to the spectrum of the standard component. Note: These criteria do not overrule the judgment of the analyst.

All ions present in the standard mass spectrum greater than 10% should be present in the sample spectrum.

The relative intensities of those ions must agree within ± 30% of those ions in the reference spectrum.

Quantitative sample analysis:

When a compound has been identified, the quantitation of that compound will be based on the integrated abundance from the extracted ion current profile (EICP) of the primary characteristic ion. See Table P for primary ion (1°) of each compound.

Aqueous: (Equations 5 and 6)

$$\text{concentration } (\mu g/L) = \frac{(C_{ex})(VF)(DF)}{(V_o)}$$

Soil:

$$\text{concentration } (\mu g/kg) = \frac{(C_{ex})(VF)(DF)}{(W_s)(D)}$$

where:

C_{ex} = Concentration of analyte in the extract (ug/ml)
DF = Dilution factor (if applicable)
V_o = Initial sample volume (L)
VF = Final extract volume (ml)
Ws = Initial sample weight extracted (kg)
D = % solids

CALCULATIONS:

Equation 1: $RF = (A_x C_{is})/(A_{is} C_x)$

where:

A_x = Area of the characteristic ion for the compound being measured

A_{is} = Area of the characteristic ion for the specific internal standard

C_{is} = Concentration of the specific internal standard

C_x = Concentration of the compound being measured

Equation 2: %RSD = 100[SD/RF]

Where:

$$\text{Mean rf} = \sqrt{\frac{\sum_{i=1}^{n} RF_i}{n}} \quad \text{and} \quad SD = \sqrt{\frac{\sum_{i=1}^{n} (RF_i - RF)^2}{n-1}}$$

The %RSD should be less than or equal to 15% for each compound. The %RSD must not exceed 30% for the Calibration Check Compounds (CCC).

Equation 3: Calculation for %D:

$$\%D = \frac{RF_{std} - RF}{RF_{ave}} X100$$

Equation 4: Surrogate % Recovery = $(C_{ex} / C_s) \times 100$

where:

C_{ex} = Concentration of analyte in the extract (mg/L).
C_s = Calculated concentration of analyte spiked into extract based on amount spiked (mg/L).

Equations 5 and 6: Aqueous:

$$\text{concentration } (\mu g/L) = \frac{(C_{ex})(VF)(DF)}{(V_o)}$$

Soil:

$$\text{concentration } (\mu g/kg) = \frac{(C_{ex})(VF)(DF)}{(W_s)(D)}$$

where:

C_{ex} = Concentration of analyte in the extract (ug/ml)
DF = Dilution factor (if applicable)
V_o = Initial sample volume (L)
VF = Final extract volume (ml)
Ws = Initial sample weight extracted (kg)
D = % solids

13.0 METHOD PERFORMANCE:

13.1 A series of 4 spikes in 1.0L are extracted and analyzed before a method is put online and whenever a new analyst is hired. Method performance validations must performed at least once a year.

13.2 See Appendix B Table J for a Initial Demonstration of Capability summary.

13.3 Limits should be established for surrogate recoveries, laboratory control spikes and matrix spikes using statistic from past samples. See Appendix B and the following tables for control limits:

Laboratory Control Spikes	Table J'
Matrix Spikes	Table J''
Surrogate Spikes	Table J'''

14.0 POLLUTION PREVENTION:

14.1 Pollution prevention encompasses any technique or procedure that reduces or eliminates the quantity or toxicity of waste at the point of generation. Laboratory staff should order where possible acceptable non-toxic alternative supplies and prepare only those quantities of reagents or standards that will be used prior to the expiration date. Other appropriate measures to minimize waste generation should be brought to the attention of laboratory management. All laboratory waste shall be handled as directed by the Laboratory Waste Management Plan and Hazardous Waste Contingency Plan.

15.0 DATA ASSESSMENT AND ACCEPTANCE CRITERIA FOR QUALITY CONTROL MEASURES:

15.1 At a minimum there are three levels of data review:

15.2 Analyst/Technician verification at the bench top are looking at instrument performance as it relates to initial calibration, calibration verification, cleanliness.

15.3 Supervisor/Analyst verification after analysis are looking for sample concentration versus linear range of the instrument, typical patterns resulting from the compounds or elements in question, and quality control measure criteria. Data is either accepted without qualification, accepted with qualification, or rejected with samples reprocessed.

15.4 Report Reviewer/Project Management personnel look at the presentation of the data on final reports. The reports are verified for holding time compliance, receipt conditions, method citation and reasonableness, which may include field duplicate analysis, analytical comments and qualifications presented by the previous reviews, and comparing results of similar analytical techniques as it relates to the project site information when available.

15.5 A fourth level of review, data validation is determined from a project or program scope of laboratory services and encompasses the generation Quality Control summaries in the media of "Form Generation."

15.6 See Appendix B and the following tables for a summary of specific items cross checked.

Analyst/Technician Data Assessment Table K

Analyst/Supervisor Data Assessment	Table L
Report Reviewer/Project Manager Data Assessment	Table M

16.0 CORRECTIVE ACTIONS FOR OUT-OF-CONTROL DATA

16.1 Assessment of Quality Control measures is done to provide a level of confidence in the data generated. The measures provide documentation that the instrument conditions were reliable during the analysis. See Table N for corrective actions taken in cases where statistical goals were not achieved.

17.0 CONTINGENCIES FOR HANDLING OUT-OF-CONTROL OR UNACCEPTABLE DATA

17.1 During analysis, events occur specific to the physical and chemical characteristics of the environmental sample. When possible, with received sample volumes, data generated along with measures that do not meet statistical goals are re-analyzed again to see if the statistical goal can be achieved. When environmental samples do not meet statistical unacceptable data is generated. These events are different from those pertaining to instrument operating conditions. These events occur when the instruments are operating under ideal conditions. Contingencies for handling sample out-of-control quality control assessments are found in Table O. Data Qualifiers are found in Table R. The tables list routine assessment and not those specific to a project/program where data validation is determined from a project or program scope of laboratory services and encompasses the generation Quality Control summaries in the media of "Form Generation."

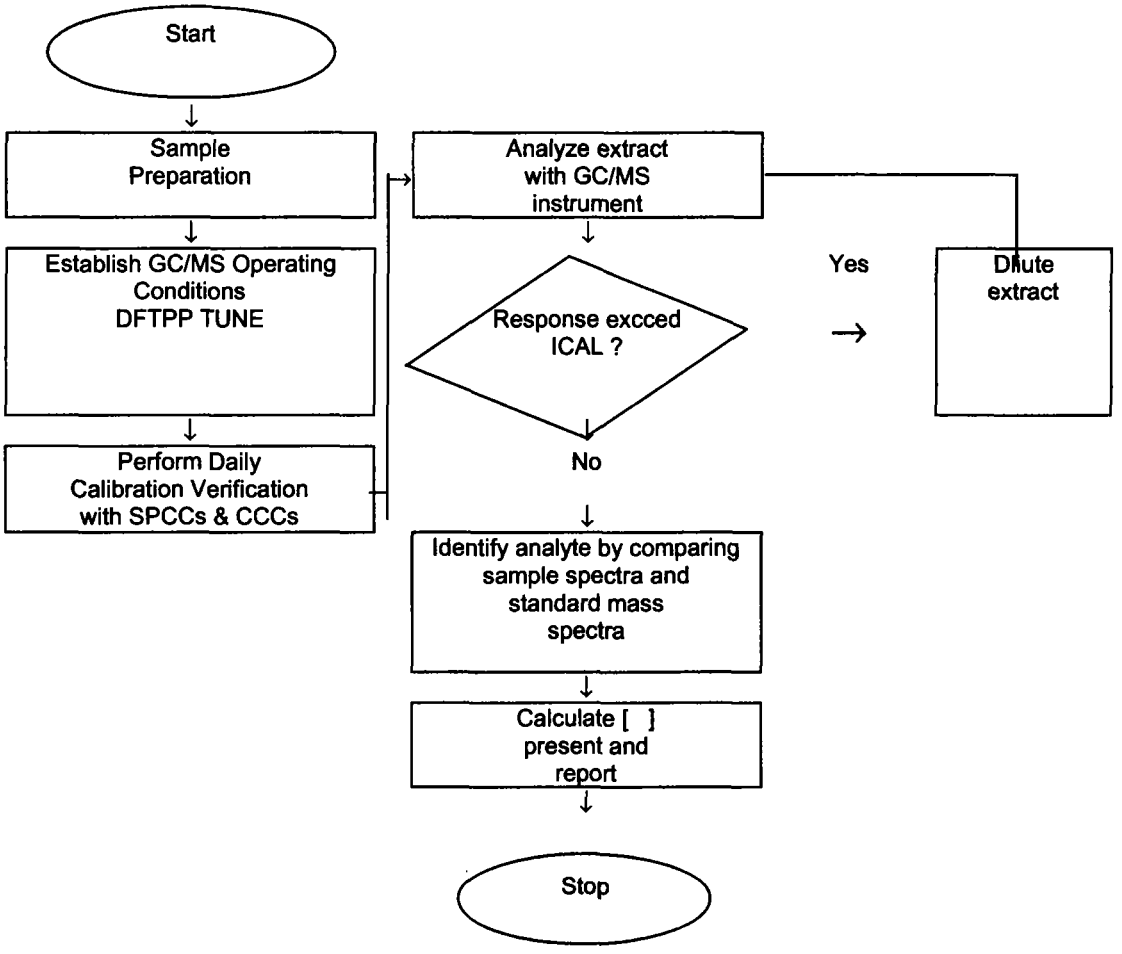
18.0 WASTE MANAGEMENT

18.1 To minimize waste during sample preparation has two benefits. The first benefit is a cost savings to the lab in materials and supplies. The second is a benefit to the environment as less materials need to be disposed.

REFERENCES:

40CFR Part 136
American Society for Quality Control (ASQC), Definitions of Environmental Quality Assurance Terms, 1996
American National Standards Institute (ANSI), Style Manual for Preparation of Proposed American National Standards, Eighth Edition, March 1991
ANSI/ASQC E4, 1994
ANSI N42.23-1995, Measurement and Associated Instrument Quality Assurance for
National Institute of Standards and Technology (NIST)
National Environmental Laboratory Accreditation Conference (NELAC), July 1998 Standards
Random House College Dictionary
US EPA Quality Assurance Division (QAD)
Webster's New World Dictionary of the American Language
SW846 Method 8000B, December 1996
SW846 Method 8270C, December 1996
40 CFR EPA 625

FLOWCHARTS


Start
Sample Preparation
Establish GC/MS Operating Conditions DFTPP TUNE
Perform Daily Calibration Verification with SPCCs & CCCs
Analyze extract with GC/MS instrument
Response exceed ICAL ?
Yes
Dilute extract
No
Identify analyte by comparing sample spectra and standard mass spectra
Calculate [ ] present and report
Stop

VALIDATION DATA:

The laboratory must demonstrate initial proficiency with each combination of sample preparation and determinative methods utilized, by generating data of acceptable accuracy and precision for a reference sample containing the target parameters in a clean matrix. The laboratory must also repeat this demonstration whenever new staff are trained or significant changes in instrumentation are made. Analysts previously trained shall perform a continued demonstration of proficiency annually by performing "Yearly Demonstration of Capability" (YDC).

The values from the determinative method are presented as guidance only and are not intended as absolute acceptance criteria. En Chem generates data for method performance and are presented along with the performance values in Table J.

MANAGEMENT APPROVAL AND REVIEW OF SOPS - POLICY AND DOCUMENTATION

REVIEWED BY: _____ Section Supervisor — Date: 01/04/2002

REVIEWED BY: M. Suha Quality Assurance Officer — Date: 01/04/2002

APPROVED BY: _____ Laboratory Manager — Date: 1/4/2002

Periodic Review Record

Review Date						
Initials						

APPENDICES

Appendix A

Acceptance Criteria: specified limits placed on characteristics of an item, process, or service defined in requirement documents. (ASQC)
Accuracy: the degree of agreement between an observed value and an accepted reference value. Accuracy includes a combination of random error (precision) and systematic error (bias) components which are due to sampling and analytical operations; a data quality indicator. (QAMS)
Analytical Detection Limit: the smallest amount of an analyte that can be distinguished in a sample by a given measurement procedure throughout a given (e.g., 0.95) confidence interval. (applicable only to radiochemistry)
Analyst: the designated individual who performs the "hands-on" analytical methods and associated techniques and who is the one responsible for applying required laboratory practices and other pertinent quality controls to meet the required level of quality. (NELAC)
Assessment Criteria: the measures established by NELAC and applied in establishing the extent to which an applicant is in conformance with NELAC requirements. (NELAC)
Data Reduction: the process of transforming raw data by arithmetic or statistical calculations, standard curves, concentration factors, etc., and collation into a more useable form. (EPA-QAD)
Deficiency: an unauthorized deviation from acceptable procedures or practices, or a defect in an item. (ASQC)
Demonstration of Capability: a procedure to establish the ability of the analyst to generate acceptable accuracy. (NELAC)
Detection Limit: the lowest concentration or amount of the target analyte that can be determined to be different from zero by a single measurement at a stated degree of confidence.
Document Control: the act of ensuring that documents (and revisions thereto) are proposed, reviewed for accuracy, approved for release by authorized personnel, distributed properly and controlled to ensure use of the correct version at the location where the prescribed activity is performed. (ASQC)
Duplicate Analyses: the analyses or measurements of the variable of interest performed identically on two sub-samples of the same sample. The results from duplicate analyses are used to evaluate analytical or measurement precision but not the precision of sampling, preservation or storage internal to the laboratory. (EPA-QAD)
Equipment Blank: a sample of analyte-free media which has been used to rinse common sampling equipment to check effectiveness of decontamination procedures. (NELAC)
estimate of Target analyte concentration is available. Matrix spikes are used, for example, to determine the effect of the matrix on a method's recovery efficiency. (QAMS)
Matrix Spike Duplicate (spiked sample or fortified sample duplicate): a second replicate matrix spike prepared in the laboratory and analyzed to obtain a measure of the precision of the recovery for each analyte. (QAMS)
May: denotes permitted action, but not required action. (NELAC)
Method Blank: a sample of a matrix similar to the batch of associated samples (when available) that is free from the analytes of interest, which is processed simultaneously with and under the same conditions as samples through all steps of the analytical procedures, and in which no target analytes or interferences are present at concentrations that impact the analytical results for sample analyses. (NELAC)
Method Detection Limit: the minimum concentration of a substance (an analyte) that can be measured and reported with 99% confidence that the analyte concentration is greater than zero and is determined from analysis of a sample in a given matrix containing the analyte. (40 CFR Part 136, Appendix B)
Must: denotes a requirement that must be met. (Random House College Dictionary)

National Institute of Standards and Technology (NIST): an agency of the US Department of Commerce's Technology Administration that is working with EPA, States, NELAC, and other public and commercial entities to establish a system under which private sector companies and interested States can be accredited by NIST to provide NIST-traceable proficiency testing (PT) to those laboratories testing drinking water and wastewater. (NIST)
Negative Control: measures taken to ensure that a test, its components, or the environment do not cause undesired effects, or produce incorrect test results. (NELAC)
(items), and services. The quality system provides the framework for planning, implementing, and assessing work performed by the organization and for carrying out required QA and QC. (ANSI/ASQC E-41994)
Quantitation Limits: the maximum or minimum levels, concentrations, or quantities of a target variable (e.g., target analyte) that can be quantified with the confidence level required by the data user. (NELAC)
Range: the difference between the minimum and the maximum of a set of values. (EPA-QAD)
Raw Data: any original factual information from a measurement activity or study recorded in a laboratory notebook, worksheets, records, memoranda, notes, or exact copies thereof that are necessary for the reconstruction and evaluation of the report of the activity or study. Raw data may include photography, microfilm or microfiche copies, computer printouts, magnetic media, including dictated observations, and recorded data from automated instruments. If exact copies of raw data have been prepared (e.g., tapes which have been transcribed verbatim, data and verified accurate by signature), the exact copy or exact transcript may be submitted. (EPA-QAD)
Reagent Blank (method blank): a sample consisting of reagent(s), without the target analyte or sample matrix, introduced into the analytical procedure at the appropriate point and carried through all subsequent steps to determine the contribution of the reagents and of the involved analytical steps. (QAMS)
Record Retention: The systematic collection, indexing and storing of documented information under secure conditions. (EPA-QAD)
Reference Material: a material or substance one or more properties of which are sufficiently well established to be used for the calibration of an apparatus, the assessment of a measurement method, or for assigning values to materials. (ISO Guide 30-2.1)
Reference Method: a method of known and documented accuracy and precision issued by an organization recognized as competent to do so. (NELAC)
Reference Standard: a standard, generally of the highest metrological quality available at a given location, from which measurements made at that location are derived. (VIM-6.08)
Replicate Analyses: the measurements of the variable of interest performed identically on two or more sub-samples of the same sample within a short time interval. (NELAC)
Requirement: denotes a mandatory specification; often designated by the term "shall". (NELAC)
Resource Conservation and Recovery Act (RCRA): the enabling legislation under 42 USC 321 *et seq.* (1976), that gives EPA the authority to control hazardous waste from the "cradle-to-grave", including its generation, transportation, treatment, storage, and disposal. (NELAC)
Sample Duplicate: two samples taken from and representative of the same population and carried through all steps of the sampling and analytical procedures in an identical manner. Duplicate samples are used to assess variance of the total method including sampling and analysis. (EPA-QAD)
Selectivity: (Analytical chemistry) the capability of a test method or instrument to respond to a target substance or constituent in the presence of non-target substances. (EPA-QAD)
Sensitivity: the capability of a method or instrument to discriminate between measurement responses representing different levels (e.g., concentrations) of a variable of interest. (NELAC)
Shall: denotes a requirement that is mandatory whenever the criterion for conformance with the specification requires that there be no deviation. This does not prohibit the use of

alternative approaches or methods for implementing the specification so long as the requirement is fulfilled. (ANSI)

Should: denotes a guideline or recommendation whenever noncompliance with the specification is permissible. (ANSI)

Spike: a known mass of target analyte added to a blank sample or sub-sample; used to determine recovery efficiency or for other quality control purposes. (NELAC)

Standard: the document describing the elements of laboratory accreditation that has been developed and established within the consensus principles of NELAC and meets the approval requirements of NELAC procedures and policies. (ASQC)

Standard Operating Procedures (SOPs): a written document which details the method of an operation, analysis or action whose techniques and procedures are thoroughly prescribed and which is accepted as the method for performing certain routine or repetitive tasks. (QAMS)

Standardized Reference Material (SRM): a certified reference material produced by the U.S. National Institute of Standards and Technology or other equivalent organization and characterized for absolute content, independent of analytical method. (EPA-QAD)

Supervisor (however named): the individual(s) designated as being responsible for a particular area or category of scientific analysis. This responsibility includes direct day-to-day supervision of technical employees, supply and instrument adequacy and upkeep, quality assurance/quality control duties and ascertaining that technical employees have the required balance of education, training and experience to perform the required analyses. (NELAC)

Surrogate: a substance with properties that mimic the analyte of interest. It is unlikely to be found in environment samples and is added to them for quality control purposes. (QAMS)

Systems Audit (also Technical Systems Audit)**:** a thorough, systematic, qualitative on-site assessment of the facilities, equipment, personnel, training, procedures, record keeping, data validation, data management, and reporting aspects of a total measurement system. (EPA-QAD)

Test: a technical operation that consists of the determination of one or more characteristics or performance of a given product, material, equipment, organism, physical phenomenon, process or service according to a specified procedure. The result of a test is normally recorded in a document sometimes called a test report or a test certificate. (ISO/IEC Guide 2-12.1, amended)

Test Method: an adoption of a scientific technique for a specific measurement problem, as documented in a laboratory SOP. (NELAC)

Tolerance Chart: A chart in which the plotted quality control data is assessed via a tolerance level (e.g. +/- 10% of a mean) based on the precision level judged acceptable to meet overall quality/data use requirements instead of a statistical acceptance criteria (e.g. +/- 3 sigma) (applies to radiobioassay laboratories). (ANSI)

Toxic Substances Control Act (TSCA): the enabling legislation in 15 USC 2601 *et seq.*, (1976), that provides for testing, regulating, and screening all chemicals produced or imported into the United States for possible toxic effects prior to commercial manufacture. (NELAC)

Traceability: the property of a result of a measurement whereby it can be related to appropriate standards, generally international or national standards, through an unbroken chain of comparisons. (VIM-6.12)

United States Environmental Protection Agency (EPA): the federal governmental agency with responsibility for protecting public health and safeguarding and improving the natural environment (i.e., the air, water, and land) upon which human life depends. (US-EPA)

Validation: the process of substantiating specified performance criteria. (EPA-QAD)

Verification: confirmation by examination and provision of evidence that specified requirements have been met. (NELAC) NOTE: In connection with the management of measuring equipment, verification provides a means for checking that the deviations between values indicated by a measuring instrument and corresponding known values of a measured quantity are consistently smaller than the maximum allowable error defined in a standard, regulation or specification peculiar to the management of the measuring equipment. The result of verification leads to a decision either to restore in service, to perform adjustment, to

repair, to downgrade, or to declare obsolete. In all cases, it is required that a written trace of the verification performed shall be kept on the measuring instrument's individual record.

Work Cell: a well-defined group of analysts that together perform the method analysis. The members of the group and their specific functions within the work cell must be fully documented. (NELAC)

Appendix B

Tables

Table A	**Reporting Limits**
Table B	**Required Safety Equipment**
Table C	**Equipment**
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Table F	**Standards**
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Table J	**Initial Demonstration of Capability**
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Table J"	**Matrix Spike Limits**
Table J'''	**Surrogate Limits**
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Table L	**Analyst/Supervisor Data Assessment**
Table M	**Report/Reviewer/Project Manager Data Assessment**
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Table A

REPORTING LIMITS

MDL STUDY - SEMIVOLATILE TARGET COMPOUND LIST WATER - GC/MS

2002

Compound	MDL	Reporting Limit	Compound	MDL	Reporting Limit
Phenol	1.0	10	Acenaphthene	4.6	10
bis(2-Chloroethyl)ether	0.87	10	2,4-Dinitrophenol	3.0	25
2-Chlorophenol	1.1	10	Dibenzofuran	4.7	10
1,3-Dichlorobenzene	2.3	10	4-Nitrophenol	1.8	25
1,4-Dichlorobenzene	2.3	10	2,4-Dinitrotoluene	3.0	10
1,2-Dichlorobenzene	2.5	10	Fluorene	4.7	10
2-Methylphenol	2.3	10	Diethylphthalate	2.7	10
2,2-oxybis(1-Chloropropene)	3.6	10	4-Chlorophenyl phenyl ether	4.7	10
N-Nitroso-di-n-propylamide	4.3	10	4-Nitroaniline	1.8	25
4-Methylphenol	2.0	10	4,6-Dinitro-2-methylphenol	1.7	25
Hexachloroethane	2.3	10	N-Nitrosodiphenylamine	2.2	10
Nitrobenzene	3.5	10	4-Bromophenyl phenyl ether	3.6	10
Isophorone	4.5	10	Hexachlorobenzene	1.4	10
2-Nitrophenol	3.7	10	Pentachlorophenol	0.78	25
2,4-Dimethylphenol	2.9	10	Phenanthrene	2.1	10
bis(2-Chloroethoxy)methane	4.5	10	Anthracene	2.8	10
2,4-Dichlorophenol	3.7	10	Carbazole	1.4	10
1,2,4-Trichlorobenzene	3.0	10	di-n-Butylphthalate	1.4	10
Naphthalene	3.8	10	Fluoranthene	1.6	10
4-Chloroaniline	4.2	10	Pyrene	1.8	10
Hexachlorobutadiene	2.9	10	Butylbenzylphthalate	1.8	10
4-Chloro-3-methylphenol	4.1	10	Benzo(a)anthracene	1.5	10
2-Methylnaphthalene	3.9	10	3,3'-Dichlorobenzidine	2.8	10
Hexachlorocyclopentadiene	1.4	10	Chrysene	1.8	10
2,4,6-Trichlorophenol	4.0	10	bis(2-Ethylhexyl)phthalate	1.4	10
2,4,5-Trichlorophenol	4.6	10	di-n-Octylphthalate	1.3	10
2-Chloronaphthalene	4.2	10	Benzo(b)fluoranthene	2.2	10
2-Nitroaniline	4.2	25	Benzo(k)fluoranthene	2.4	10
Dimethylphthalate	3.5	10	Benzo(a)pyrene	1.5	10
Acenaphthylene	4.7	10	Indeno(1,2,3-cd)pyrene	1.6	10
2,6-Dinitrotoluene	3.7	10	Dibenzo(a,h)anthracene	2.4	10
3-Nitroaniline	2.8	25	Benzo(g,h,i)perylene	2.1	10

Units = ug/L

MDL STUDY - SEMIVOLATILE TARGET COMPOUND LIST SOIL - GC/MS

2002

Compound	MDL	Reporting Limit	Compound	MDL	Reporting Limit
Phenol	112	250	Acenaphthene	124	250
bis(2-Chloroethyl)ether	96	250	2,4-Dinitrophenol	59	500
2-Chlorophenol	109	250	Dibenzofuran	143	250
1,3-Dichlorobenzene	110	250	4-Nitrophenol	107	500
1,4-Dichlorobenzene	111	250	2,4-Dinitrotoluene	128	250
1,2-Dichlorobenzene	104	250	Fluorene	148	250
2-Methylphenol	149	250	Diethylphthalate	142	250
2,2-oxybis(1-Chloropropene)	96	250	4-Chlorophenyl phenyl ether	140	250
N-Nitroso-di-n-propylamide	99	250	4-Nitroaniline	160	500
4-Methylphenol	128	250	4,6-Dinitro-2-methylphenol	113	250
Hexachloroethane	111	250	N-Nitrosodiphenylamine	179	250
Nitrobenzene	101	250	4-Bromophenyl phenyl ether	145	250
Isophorone	93	250	Hexachlorobenzene	156	250
2-Nitrophenol	121	250	Pentachlorophenol	125	500
2,4-Dimethylphenol	124	500	Phenanthrene	153	250
bis(2-Chloroethoxy)methane	112	250	Anthracene	165	250
2,4-Dichlorophenol	138	250	Carbazole	135	250
1,2,4-Trichlorobenzene	108	250	di-n-Butylphthalate	139	250
Naphthalene	132	250	Fluoranthene	130	250
4-Chloroaniline	141	500	Pyrene	137	250
Hexachlorobutadiene	130	250	Butylbenzylphthalate	121	250
4-Chloro-3-methylphenol	133	250	Benzo(a)anthracene	110	250
2-Methylnaphthalene	109	250	3,3'-Dichlorobenzidine	211	250
Hexachlorocyclopentadiene	142	250	Chrysene	110	250
2,4,6-Trichlorophenol	125	250	bis(2-Ethylhexyl)phthalate	157	250
2,4,5-Trichlorophenol	130	250	di-n-Octylphthalate	134	500
2-Chloronaphthalene	132	250	Benzo(b)fluoranthene	143	250
2-Nitroaniline	122	500	Benzo(k)fluoranthene	115	250
Dimethylphthalate	130	250	Benzo(a)pyrene	52	250
Acenaphthylene	137	250	Indeno(1,2,3-cd)pyrene	203	250
2,6-Dinitrotoluene	138	250	Dibenzo(a,h)anthracene	58	250
3-Nitroaniline	158	500	Benzo(g,h,i)perylene	138	250

Units = ug/kg

Accepted:

Table B

REQUIRED SAFETY EQUIPMENT

Item:	Safety Apparel Description	Mandatory for Procedure	Optional
1	Safety Goggles or Glasses		x
2	Lab Coat		x
3	Gloves		x
4	Face Shield		x

Table C

EQUIPMENT

Equipment	Manufacturer	Model(s)	Serial #	Date In Service
SVMS1 Gas chromatograph	Hewlett Packard	5890 series II plus	3336A57925	March 1995
SVMS1 Mass spectrometer	Hewlett Packard	5972	3501A41845	March 1995
SVMS1 Injector	Hewlett Packard	18593B	3508A41845	March 1995
SVMS1 Autosampler tray	Hewlett Packard	18596M	3507A38055	March 1995
SVMS1 Controller	Hewlett Packard	G1512A	3448A00433	March 1995
SVMS1 Computer	Hewlett Packard	Vectra XM2	3305A65414	March 1995

Table D

SUPPLIES

Supplies	Manufacturer	Vendor	Catalog #
2 mL autosampler vials	VWR	VWR	66020-953
12 mL screw cap vials with caps		Fisher Scientific	03-391-7D
Aluminum crimp seals	VWR	VWR	66010-847
Rubber pipette bulb	NJ Rubber	VWR	R5002-2
N-Dex Nitrile Glove Powder free (medium)	Best	Fisher Scientific	6005 PFM
N-Dex Nitrile Glove Powder free (large)	Best	Fisher Scientific	7005L
Labtone glassware detergent	VWR	VWR	21851-006
Tube and bottle brush	VWR	VWR	PK-5-17041-002
9" disposable pipets	Fisher Scientific	Fisher Scientific	13-678-20-C
Glasswool 8 micron	Fisher Scientific	Fisher Scientific	430330 9989
pH indicator strips (0-14 range)	Whatman	VWR	EM-9590-3
Disposable culture tubes	VWR	VWR	60825-618
25 mL adjustable repipetor	Brinkman	VWR	
Lab notebooks	VWR	VWR	51280-108
Turbovap concentrator tube (200 mL; 1.0 mL endpoint)	Zymark	Zymark	45817
600 mL beakers	Kimble	Fisher Scientific	13910-145
250 mL beakers	Kimble	Fisher Scientific	14005-250
150 mL beakers	Kimble	Fisher Scientific	02539J
60 degree powder funnel	Pyrex	VWR	30240-047
2.0 Liter mechanical separatory funnel	Blue 92	Blue 92	PRG-1542-03
2.0 mL class A Volumetric Flask	Pyrex	Fisher Scientific	20-814-B
5.0 mL class A Volumetric Flask	Pyrex	Fisher Scientific	29623-300
25.0 mL class A Volumetric Flask	Pyrex	Fisher Scientific	10-210-A
50.0 mL class A Volumetric Flask	Pyrex	Fisher Scientific	10-210-B
XTI -5 30 meter capillary column with guard column .25 mm ID and 0.25 um film thickness	Restek	VWR	12223-124
1 liter Amber bottles; level 2 precleaned; with Teflon lined cap and 5 mL HCL	QEC	QEC	2121-0033
1 liter Amber bottles; level 3 certified; with Teflon lined cap	QEC	QEC	3121-0033
25 uL gastight syringe	Hamilton	Fisher Scientific	24559
50 uL gastight syringe	Hamilton	Fisher Scientific	111111
100 uL gastight syringe	Hamilton	Fisher Scientific	13-684-100
250 uL gastight syringe	Hamilton	Fisher Scientific	81100
500 uL gastight syringe	Hamilton	Fisher Scientific	81217
1000 uL gastight syringe	Hamilton	Fisher Scientific	1482425

Table E

REAGENTS

Reagent	Purity	Manufacturer	Vendor	Catalog #
Methylene Chloride	Pesticide Quality	Burdick & Jackson	Fisher Scientific	CS300-200
Acetonitrile	INSTRA Grade	Burdick & Jackson	VWR	015-4
Nitrogen	99.99%	Whatman	—	—
De-ionized Water	Type I ASTM	—	—	—
Sodium sulfate (anhydrous; 10-60 mesh)	ASC grade	Fisher Scientific	Fisher Scientific	MK802406
Sodium Hydroxide pellets	97+%	ACROS	Fisher Scientific	42433-0025
Nitric Acid	69-70%	JT Baker	VWR	9598-34
Acetone	Pesticide Quality	Burdick & Jackson	Fisher Scientific	010-4
Acetonitrile	Pesticide Quality	Burdick & Jackson	Fisher Scientific	015-4
Hexane	Pesticide Quality	Burdick & Jackson	Fisher Scientific	217-4
Water	HPLC Grade	EM Science	Fisher Scientific	EM-WX0004-1

Table F

STANDARDS

Standard	Acronym	Concentration	Direction found in Section:	Alias
Equity 8270 GC/MS Tuning Solution II	Tune Stock	1000 ug/ml	Stock Standard	Tune Stock
Custom LCS Spike Mix	Spk Mix	200 ug/ml	Stock Standard	Spk Mix
Equity n-Nitrosodiphenylamine	n-NDPA	5000 ug/ml	Stock Standard	n-NDPA
EPA CLP SOW OLMO4 Semivolatile Mix	Spk Mix	2000 ug/ml	Stock Standard	Spk Mix
Custom Mix	Cal Mix	400 ug/ml	Stock Standard	Calibration mix
B/N Surrogate	B/N Surr	5000 ug/ml	Stock Standard	B/N Surr
Acid Surrogate	Acid Surr	7500 ug/ml	Stock Standard	Acid Surr
SV Internal Standard Mix	INT STD	4000 ug/ml	Stock Standard	INT STD
8270 Tune/Degrad check standard	DFTPP	50 ug/ml	Reagents & Standards	DFTPP/Degrad
8270 Surrogate	Surrogate spike	500/750 ug/ml	Reagents & Standards	Surrogate spike
Continuing Calibration check standard	CCV	50 ug/ml	Reagents & Standards	Continuing Cal
Initial Calibration check standard	ICV	50 ug/ml	Reagents & Standards	Initial Cal
8270 Calibration Spike Mix		200 ug/ml	Reagents & Standards	Mix #1
8270 Spike Mix		200 ug/ml	Reagents & Standards	Mix #2

Table G

SAMPLE COLLECTION, PRESERVATION, SHIPMENT AND STORAGE

Matrix	Prep Method	Container(s)	Preservation	Shipment Conditions	Lab Storage Conditions
Aqueous	3510C/625	1 L amber glass with teflon lined cap	None/ Sodium Thiosulfate when residual chlorine present	On ice 4+/- 2° Celsius	Refrigerate 4+/- 2° Celsius
Solids	3545/625	amber glass jar with teflon lined cap	none	On ice 4+/- 2° Celsius	Refrigerate 4+/- 2° Celsius

Table H

QUALITY CONTROL

Preparation Method ⇨ Quality Control Measure ⇩	**SW846 8270C**	**EPA - 625**
Initial Calibration	Minimum of five levels; lowest level near but above MDL.	Minimum of five levels; lowest level near but above MDL.
Method Blank	One per batch of samples, up to 20 environmental samples, whichever is more frequent.	One per batch of samples, up to 20 environmental samples, whichever is more frequent.
DFTPP Tune Check	One at the beginning of a 12 hour time clock or more frequent.	One at the beginning of a 12 hour time clock or more frequent
Calibration Verification Standard (CVS)	One at the beginning of a 12 hour time clock or more frequent.	One at the beginning of a 12 hour time clock or more frequent.
Independent Calibration Verification	Independent source run immediately after the calibration curve.	Independent source run immediately after the calibration curve.
Laboratory Control Spike and Duplicate	One pair per batch of samples up to 20 environmental samples, whichever is more frequent.	One pair per batch of samples, up to 20 environmental samples, whichever is more frequent.
Matrix Spike and Duplicate	One pair per batch of samples up to 20 environmental samples, whichever is more frequent.	One pair per batch of samples, up to 20 environmental samples, whichever is more frequent.
Internal Standards	Added to every sample.	Added to every sample.
Method Validation	Annually	Annually
MDL	Annually	Annually
Surrogate Standards	Added to every sample.	Added to every sample.

Table I

CALIBRATION AND STANDARDIZATION

Analytical Method Acceptance Criteria⇨ Calibration Measure ⇩	**Method Cited**	**Frequency**	**Acceptance Criteria**
Initial Calibration	• SW846- 8270C • EPA 625	• Installation of new column • After cleaning mass spectrometer source • After mass spectrometer has been tuned • Creation of new analytical method • CVS fails criteria	• % RSD ≤ 15% for every compound • Use of Average Response factors if: % RSD is ≤ 15% for any reportable compounds Otherwise, use Linear Regression and Correlation Coefficient is > 0.9900.
Independent Calibration Verification Standard	• SW846- 8270C • EPA 625	• After every calibration	• RF of CVS must be ≤ 20% from initial calibration.
Continuing Calibration Verification	• SW846- 8270C • EPA 625	• At the beginning of every 12 hour shift or more frequent	• RF of CVS must be ≤ 20% from initial calibration.
DFTPP Tune Check	• SW846- 8270C • EPA 625	• At the beginning of every 12 hour shift or more frequent	• Must pass ion abundance criteria in Table Q
Laboratory Control Spike and Duplicate	• SW846- 8270C • EPA 625	• One pair per batch of samples, up to 20 environmental samples, whichever is more frequent. • One pair per batch of samples, up to 10 environmental samples, whichever is more frequent.	• See Table J under Lab Control Spike Recovery Limits
Matrix Spike and Duplicate	• SW846- 8270C • EPA 625	• One pair per batch of samples, up to 20 environmental samples, whichever is more frequent. • One pair per batch of samples, up to 10 environmental samples, whichever is more frequent.	• See Table J under Matrix Spike Recovery Limits
Internal Standards	• SW846- 8270C • EPA 625	• Added to every sample.	• Retention times monitored from the first CVS must be +/- 0.5 minutes. • Internal Standard area limits established from the area of the first CVS starting a sequence or method. Limit is - 50% to +100%
MDLs	• SW846- 8270C • EPA 625	• Annually	• 1-10 times the MDL should be equal to the spike concentration that was used to determine the MDLs
Method Validation	• SW846- 8270C • EPA 625	• Annually	• See Table J under Initial Demonstration of Capability
Surrogate Standards	• SW846- 8270C • EPA 625	• Added to every sample.	• In house limits determined. See Table J"" under Surrogate recovery Limits

Table J

En Chem Initial Demonstration of Capability

Compound	Recovery Range EPA 625	En Chem Initial Demonstration Recovery
Acenaphthene	47-145	88%
Acenaphthylene	33-145	90%
Anthracene	27-133	93%
Benz(a)anthracene	33-143	97%
Benzo(b)fluoranthene	24-159	98%
Benzo(k)fluoranthene	11-162	102%
Benzo(a)pyrene	17-163	97%
Benzo(g,h,i)perylene	D-219	96%
Benzyl butyl phthalate	D-152	101%
Bis(2-chloroethyl) ether	12-158	84%
Bis(2-chloroethoxy)methane	33-184	86%
Bis(2-chloroisopropyl) ether	36-166	85%
Bis(2-ethylhexyl) phthalate	8-158	103%
4-Bromophenyl phenyl ether	53-127	91%
2-Chloronaphthalene	60-118	87%
4-Chlorophenyl phenyl ether	25-158	92%
Chrysene	17-168	96%
Dibenzo(a,h)anthracene	D-227	93%
Di-n-butyl phthalate	1-118	100%
1,2-Dichlorobenzene	32-129	85%
1,3-Dichlorobenzene	D-172	82%
1,4-Dichlorobenzene	20-124	84%
3,3'-Dichlorobenzidine	D-262	113%
Diethyl phthalate	D-114	95%
Dimethyl Phthalate	D-112	93%
2,4-Dinitrotoluene	39-139	94%
2,6-Dinitrotoluene	50-158	91%
Di-n-octyl phthalate	4-146	101%
Fluoranthene	26-137	99%
Fluorene	59-121	93%
Hexachlorobenzene	D-152	91%
Hexachlorobutadiene	24-116	83%
Hexachloroethane	40-113	84%
Indeno(1,2,3-cd)Pyrene	D-171	95%
Isophorone	21-196	86%
Naphthalene	21-133	86%
Nitrobenzene	35-180	82%

Compound	Recovery Range EPA 625	En Chem Inital Demonstration Recovery
N-Nitroso-Di-n-Propylamine	D-230	86%
Phenanthrene	54-120	93%
Pyrene	52-115	100%
1,2,4-Trichlorobenzene	44-142	83%
4-Chloro-3-Methylphenol	22-147	87%
2-Chlorophenol	23-134	80%
2,4-Dichlorophenol	39-135	85%
2,4-Dimethylphenol	32-119	86%
2,4-Dinitrophenol	D-191	89%
4,6-Dinitro-2-Methylphenol	D-181	96%
2-Nitrophenol	29-182	85%
4-Nitrophenol	D-132	74%
Pentachlorophenol	14-176	98%
Phenol	5-112	34%
2,4,6-Trichlorophenol	37-144	89%
Additional Compounds		
2,4,5-Trichlorophenol		87%
2-Methylnaphthalene		88%
2-Methylphenol		74%
2-Nitroaniline		88%
3-Nitroaniline		102%
4-Chloroaniline		86%
4-Methylphenol		69%
4-Nitroaniline		126%
Benzo(a)Anthracene		97%
Benzoic Acid		36%
Benzyl Alcohol		77%
Carbazole		113%
Dibenz(a,h)Anthracene		93%
Hexachlorocyclopentadiene		88%
N-Nitrosodimethylamine		53%
N-Nitrosodiphenylamine		91%
Pyridine		25%
Aniline		51%
Benzaldehyde		103%
Acetophenone		84%
Caprolactum		27%
Azobenzene		92%
Atrazine		103%
Biphenyl		90%

Compound	Recovery Range EPA 625	En Chem Inital Demonstration Recovery
1-Methylnaphthalene		84%
Dibenzofuran		91%

Table J'

Laboratory Control Spike Limits

Spike Parameter	Aqueous		Soil	
	LCS	RPD	LCS	RPD
1,2 - Dichlorobenzene	46-104	21	62-113	16
1,2,4 - Trichlorobenzene	41-90	23	67-115	15
1,3 - Dichlorobenzene	39-87	20	60-110	16
1,4 - Dichlorobenzene	39-106	20	60-111	16
2 - Chloronaphthalene	56-123	24	76-119	16
2 - Chlorophenol	43-92	20	63-115	15
2 - Methylnaphthalene	52-114	26	67-116	15
2 - Methylphenol	40-86	19	57-123	18
2 - Nitroaniline	67-127	21	64-122	18
2 - Nitrophenol	50-115	24	68-118	15
2,2' - oxybis (1 Chloropropane)	49-109	18	58-125	16
2,4 - Dichlorophenol	53-116	24	72-120	14
2,4 - Dimethylphenol	42-124	31	49-119	14
2,4 - Dinitrophenol	24-145	31	42-131	17
2,4 - Dinitrotoluene	25-156	23	44-156	17
2,4,5 - Trichlorophenol	58-125	25	64-134	18
2,4,6 - Trichlorophenol	56-120	23	68-119	16
2,6 - Dinitrotoluene	68-127	20	70-128	18
3 - Nitroaniline	63-126	27	55-107	19
3,3 - Dichlorobenzidine	44-122	19	50-114	14
4 - Bromophenyl phenylether	64-124	23	67-122	17
4 - Chloro - 3 - methylphenol	53-116	24	72-128	17
4 - Chloroaniline	23-132	35	38-101	20
4 - Chlorophenyl phenylether	65-121	24	72-121	38
4 - Methylphenol	24-77	13	62-122	36
4 - Nitroaniline	38-196	36	58-147	27
4 - Nitrophenol	24-91	20	53-131	26
4,6 - Dinitro - 2 - Methylphenol	62-146	27	56-133	17
Acenaphthene	60-118	25	66-121	24
Acenaphthylene	57-120	26	71-116	23
Anthracene	74-117	17	68-115	24
Benzo(a)anthracene	76-127	22	68-128	24
Benzo(a)pyrene	68-131	26	64-125	22
Benzo(b)fluoranthene	61-144	22	65-133	21
Benzo(g,h,i)perylene	48-159	26	59-130	25
Benzo(k)fluoranthene	66-141	30	66-128	22
bis(2-Chloroethoxy)methane	52-118	25	68-116	26
bis(2-Chloroethyl)ether	50-108	21	59-116	20
bis(2-Ethylhexyl)phthalate	72-135	24	60-141	23
Butylbenzylphthalate	69-135	25	70-123	23
Carbazole	72-144	17	77-128	22
Chrysene	76-122	23	68-117	22
Di-n-butylphthalate	75-136	16	67-134	20
Di-n-octylphthalate	54-160	20	63-142	23

Spike Parameter	Aqueous		Soil	
	LCS	RPD	LCS	RPD
Dibenzo(a,h)anthracene	40-149	20	54-117	25
Dibenzofuran	60-121	24	71-119	26
Diethylphthalate	68-133	19	68-129	20
Dimethylphthalate	68-121	22	64-126	18
Fluoranthene	63-139	18	71-123	23
Fluorene	62-125	26	64-127	28
Hexachlorobenzene	67-123	18	65-121	26
Hexachlorobutadiene	38-104	21	62-118	26
Hexachlorocyclopentadiene	40-115	26	44-114	18
Hexachloroethane	58-128	18	59-118	18
Indeno(1,2,3-cd)pyrene	56-131	20	66-136	24
Isophorone	53-103	20	57-117	17
N-Nitrosodi-n-propylamine	57-107	22	64-120	25
N-Nitrosodiphenylamine	69-132	21	64-127	19
Naphthalene	51-113	22	68-119	17
Nitrobenzene	46-119	23	62-123	20
Pentachlorophenol	55-141	23	57-127	20
Phenanthrene	74-122	16	70-120	18
Phenol	40-110	20	64-119	21
Pyrene	60-136	20	66-129	23

Table J"

Matrix Spike Limits

Spike Parameter	Aqueous			Soil	
	MS	RPD	RPD	MS	RPD
1,2 - Dichlorobenzene	30-108	30	16	49-104	33
1,2,4 - Trichlorobenzene	36-107	18	15	49-109	35
1,3 - Dichlorobenzene	56-106	30	16	44-103	35
1,4 - Dichlorobenzene	28-118	30	16	45-103	35
2 - Chloronaphthalene	47-124	30	16	57-113	24
2 - Chlorophenol	33-98	24	15	46-109	27
2 - Methylnaphthalene	42-116	30	15	53-110	25
2 - Methylphenol	22-92	28	18	40-120	34
2 - Nitroaniline	56-131	23	18	49-120	30
2 - Nitrophenol	40-118	27	15	45-119	33
2,2' - oxybis (1 Chloropropane)	37-115	21	16	41-120	32
2,4 - Dichlorophenol	43-119	29	14	48-116	29
2,4 - Dimethylphenol	38-126	34	14	45-126	38
2,4 - Dinitrophenol	26-146	34	17	23-127	28
2,4 - Dinitrotoluene	26-156	24	17	24-145	28
2,4,5 - Trichlorophenol	55-124	26	18	45-123	32
2,4,6 - Trichlorophenol	51-121	25	16	42-115	30
2,6 - Dinitrotoluene	65-130	22	18	54-121	29
3 - Nitroaniline	61-141	33	19	41-107	31
3,3 - Dichlorobenzidine	41-151	22	14	40-130	35
4 - Bromophenyl phenylether	58-120	22	17	54-113	35
4 - Chloro - 3 - methylphenol	45-119	30	17	43-127	31
4 - Chloroaniline	16-135	42	20	35-107	26
4 - Chlorophenyl phenylether	56-123	26	38	55-116	37
4 - Methylphenol	18-82	30	36	44-117	31
4 - Nitroaniline	35-198	38	27	43-168	27
4 - Nitrophenol	23-103	30	26	36-132	29
4,6 - Dinitro - 2 - Methylphenol	53-120	33	17	45-130	32
Acenaphthene	49-123	33	24	56-110	31
Acenaphthylene	49-125	32	23	54-110	28
Anthracene	49-125	32	24	52-110	31
Benzo(a)anthracene	75-131	39	24	54-118	31
Benzo(a)pyrene	66-134	27	22	48-122	31
Benzo(b)fluoranthene	55-150	31	21	46-135	30
Benzo(g,h,i)perylene	33-171	31	25	40-132	33
Benzo(k)fluoranthene	66-143	34	22	60-126	27
bis(2-Chloroethoxy)methane	42-119	30	26	60-113	32
bis(2-Chloroethyl)ether	38-112	25	20	53-112	36
bis(2-Ethylhexyl)phthalate	72-138	26	23	64-135	32
Butylbenzylphthalate	66-139	32	23	63-127	34
Carbazole	65-154	19	22	65-131	36
Chrysene	74-124	26	22	63-113	28
Di-n-butylphthalate	75-137	21	20	66-127	29
Di-n-octylphthalate	45-155	36	23	63-148	37
Dibenzo(a,h)anthracene	35-129	22	25	43-121	35

Spike Parameter	Aqueous			Soil	
	MS	RPD	RPD	MS	RPD
Dibenzofuran	53-122	28	26	54-114	32
Diethylphthalate	68-139	27	20	54-120	32
Dimethyphthalate	67-119	25	18	55-114	32
Fluoranthene	63-141	21	23	46-129	34
Fluorene	56-126	27	28	55-118	30
Hexachlorobenzene	65-122	30	26	53-112	32
Hexachlorobutadiene	31-105	30	26	50-110	27
Hexachlorocyclopentadiene	41-129	30	18	39-136	28
Hexachloroethane	36-118	30	18	45-107	27
Indeno(1,2,3-cd)pyrene	51-162	33	24	45-141	33
Isophorone	49-115	30	17	44-125	25
N-Nitrosodi-n-propylamine	43-125	30	25	69-137	32
N-Nitrosodiphenylamine	61-132	24	19	43-124	26
Naphthalene	38-118	30	17	50-114	21
Nitrobenzene	34-121	26	20	44-117	22
Pentachlorophenol	35-142	26	20	38-125	28
Phenanthrene	73-124	24	18	54-115	25
Phenol	21-114	30	21	43-117	24
Pyrene	59-140	24	23	46-118	21

Table J'''
Surrogate Limits

Spike Parameter	Aqueous	Soil
2-Fluorophenol	13-70	35-113
Phenol-$_{d5}$	8-44	29-114
2-Chlorophenol-$_{d4}$	29-104	34-107
1,2-Dichlorobenzene-$_{d4}$	34-112	27-116
Nitrobenzene-$_{d5}$	34-126	32-118
2-Fluorobiphenyl	36-126	26-126
2,4,6-Tribromophenol	39-133	17-129
Terphenyl-$_{d14}$	56-139	23-141

Table K
ANALYST/TECHNICIAN DATA ASSESSMENT

Analytical Method Acceptance Criteria⇨ Data Assessment Measure ⇩	**Method Citation SW If these conditions are not achieved ⇒**	**Method Citation EPA If these conditions are not achieved ⇒**	**Method Citation EPA If these conditions are not achieved ⇒**
Initial Calibration	• 1	• 1	•
Initial Calibration Verification	• 1	• 1	•
Calibration Blank	• 1	• 1	•
Method Blank	• 2	• 2	•
Internal Standard Area	• 3	• 3	•
Continuing Calibration Verifications	• 4	• 4	•
Surrogate Recovery	• 5	• 5	•
Accuracy & Precision Laboratory Control Spikes	• 6	• 6	•
Accuracy & Precision Matrix Spikes	• 7	• 7	•

1. Check instrumentation/equipment condition; enter maintenance in instrument log; perform another initial calibration. No data can be reported. Generate on Non-Conformance Memo.

2. In the absence of project specific requirements, sample detects greater than 20 times the method blank contamination level are reported with those not exceeding 20 times re-analyzed or qualified. Generate on Non-Conformance Memo.

3. In the absence of project specific requirements, check to be sure that there are no errors in the calculations. If errors are found, recalculate the data accordingly. Examine chromatograms for interfering peaks and integrated peak areas. Re analyze sample if volume allows. Generate on Non-Conformance Memo.

4. In the absence of project specific requirements, immediately after the flawed CCV analyze another CCV or re-analyzed all samples set after the last acceptable CCV. Generate on Non-Conformance Memo.

5. In the absence of project specific requirements, check to be sure that there are no errors in the calculations. If errors are found, recalculate the data accordingly. Examine chromatograms for interfering peaks and integrated peak areas. If the program allows report the concentrations noting the surrogate recovery is most likely due to co-elution with target compounds. Some samples may require dilution in order to bring one or more target analytes within the calibration range or to overcome significant interferences with some analytes. Re analyze sample if volume allows. If the surrogate recoveries are available from a less-diluted or undiluted aliquot of the sample or sample extract, those recoveries may be used to demonstrate that the surrogates were within the in-house QC limits, and no further action is required. If, upon re-analysis the recovery is again not within limits, report the data as an "estimated concentration." If the recovery is within the limits in the re-analysis, provide the re-analysis data to the data user. Generate on Non-Conformance Memo.

6. In the absence of project specific requirements, the LCS and LCSD are spiked with the same analytes at the same concentrations as the matrix spike. When the results of the matrix spike analysis indicate a potential problem due to the sample matrix itself, the LCS results are used to verify that the laboratory can perform the analysis in a clean matrix. If the LCS and LCSD recoveries do not meet those limits as required in the method or program, the samples shall be re-analyzed. If sample volume does not allow re-analysis the entire prep/analytical batch of samples shall be flagged to reflect the deficiencies. Generate on Non-Conformance Memo.

7. In the absence of project specific requirements, when the results of the matrix spike analysis indicate a potential problem due to the sample matrix itself, the LCS results are used to verify that the laboratory can perform the analysis in a clean matrix. If sample volume allows re-analysis, it shall be performed to verify the matrix effect. When sample volume does not re-analysis, the parent sample and samples from that project site shall be flagged to reflect the deficiencies. Generate a Non- Conformance Memo.

Table L
ANALYST/SUPERVISOR DATA ASSESSMENT

Analytical Method Acceptance Criteria⇨ Data Assessment Measure ⇩	**Method Citation SW If these conditions are not achieved ⇒**	**Method Citation EPA If these conditions are not achieved ⇒**	**Method Citation EPA If these conditions are not achieved ⇒**
Initial Calibration	• 1	• 1	•
Initial Calibration Verification	• 1	• 1	•
Calibration Blank	• 1	• 1	•
Method Blank	• 2	• 2	•
Internal Standard Area	• 3	• 3	•
Continuing Calibration Verifications	• 4	• 4	•
Surrogate Recovery	• 5	• 5	•
Accuracy & Precision Laboratory Control Spikes	• 6	• 6	•
Accuracy & Precision Matrix Spikes	• 7	• 7	•

1. Check instrumentation/equipment condition; enter maintenance in instrument log; perform another initial calibration. No data can be reported. Generate on Non-Conformance Memo.

2. In the absence of project specific requirements, sample detects greater than 20 times the method blank contamination level are reported with those not exceeding 20 times re-analyzed or qualified. Generate on Non-Conformance Memo.

3. In the absence of project specific requirements, check to be sure that there are no errors in the calculations. If errors are found, recalculate the data accordingly. Examine chromatograms for interfering peaks and integrated peak areas. Re analyze sample if volume allows. Generate on Non-Conformance Memo.

4. In the absence of project specific requirements, immediately after the flawed CCV analyze another CCV or re-analyzed all samples set after the last acceptable CCV. Generate on Non Conformance Memo.

5. In the absence of project specific requirements, check to be sure that there are no errors in the calculations. If errors are found, recalculate the data accordingly. Examine chromatograms for interfering peaks and integrated peak areas. If the program allows report the concentrations noting the surrogate recovery is most likely due to co-elution with target compounds. Some samples may require dilution in order to bring one or more target analytes within the calibration range or to overcome significant interferences with some analytes. Re analyze sample if volume allows. If the surrogate recoveries are available from a less-diluted or undiluted aliquot of the sample or sample extract, those recoveries may be used to demonstrate that the surrogates were within the in-house QC limits, and no further action is required. If, upon re-analysis the recovery is again not within limits, report the data as an "estimated concentration." If the recovery is within the limits in the re-analysis, provide the re-analysis data to the data user. Generate on Non-Conformance Memo.

6. In the absence of project specific requirements, the LCS and LCSD are spiked with the same analytes at the same concentrations as the matrix spike. When the results of the matrix spike analysis indicate a potential problem due to the sample matrix itself, the LCS results are used to verify that the laboratory can perform the analysis in a clean matrix. If the LCS and LCSD recoveries do not meet those limits as required in the method or program, the samples shall be re-analyzed. If sample volume does not allow re-analysis the entire prep/analytical batch of samples shall be flagged to reflect the deficiencies. Generate on Non-Conformance Memo.

7. In the absence of project specific requirements, when the results of the matrix spike analysis indicate a potential problem due to the sample matrix itself, the LCS results are used to verify that the laboratory can perform the analysis in a clean matrix. If sample volume allows re-analysis, it shall be performed to verify the matrix effect. When sample volume does not re-analysis, the parent sample and samples from that project site shall be flagged to reflect the deficiencies. Generate a Non- Conformance Memo.

Table M
REPORT REVIEWER/PROJECT MANAGEMENT DATA ASSESSMENT

Analytical Method Acceptance Criteria⇨ Data Assessment Measure ⇩	**Method Citation SW If these conditions are not achieved ⇒**	**Method Citation EPA If these conditions are not achieved ⇒**	**Method Citation EPA If these conditions are not achieved ⇒**
Holding Time Compliance	• 1	• 1	•
Sample Receipt Conditions and Preservation	• 2	• 2	•
Method Citation	• 3	• 3	•
Reasonableness: Field Duplicate	• 4	• 4	•
Reasonableness: Analytical Comments Qualifications	• 5	• 5	•

1. Flag results with an H and the number of days past hold in parenthesis e.g. H(5).

2. In prose describe the receipt conditions as they relate to the acceptance criteria list in Table G

3. Compare to regulatory program in QAPjP or Chain of Custody.

4. Are duplicate with 40% agreement. If not, internally flag for review?

5. Do related suites of tests agree or are reasonable?

Table N
CORRECTIVE ACTIONS

Analytical Method Acceptance Criteria⇒ Quality Control Measure ⇓	**Method Citation SW If these conditions are not achieved ⇒**	**Corrective Action (Key below)**	**Method Citation EPA If these conditions are not achieved ⇒**	**Corrective Action (Key below)**
Initial Calibration		• 1	•	• 1
Method Blank		• 2	•	• 2
Continuing Calibration Verification		• 3	•	• 3

1. Check instrumentation/equipment condition; enter maintenance in instrument log; perform another initial calibration

2. Samples detects greater than 20 times the method blank contamination level are reported with those not exceeding 20 times re-analyzed or qualified.

3. Immediately after the flawed CCV analyze another CCV or re-analyzed all samples set after the last acceptable CCV.

Table O
CONTINGENCIES FOR HANDLING OUT-OF-CONTROL OR UNACCEPTABLE DATA

Analytical Method Acceptance Criteria⇨ Quality Control Measure ⇩	**Method Citation SW If these conditions are not achieved ⇒**	**Corrective Action (Key below)**	**Method Citation EPA If these conditions are not achieved ⇒**	**Corrective Action (Key below)**
Matrix Spike and Duplicate	•	• 1	•	• 1
Internal Standards	•	• 2	•	• 2
Surrogate Standards	•	• 2	•	• 2

1. If Lab Control Spikes pass acceptance criteria, flag parent sample to indicate matrix effects.

2. When sample volume is available reanalyze the samples. If the second analysis indicates the same internal standard recoveries add analytical comments and effects on data.

TABLE P:
Quantitation Ions

Compound	Retention Time (min)	Primary Ion	Secondary Ion(s)
N-Nitrosodimethylamine	3.12	42	74
Pyridine	3.13	79	52,53
2-Fluorophenol (surr.)	4.26	112	65
Benzaldehyde	5.14	105	106,77
Phenol-d(5) (surr.)	5.26	99	71
Phenol	5.27	94	66,65
Aniline	5.31	93	66,39
bis(2-Chloroethyl) ether	5.36	63	93,95
2-Chlorophenol-d(4) (surr.)	5.41	132	134,68
2-Chlorophenol	5.42	128	64,130
1,3-Dichlorobenzene	5.58	146	148,111
1,4-Dichlorobenzene-d(4)(I.S.)	5.62	152	150,115
1,4-Dichlorobenzene	5.65	146	148,111
1,2-Dichlorobenzene-d(4) (surr.)	5.86	152	150,115
Benzyl alcohol	5.85	108	79,107
1,2-Dichlorobenzene	5.88	146	148,111
2-Methylphenol	6.00	108	107,79
2,2-oxybis(1-Chloropropane)	6.03	45	77,121
Acetophenone	6.20	105	77,120
4-Methylphenol	6.20	108	107,79
N-Nitroso-di-n-propylamine	6.23	70	43,101
Hexachloroethane	6.27	117	201,199
Nitrobenzene-d(5) (surr.)	6.40	82	128,54
Nitrobenzene	6.43	77	123,65
Isophorone	6.74	82	95,138
2-Nitrophenol	6.86	139	109,65
2,4-Dimethylphenol	6.92	107	122,121
bis(2-Chloroethoxy) methane	7.06	93	95,123
2,4-Dichlorophenol	7.20	162	164,98
Benzoic acid	7.12	122	105,77
1,2,4-Trichlorobenzene	7.30	180	182,145
Naphthalene-d(8) (I.S.)	7.35	136	68
Naphthalene	7.39	128	129
4-Chloroaniline	7.53	127	129
Hexachlorobutadiene	7.64	225	223,227
Caprolactum	8.08	55	56,113
4-Chloro-3-methylphenol	8.28	107	144,142
2-Methylnaphthalene	8.42	142	141
1-Methylnaphthalene	8.58	142	141
Hexachlorocyclopentadiene	8.76	237	235,272
2,4,6-Trichlorophenol	8.92	196	198,200
2,4,5-Trichlorophenol	8.98	196	198,200
2-Fluorobiphenyl (surr.)	9.03	172	171
2-Chloronapthalene	9.17	162	127,164

Compound	Retention Time (min)	Primary Ion	Secondary Ion(s)
Biphenyl	9.16	154	153,152
2-Nitroaniline	9.43	65	92,138
Dimethylphthalate	9.76	163	194,164
Acenaphthylene	9.82	152	153
2,6-Dinitrotoluene	9.89	165	63,89
Acenaphthene-d(10) (I.S.)	10.07	164	162,160
3-Nitroaniline	10.10	138	108,92
Acenaphthene	10.11	154	153,152
2,4-Dinitrophenol	10.27	184	63,154
Dibenzofuran	10.36	168	139
4-Nitrophenol	10.40	139	109,65
2,4-Dinitrotoluene	10.50	165	63,89
Diethylphthalate	10.88	149	177,150
Fluorene	10.92	166	165,167
4-Chlorophenyl phenyl ether	10.93	204	206,141
4-Nitroaniline	11.09	138	108,92
4,6-Dinitro-2-methylphenol	11.14	198	51,105
N-Nitrosodiphenylamine	11.15	169	168,167
Azobenzene	11.18	77	51,182
2,4,6-Tirbromophenol (surr.)	11.35	330	332,141
4-Bromophenyl phenyl ether	11.70	248	250,141
Hexachlorobenzene	11.92	284	142,249
Atrazine	12.08	200	215,58
Pentachlorophenol	12.24	266	264,268
Phenanthrene-d(10) (I.S.)	12.40	188	94,80
Phenanthrene	12.44	178	179,176
Anthracene	12.51	178	176,179
Carbazole	12.81	167	
di-n-Butylphthalate	13.46	149	150,104

Compound	Retention Time (min)	Primary Ion	Secondary Ion(s)
Benzidine	14.66	184	92,185
Pyrene	14.71	202	200,203
Terphenyl-d(14) (surr.)	15.01	244	122,212
Butylbenzylphthalate	15.87	149	91,206
Benzo(a)anthracene	16.64	228	229,226
Chrysene-d(12) (I.S.)	16.67	240	120,236
3,3'-Dichlorobenzidine	16.68	252	254,126
Chrysene	16.72	228	226,229
bis(2-Ethylhexyl)phthalate	16.81	149	167,279
di-n-Octylphthalate	17.80	149	167,43
Benzo(b)fluoranthene	18.49	252	253,125
Benzo(k)fluoranthene	18.53	252	253,125
Benzo(a)pyrene	19.16	252	253,125
Perylene-d(12) (I.S.)	19.29	264	260,265
Indeno(1,2,3-cd)pyrene	22.18	276	138,277
Dibenz(a,h)anthracene	22.27	278	139,279
Benzo(g,h,i)perylene	23.05	276	138,277

I.S. = internal standard.
surr. = surrogate.
(a) Estimated retention times.

TABLE Q:
DFTPP Key Ions /Ion Abundance Criteria And CCC/SPCC Compounds

Mass	Ion Abundance Criteria
51	30-80% of mass 198
68	< 2% of mass 69
70	< 2% of mass 69
127	25-75% of mass 198
197	< 1% of mass 198
198	Base peak, 100% relative abundance
199	5-9% of mass 198
275	10-30% of mass 198
365	> 0.75% of mass 198
441	Present but less than mass 443
442	40-110% of mass 198
443	15-24% of mass 442

CCC Compounds

Base/Neutral Fraction	Acid Fraction
Acenaphthene	4-Chloro-3-methylphenol
1,4-Dichlorobenzene	2,4-Dichlorophenol
Hexachlorobutadiene	2-Nitrophenol
Diphenylamine	Phenol
Di-n-octyl phthalate	Pentachlorophenol
Fluoranthene	2,4,6-Trichlorophenol
Benzo(a)pyrene	

SPCC Compounds

N-nitroso-di-n-propylamine

Hexachlorocyclopentadiene

2,4-dinitrophenol

4-nitrophenol.

Table R
Data Qualifiers

B	Analyte is present in the method blank. Method blank criteria is evaluated to the laboratory method detection limit. Additionally, method blank acceptance may be based on project specific criteria or determined from analyte concentrations in the sample and are evaluated on a sample by sample basis.
C	Elevated detection limit (see Sample Narrative).
D	Analyte value from diluted analysis.
E	Analyte concentration exceeds calibration range (see Sample Narrative).
F	Surrogate results outside control criteria or not available due to sample dilution.
H(n)	Extraction or analysis performed n days past holding time.
J	Qualitative evidence of analyte present: concentration detected is greater than the method detection limit but less than the reporting limit.
K	Detection limit may be elevated due to the presence of an unrequested analyte (see Sample Narrative).
N	Spiked sample recovery not within control limits.
P	The relative percent difference between the two GC columns for detected concentrations was greater than 40%.
Q	The analyte has been detected between the limit of detection (LOD) and limit of quantitation (LOQ). The results are qualified due to the uncertainty of analyte concentrations within this range.
U	The analyte was not detected above the reporting limit.
W	Sample received with headspace.
X	See Sample Narrative.
&	Laboratory Control Spike recovery not within control limits.
*	Duplicate analyses not within control limits.

Standard Operating Procedure

TITLE: Aqueous Sample Preparation for the Analysis of Semivolatile Base/Neutral/Acids Full Scan

DEPARTMENT: Semivolatile Organics

REFERENCES: SW-846 3500B Organic Extraction and Preparation
Revision 2, December 1996
SW-846 3510C Separatory Funnel Liquid-Liquid Extraction
Revision 3,December 1996
U.S. EPA 40 CFR Part 136, "Guidelines Establishing Test Procedures for the Analysis of Pollutants Under the Clean Water Act; Final Rule and nterim Final Rule and Proposed Rule," October 26, 1984.

PROCEDURE SUMMARY: A measured volume of sample, usually 1 liter, at a specified pH, is serially extracted with methylene chloride using a separatory funnel. The extract is concentrated to specified volume.

REVIEWED BY: _____ 4/28/00
Nickolas J. Severin
Semivolatile Section Supervisor
Date

_____ 4/28/2000
Michael C. Suha
Quality Control Officer
Date

APPROVED BY: _____ 4/28/2000
Nils K. Melberg
Laboratory Manager**
Date

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SAFETY PRECAUTIONS:

- The toxicity or carcinogenicity of chemicals used in this method have not been precisely defined; each chemical should be treated as a potential health hazard, and exposure to these chemicals should be minimized. Each analyst is responsible for maintaining awareness of OSHA regulations regarding safe handling of chemicals used in this method. Additional references to laboratory safety are available for the information of the analyst.

1.0 SCOPE AND APPLICATION

1.1 This method describes a procedure for isolating organic compounds from aqueous samples. The method also describes concentration techniques suitable for preparing the extract for the appropriate determinative methods described in Section 4.3 of Chapter Four.

1.2 This method is applicable to the isolation and concentration of water insoluble and slightly water-soluble organics in preparation for a variety of chromatographic procedures.

3.0 INTERFERENCES

3.1 Raw GC/MS data from all blanks, samples, and spikes must be evaluated for interferences. Determine if the source of interference is in the preparation and/or cleanup of the samples and take corrective action to eliminate the problem.

3.2 Contamination by carryover can occur whenever high-concentration and low-concentration samples are sequentially analyzed. To reduce carryover, the sample syringe must be rinsed out between samples with solvent. Whenever an unusually concentrated sample is encountered, it should be followed by the analysis of solvent to check for cross contamination.

4.0 APPARATUS AND MATERIALS

4.1 Lab-Line Separatory funnel - 2 liter, with Teflon stopcock.

4.2 Drying column - glass funnel with glass wool plug; filled with baked sodium sulfate.

4.3 Vials - 2 mL, amber glass with Teflon lined crimp tops.

4.4 pH indicator paper - pH range including the desired extraction pH.

4.5 Syringe - 50 ul, 100ul, 1.0 ml.

4.6 Graduated cylinder - 1 liter.

4.7 Zymark - TurbovapII set at 30 C

4.8 200 ml Turbovap tubes with 1.0 ml stems.

4.9 Lab-Line Extraction Mixer.

5.0 REAGENTS

5.1 Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available. Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination. Reagents should be stored in glass to prevent the leaching of contaminants from plastic containers.

5.2 Organic-free reagent water - All references to water in this method refer to organic-free reagent water, as defined in Chapter One.

5.3 Sodium hydroxide solution (10N), NaOH. Dissolve 80 g NaOH in organic free reagent water and dilute to 200 mL.

5.4 Sodium sulfate (Mallinckrodt AR), Na(2)SO(4). Purify by heating at 400-C for 4 hours in a shallow tray

5.5 Sulfuric acid solution - 1:1 slowly add 100mls H2SO4 to 100mls reagent water

5.6 Extraction/exchange solvents

5.6.1 Methylene chloride, CH(2)Cl(2) - Pesticide quality or equivalent.

5.7 Surrogates - Restek - 2-Fluorophenol, Phenol-d5, 2-Chlorophenol-d4, 1,2-Dichlorobenzene-d4, Nitrobenzene-d5, 2-Fluorobiphenyl, 2,4,6- Tribromophenol, Terphenyl-d14. See Appendix A for more details.

5.8 Matrix Spiking Solution - 200 ug/ml of 8270 list and surrogates in Methylene Chloride - Restek. See Appendix B for more details.

6.0 SAMPLE COLLECTION, PRESERVATION, AND HANDLING

6.1 Samples must be collected in nonpreserved 1.0 L amber jars with teflon lined septum caps. Samples must be transported on ice and stored between 0.0 & 4.0 C. Extraction must take place within 7 days of collection.

7.0 PROCEDURE

7.1 Using a 1 liter graduated cylinder, measure 1 liter (nominal) of sample and transfer it quantitatively to the separatory funnel. Mark water level on the sample jar if it is less than 1.0 liter. Label separatory funnel and turbovap tube with the sample ID.

If high concentrations are anticipated, a smaller volume may be used and then diluted with organic-free reagent water to 1 liter. Add 1.0ml of the surrogate standard (See details in Appendix A) to all samples, spikes, and blanks.

For the matrix spikes samples, add 250 ul of the matrix spiking standard (See details in Appendix B). For base/neutral-acid analysis, the amount added of the matrix spiking compounds should result in a final concentration of 50 ng/uL of each base/neutral analytes and 50 ng/uL of each acid analytes in the extract to be analyzed (assuming a 1 uL injection).

Add 1.0 ml of laboratory control spike solution (See details in Appendix C) to 1.0 L reagent water.

If Method 3640, Gel-Permeation Cleanup, is to be used, add twice the volume of surrogates and matrix spiking compounds since half the extract is lost due to loading of the GPC column.

7.2 Add 3.0ml sulfuric acid solution. Check the pH of the sample withbwide-range pH paper and, if necessary, adjust the pH to <2.

7.3 Rinse sample jar with 60 mL of Methylene Chloride and add this to the separatory funnel. Record the Methylene Chloride lot # on the extraction log.

7.4 Seal the separatory funnel and set the Lab-Line extraction mixer to shake for 3.0 minutes at a speed of 30 cycles a minute.

NOTE: Methylene chloride creates excessive pressure very rapidly; but the Lab-Line separatory funnels automatically vent when the pressure exceeds 1.0 psi.

7.5 Allow the organic layer to separate from the water phase for a minimum of 10 minutes. Pass the methylene chloride phase(bottom layer) through the drying column into a methylene chloride rinsed 200 ml Turbovapvap tube.

If the emulsion interface between layers is more than one-third the size of the solvent layer, the analyst must add the emulsion to approximately 50 grams of sodium sulfate in a separate beaker.

After stirring, tranfer this solution through the drying column and into the 200 ml Turbovap tube.

Rinse the sodium sulfate in the beaker with 40 ml of methylene chloride and transfer this solution through the drying column.

7.6 Repeat the extraction two more times using fresh portions of solvent - Sections 7.3 through 7.5. Combine the three solvent extracts.

Start concentrating the extract in the turbovapII while the basic extraction is being performed.

7.7 Add 8.0ml or more of 10.0 N NaOH to adjust the pH to >11. Check the pH of the sample with wide-range pH paper and, if necessary, adjust the pH to >11.

Add 60 mls of methylene chloride repeat steps in Sections 7.4 thru 7.6

7.8 If performing GC/MS analysis (Method 8270), the acid/neutral and base extracts may be combined prior to concentration.

However, in some situations, separate concentration and analysis of the acid/neutral and base extracts may be preferable (e.g., if for regulatory purposes the presence or absence of specific acid/neutral or base compounds at low concentrations must be determined, separate extract analyses may be warranted).

7.9 Perform the solvent concentration using the TurbovapII set at 30C and 11 psi.

7.10 The sample is taken down to .75 ml and the bottom 1/3 of the tube is rinsed and brought up to a 1.0 ml final volume with methylene chloride.

7.11 The sample is transferred to 2.0ml crimp top vials and 10 ul of internal standard is added if the sample is to be analzed without dilution.

7.12 The extract may now be analyzed for the target analytes using the appropriate determinative technique(s).

If analysis of the extract will not be performed immediately, stopper the concentrator tube and store refrigerated.

If the extract will be stored longer than 2 days it should be transferred to a vial with a Teflon lined screw-cap or crimp top, and labeled appropriately.

8.0 QUALITY CONTROL

8.1 Any reagent blanks or matrix spike samples should be subjected to exactly the same analytical procedures as those used on actual samples.

8.2 An extraction blank, matrix spike. matrix spike duplicate and laboratory control spike is run with every batch of 20 samples or with each extraction set.

If a matrix spike sample is not provided, a laboratory control spike duplicate is run instead.

9.0 DEVIATIONS FROM THE METHOD

9.1 Turbovapll concentrators are used for solvent concentration instead of K-D apparatus.

9.2 The matrix spike sample are spiked with the entire 8270 list not just the spike compounds listed in the method.

9.3 Lab-line automated extractors are used for shaking the samples.

9.4 If a EPA 625 is to referenced, the QC frequecies listed in that method are followed.

Appendix A

SPIKING STANDARDS FOR BASE/NEUTRAL/ACIDS

SURROGATE SPIKING SOLUTION

The mixture contains the following components at:

75 µg/mL	50 µg/mL
2-Fluorophenol	Terphenyl-d14
2,4,6-Tribromophenol	2-Flourobiphenyl
Phenol-d5	1,2-Dichlorobenzene-d4
2-Chlorophenol-d4	Nitrobenzene-d5

Appendix B

SPIKING STANDARDS FOR BASE/NEUTRAL/ACIDS

MATRIX SPIKING SOLUTION

The mixture contains the following components at 200 µg/mL:

Phenol	Anthracene
2-Chlorophenol	N-nitrosodimethylamine
Benzyl alcohol	bis(2-Chloroethyl)ether
2-Methylphenol	1,3-Dichlorobenzene
4-Methylphenol	1,4-Dichlorobenzene
2,4-Dimethylphenol	1,2-Dichlorobenzene
Benzoic acid	2,2'-oxybis(1-Chloropropane)
2,4-Dichlorophenol	N-nitroso-di-n-propylamine
4-Chloroaniline	Hexachlorethane
4-Chloro-3-methylphenol	Nitrobenzene
2-Nitrophenol	Isophorone
2,4,6-Trichlorophenol	Carbazole
2,4,5-Trichlorophenol	bis(2-Chloroethoxy)methane
2-Nitroaniline	1,2,4-Trichlorobenzene
3-Nitroaniline	Napthalene
2,4-Dinitrophenol	Hexachlorobutadiene
4-Nitrophenol	2-Chloronaphthalene
Dibenzofuran	Dimethylphthalate
4-Nitroaniline	Acenaphthylene
1,2-Diphenylhydrazine	2,6-Dinitrotoluene
Pentachlorophenol	Acenaphthene
2,4-Dinitrotoluene	Diethylphthalate
3,3'-Dichlorobenzidine	4-Chlorophenyl-phenylether
Benzo(a)pyrene	Fluorene
Indeno(1,2,3-cd)pyrene	4,6-Dinitro-2-methylphenol
Dibenz(a,h)anthracene	N-nitrosodiphenylamine
Benzo(g,h,i)perylene	4-Bromophenyl-phenylether
2-Methylnaphthalene	Fluoranthene
Hexachlorobenzene	Benzo(a)anthracene
Butylbenzylphthalate	Chrysene
Di-n-butylphthalate	Pyrene
bis(2-Ethylhexyl)phthalate	Benzo(b)fluoranthene
Di-n-octylphthalate	Benzo(k)fluoranthene
Phenanthrene	

Appendix C

SPIKING STANDARDS FOR BASE/NEUTRAL/ACIDS

LABORATORY CONTROL SPIKING SOLUTION

The mixture contains the following components at:

150 µg/mL	100 µg/mL
4-Chloro-3-methylphenol	Acenaphthene
2-Chlorophenol	1,4-Dichlorobenzene
4-Nitrophenol	2,4-Dinitrotoluene
Pentachlorophenol	n-Nitroso-di-n-propylamine
Phenol	Pyrene
	1,2,4-Trichlorobenzene

TITLE: Volatile Organic Compounds By Gas Chromatography/Mass Spectrometry;

MATRICES:

This SOP pertains to Aqueous, Non-Aqueous Liquids, Chemical Wastes, Air, and Solid Matrices.

DETECTION LIMITS:

The Reporting Limits used in the absence of Project or State Specific Required Detection are found in Appendix B Table A.

The method detection limits were obtained by multiplying 3.143 by the standard deviation of seven spikes. The spikes went though the same preparation procedure and were spiked at a concentration estimated to be 10 times greater than the MDL. Cited from 40 CFR part 136.

1.0 **SCOPE AND APPLICATION:**

1.1 Method 8260B is used to determine volatile organic compounds in a variety of solid waste matrices. This method is applicable to nearly all types of samples, regardless of water content, including ground water, aqueous sludges, caustic liquors, acid liquors, waste solvents, oily wastes, mousses, tars, fibrous wastes, polymeric emulsions, filter cakes, spent carbons, spent catalysts, soils, and sediments. The following list of compounds can be determined but not limited to if the compounds can be demonstrated to perform by this method:

1.2 Method detection limits (MDLs) are compound dependent and vary with purging efficiency and concentration. The applicable concentration range of this method is compound and instrument dependent but is approximately 0.1 to 200 µg/L. Analytes that are inefficiently purged from water will not be detected when present at low concentrations, but they can be measured with acceptable accuracy and precision when present in sufficient amounts. Determination of some structural isomers (i.e., xylenes) may be hampered by coelution. These are presented as a summation of the isomers.

1.3 The estimated quantitation limit (EQL) of individual compound is approximately 5 µg/kg (wet weight) for soil/sediment samples, and between 1 and 2 ug/L for ground water. EQLs will be proportionately higher for sample extracts and samples that require dilution or reduced sample size to avoid saturation of the detector Soil extraction EQLs are 50 ug/kg (25 ug/kg in the State of Wisconsin and Minnesota). The Practical Quantitation Limit (PQL) of this method for individual maybe chosen as the lowest point in the calibration curve.

1.4 This method is restricted for use by, or under the supervision of, analysts experienced in the use of gas chromatographs for measurement of purgeable organics at low µg/L concentrations and skilled in the interpretation of mass spectra. Each analyst must demonstrate the ability to generate acceptable results with this method.

2.0 SUMMARY OF THE TEST METHOD:

2.1 The volatile compounds are introduced into the gas chromatograph by the purge-and-trap method. The analytes are introduced directly to a to a micro-bore capillary column for analysis. The column is temperature-programmed to separate the analytes, which are then detected with a mass spectrometer (MS) interfaced to the gas chromatograph (GC).

2.2 Analytes eluted from the capillary column are introduced into the mass spectrometer via a direct connection. Micro-bore capillary columns may be directly interfaced to the ion source. Identification of target analytes is accomplished by comparing their mass spectra with the electron impact (or electron impact like) spectra of authentic standards. Quantitation is accomplished by comparing the response of a major quantitation ions relative to an internal standard using a six point calibration curve.

2.3 This method includes specific calibration and quality control steps that supersede the general requirements provided in authoritative sources.

3.0 DEFINITIONS:

3.1 Definition of terms used throughout this document are defined in Appendix A.

4.0 INTERFERENCES:

4.1 Major contaminant sources are volatile materials in the laboratory and impurities in the inert purging gas and in the sorbent trap. The use of non polytetrafluoroethylene (PTFE) thread sealants, plastic tubing, or flow controllers with rubber components should be avoided, since such materials outgas organic compounds which will be concentrated in the trap during the purge operation. Analyses of calibration and reagent blanks provide information about the presence of contaminants. When potential interfering peaks are noted in blanks, the analyst should change the purge gas source and regenerate the molecular sieve purge gas filter.

4.2 Subtracting blank values from sample results are not permitted. If reporting values without correcting for the blank results in what the laboratory feels is a false positive result for a sample, the analyst should fully explained this in text accompanying the uncorrected data.

4.3 Contamination may occur when a sample containing low concentrations of volatile organic compounds is analyzed immediately after a sample containing high concentrations of volatile organic compounds. A technique to prevent this problem is to rinse the purging apparatus and sample syringes with portions of organic free reagent water between samples. After the analysis of a sample containing high concentrations of volatile organic compounds, one or more blanks should be analyzed to check for cross-contamination. A low concentration sample (i.e. not detected) following the injection of a high concentration sample is also used to check for cross contamination.

4.4 For samples containing large amounts of water-soluble materials, suspended solids, high boiling compounds, or high concentrations of compounds being determined, it may be necessary to wash the purging device with a soap solution, rinse it with organic free reagent water, and then dry the purging device in an oven at 105°C. In extreme situations, the entire purge-and-trap device may require dismantling and cleaning. Screening of the samples prior to purge-and-trap GC/MS analysis is highly recommended to prevent contamination of the system.

Note: This is especially true for soil and waste samples. Screening may be accomplished with an automated headspace technique or by Hexadecane Extraction.

4.5 Special precautions must be taken to analyze for methylene chloride. The analytical and sample storage area should be isolated from atmospheric sources of methylene chloride. Otherwise, random background levels will result. Since methylene chloride will permeate through PTFE tubing, all gas chromatography carrier gas lines and purge gas plumbing should be constructed from stainless steel or copper tubing. Laboratory clothing worn by the analyst should be clean, since clothing previously exposed to methylene chloride fumes during extraction procedures can contribute to sample contamination.

4.6 Samples can be contaminated by diffusion of volatile organics (particularly methylene chloride and fluorocarbons) through the septum seal of the sample container into the sample during shipment and storage. A trip blank prepared from organic free reagent water and carried through the sampling, handling, and storage protocols can serve as a check on such contamination.

Note: Use of sensitive mass spectrometers to achieve lower detection level will increase the potential to detect laboratory contaminants as interferences.

5.0 **SAFETY:**

5.1 Safety is everyone's business at En Chem, Inc. and everyone is responsible for assisting in reducing unsafe and unhealthy working conditions or potential hazards. The company provides you with a safe place to work, but we need your cooperation to keep it safe. When you see something that does not look safe, or you see someone working in an unsafe manner, mention it to your Supervisor.

5.2 All samples should be treated as hazardous. Safety glasses, gloves, and lab coats are to be worn. The toxicity or carcinogenicity of each reagent used in this method has not been precisely defined. However, each chemical compound should be treated as a potential health hazard. Exposure to these chemicals must be reduced to the lowest

possible level by a safe technique. A reference file of material safety data sheets should also be made available to all personnel involved in the chemical analysis.

5.3 Required Safety Equipment is listed in Appendix B Table B

6.0 EQUIPMENT AND SUPPLIES:

6.1 See Appendix B Table C and Table D for a summary.

7.0 REAGENTS AND STANDARDS:

7.1 See Appendix B Table E and Table F for a summary.

8.0 SAMPLE COLLECTION, PRESERVATION, SHIPMENT AND STORAGE:

8.1 Aqueous samples should be collected in triplicate without agitation and without headspace in contaminant-free 40 ml glass VOC vials with Teflon-lined septa in the caps. The Teflon liner must contact the sample. Samples must be preserved with 500 ul of Hydrochloric Acid (HCl) at the time of collection, (acid should be added to the vial prior to adding the sample).

> Note: En Chem uses pre-cleaned, level 3 certified, 40 ml VOC vials purchased from QEC. These vials contain 0.5 ml of 18 % HCL.

> Note: If targeted compounds breakdown with the addition of HCl , samples must be collected unpreserved. Unpreserved samples have a 7 day holding time.

8.2 The samples will need to be cooled to 4° C after collection. Aqueous samples will be stored in refrigerators at En Chem and must be held at 4° C (+/- 2C). Preserved samples have a 14 day holding time or as specified by the Accrediting Authority.

8.3 One trip blank is required per sampling event and site.

8.4 Solid samples for SW846 5035 must be collected with the ENCORE sampler. A sufficient number of samplers should be collected to provide for backup analyses in the event of breakage or instrument malfunctions and to allow for screening. One vial should be collected for dry weight determination.

8.5 Soil samples for volatile analysis that are preserved with the sodium bisulfate preservative solution are to be cooled to approximately 4° C, packed in appropriate containers, and shipped to the laboratory on ice. These are to be analyzed within 14 days of sample collection. Samples receiving no preservation other than cooling to 4°C must be shipped to the laboratory the same day that they are collected. Once in the laboratory, the samples must be analyzed or frozen within 48 hours of sample collection. All samples need to be analyzed as soon as practical, but within the designated holding time of 14 days from sample collection for preserved samples or 48 hours for unpreserved samples.

> Note: Unpreserved ENCORE samplers will be frozen by En Chem personnel upon receipt to the lab or prepared for analysis immediately.

8.6 Solid samples for SW846 5030 are routinely collected in a 2 oz. jar. One vial should be collected for dry weight determination.

Note: Zero headspace jar sampling can only be used and is limited to projects where it is approved by the Accrediting Authority.

8.7 Samples need to be shipped to the lab as soon as the samples are collected.

8.8 Sample temperature must be determined upon receipt to the lab.

Note: Sample temperature is recorded by use of a temperature blank. The temperature must be between 4° C (+/- 2C) unless superceded by the accrediting authority. For certain authorities recording sample temperature as "received on ice (ROI)" is acceptable

8.9 Samples will be stored at En Chem at 4° C (+/- 2C).

8.10 All preserved samples must be analyzed within 14 days from the date of collection unless project or authority specific holding times differ.

8.11 See Appendix B Table G for a summary.

9.0 **QUALITY CONTROL:**

9.1 Quality control procedures to ensure the proper production operation in all of the various sample preparation and/or sample introduction techniques. The laboratory must also maintain records to document the quality of the data generated.

9.2 An initial demonstration of capability (IDOC) is performed by analysts prior to analyzing samples

9.3 To demonstrate capability, analyze at least four replicate aliquots of the well-mixed reference samples by the same procedures used to analyze actual samples.

9.4 Calculate the average recovery (x) in µg/L, and the standard deviation of the recovery (s) in µg/L, for each analyte of interest using the four results.

9.5 The data generated is compared to the single-laboratory performance in the authoritative sources . Certain analytes may be outside the limits, however, the majority should be within the acceptance limits.

Note. Acceptable limits are set by the Accrediting Authority. South Carolina IDOC and LCS limits are and 70-130%.

9.6 Analytes that are outside source IDOC limits are reviewed as outlined below and IDOC are repeated.

9.7 Locate and correct the source of the problem. Document the changes made in the maintenance log.

9.8 Repeated failure, however, will confirm a general problem with the measurement system. If this occurs, locate and correct the source of the problem. Document the changes made in the maintenance log.

9.9 Repeat the test for all compounds of interest until resolved.

> Note: In-house acceptance limits may be generated based on the purging efficiency and other properties of the compounds as outlined in authoritative sources. This is not done to justify poor recoveries (i.e. <50% recovery).

9.10 See Appendix B Table K for a summary of the method IDOC.

9.11 MDL studies are performed yearly. Detection, quantitation and reporting limits are set using the data generated from these studies. The studies conform to the processes as described Chapter One of SW846 and CFR 40 part 136.

9.12 See Appendix B Table A for a summary of detection limits.

9.13 Reference samples are purchased from a NIST certified vendor. Results of these studies are made available to analysts, management and accrediting authorities.

9.14 Retention time windows are crucial to the identification of target compounds. Retention time windows are established to compensate for minor shifts that occur as a result of sample loading and normal chromatographic variability. Establishment of the width of this window is of considerable importance. A window that is "tight" may lead in the reporting of both false negative and false positive results. Re- analysis occurs in this situation due to the non-identification of surrogate and spike components in the analysis. "Wide" retention times lead to false positive results that cannot be confirmed upon re-analysis.

9.15 Before establishing retention time windows, make sure that the chromatographic system is operating reliably and that the system conditions have been optimized for the target analytes and surrogates in the sample matrix to be analyzed. Make three injections of all single component standard mixtures over the course of a 72-hour period.

9.16 Record the retention time for each single component analyte and surrogate to three decimal places (e.g., 0.007). Calculate the mean and standard deviation of the three absolute retention times for each single component analyte and surrogate

9.17 If the standard deviation of the retention times for a target compound is much smaller than the default deviation (i.e., no difference between the absolute retention times), then the laboratory may use a default standard deviation of 0.01 minutes.

9.18 En Chem has in place procedures for documenting the effect of the matrix on method performance (precision and accuracy). Development of limits is done by randomly choosing points by matrix from the previous year. When new compounds are added for analysis, default limits are utilized until new limits can be established. The average percent recovery (p) and standard deviation (s) are calculated and the upper and lower warning and control limits are established by the following equations:

Warning Limits-

Upper Warning Limit (UWL) = p+2s
Lower Warning Limit (LWL) = p-2s

Control Limits-

Upper Control Limit (UCL) = p+3s
Lower Control Limit (LCL) = p-3s

Relative Percent Difference Limits -

%RPD = p'+3s'

where:

p = is the average relative percent difference
s = is the standard deviation of the relative percent difference.

NOTE: If %RPD control limits are calculated to be below 10%, the limit for that individual compound will be set at 10%. This limit allows for assessment of the data without compromising the known quality in a production setting. Most samples received are not for research.

9.19 *Continuous charting for precision and accuracy is done. Limits are set yearly for Laboratory* Control Spikes (LCS) and Matrix Spikes (MS) and surrogates.

9.20 Surrogate recoveries - The laboratory must evaluate surrogate recovery data from individual samples against the surrogate control limits developed by the laboratory. Development of limits is done by randomly choosing a minimum of 20 points by matrix from the previous year. The average percent recovery (p) and standard deviation (s) are calculated and the upper and lower control limits are established by the following equations:

Control Limits-

Upper Control Limit (UCL) = p+3s
Lower Control Limit (LCL) = p-3s

NOTE: Remember that even for a 95% confidence interval, 1out of every 20 observations is likely to fall outside of the control limits established.

9.21 For each analytical batch, not to exceed 20 environmental samples , a method blank, matrix spike and matrix spike duplicate/duplicate must be analyzed (the frequency of the spikes may be

different for different monitoring programs but not to exceed 1 per 20 samples). The blank and spiked samples must be carried through all stages of the sample preparation and measurement steps.

9.22 A Laboratory Control Sample (LCS) and Duplicate (LCSD) also known as a Blank Spike (BS) and Duplicate (BSD) pair is also included with each analytical batch. The pair consists of an aliquot of a clean (control) matrix similar to the sample matrix and of the same weight or volume. The pair is spiked with the same analytes at the same concentrations as the matrix spike. When the results of the matrix spike analysis indicate a potential problem due to the sample matrix itself, these results are used to verify that the instrument was in control.

9.23 See Appendix B Table H for a summary of frequency by method citation.

9.24 When results of sample spikes indicate atypical method performance, if possible the sample is reanalyzed to confirm matrix interferences.

> NOTE: The large number of analytes analyzed present a substantial probability that one or more will fail at least one of the acceptance criteria when all analytes of a given method are determined. Remember that even for a 95% confidence interval, 1 out of every 20 observations is likely to fall outside of the control limits established.

9.25 See Appendix B Tables listed below for limit summaries:

Surrogate Limits	Table K1
Matrix Spike Limits	Table K2

9.26 See Appendix B Tables listed below for summaries of review and corrective actions:

Analyst/Technician Data Assessment	Table L
Analyst/Supervisor Data Assessment	Table M
Report Reviewer/Project Management Data Assessment	Table N
Corrective Actions	Table O
Contingencies for Out of Control Data	Table P

10.0 **CALIBRATION AND STANDARDIZATION:**

10.1 Stock standards may be prepared from neat standards and/or prepared mixes can be purchased. A reference standard prepared from a neat standard is made up at a 10,000 ug/mL concentration as follows.

10.2 Place about 9.8 ml of methanol into a 10 ml ground-glass stoppered volumetric flask.

10.3 Allow the flask to stand unstoppered for a few minutes until all alcohol-wetted surfaces have dried and weigh to the nearest 0.1 mg.

10.4 Using a 100 µl gastight syringe, add about 20-25 drops of the neat standard to the flask to obtain about 100 mg of standard. Let the drops fall just above the surface of the methanol but do not let the syringe needle touch the sides of the flask neck.

10.5 Reweigh the flask, dilute to volume, stopper, then mix by inverting the flask several times.

10.6 Store without headspace in a 7 mL amber vial in a freezer.

10.7 All documentation regarding standard preparation must be entered into the standard preparation logbook. A number is assigned to each prepared standard solution and that number is entered in logs where a solution is used. The vial containing a prepared standard solution must be labeled in accordance with the En Chem policy, and include the assigned log number. A brief description of the standard must also appear on the label.

10.8 Secondary dilution standards - Using stock standard solutions, prepare in methanol, secondary dilution standards containing the compounds of interest, either singularly or mixed together. Secondary dilution standards must be stored without headspace and should be checked frequently for signs of degradation or evaporation, especially just prior to preparing calibration standards from them.

10.9 Stock solutions certified by the manufacturer are purchased. Open a new stock standard if comparison with check standards indicates a problem. Standards for the permanent gases are monitored frequently by comparison to the initial calibration curve. Fresh standards are prepared if this check exceeds a 20% drift. Standards for gases usually need to be replaced after one week or sooner if significant degradation is observed. Dichlorodifluoromethane and chloromethane will usually be the first compounds to evaporate from the standard.

Note: South Carolina requires use of a one week holding time for all secondary standards, including all standards that are diluted from stock standards.

10.10 Standards for the non-gases are monitored frequently by comparison to the initial calibration. Fresh standards are prepared if this check exceeds a 20% drift. Standards for non-gases usually need to be replaced after six months or as recommended by the standard manufacturer, unless the acceptability of the standard can be documented. Standards of reactive compounds such as 2 Chloroethylvinyl ether and Styrene may need to be prepared more frequently.

10.11 Holding times assigned by the manufacturer must be adhered to for stock standards.

10.12 Prepare working standards of the volatile target compounds at 100ug/mL and the surrogates at 250 ug/ml in P&T methanol.

10.13 Prepare an Internal Standard and an Internal Standard/Surrogate solution (IS/SS) at a concentration of 250 ug/mL in P&T methanol.

10.14 Record all data regarding preparation of each standard solution in the standard logbook as described above for stock standards. Label the working standard vials as described above for the stock standards.

10.15 All standards should be stored in a freezer at -15 to -20°C.

10.16 Surrogate standards - The surrogates are Toluene-d_8,4-Bromofluorobenzene,and Dibromofluoromethane. Other compounds may be used as surrogates, depending upon the analysis requirements. A stock surrogate solution will be purchased, 2500 ul of this will be brought up to 25 ml in methanol. Each sample undergoing analysis must be spiked with this secondary surrogate spiking solution that is at 50 ug/L prior to analysis.

10.17 Internal standards - The internal standards are Chlorobenzene-d_5,1,4-Difluorobenzene, 1,4-Dichlorobenzene-d_4, and Pentafluorobenzene. Other compounds may be used as internal standards as long as they have retention times similar to the compounds being detected by GC/MS. Internal standard stock will be purchased. It is recommended that the each internal standard compound (2500 uL of 2500ppm internal standard mixture up to 25 ml in meoh). Each sample undergoing analysis must be spiked with this secondary internal standard spiking solution that is at 50 ug/L prior to analysis.

10.18 4-Bromofluorobenzene (BFB) standard - A standard solution containing 50 ng/uL of BFB in methanol should be prepared from a purchased stock of a 2500 ug/ml certified standard.

10.19 Calibration standards There are two types of calibration standards used for this method: Initial Calibration Standards and Continuing Calibration Check Standards. Concentration levels are adjustable to the project plan submitted according to the action limits of the compounds of interest and preservation matching.

10.20 See Appendix B Table J for a summary of instructions to create these standards

10.21 See Appendix B Table I for a summary of frequency and acceptance criteria.

10.22 Each GC/MS system must be hardware tuned to meet the criteria in Table 1 for a 50 ng injection or purging of 4-Bromofluorobenzene (1-uL injection of the BFB standard). Analyses must not begin until these criteria are met.

Table 1

BFB (4-BROMOFLUOROBENZENE) MASS INTENSITY CRITERIA[a]

m/z	**Required Intensity (relatvie abundance)**
50	15 to 40 % of m/z 95
75	30 to 60 % of m/z 95
95	Base peak, 100 % relativew abundance
96	5 to 9 % of m/z 95
173	less than 2 % of m/z 174
174	Greater than 50 % of m/z 95
175	5 to 9 % of m/z 174
176	Greater than 95 % but less than 101 % of m/z 174
177	5 to 9 % of m/z 176

a. Alternate tuning criteria may be used, (e.g. CLP, Method 524.2, or project/Accrediting Authority, provided that method performance is not adversely affected.

NOTE: All subsequent standards, samples, MS/MSDs, LCSs, and blanks associated with a BFB analysis must use identical mass spectrometer instrument conditions.

10.23 Initial Calibration Standards should be prepared at a minimum of six different concentrations from the secondary dilution of stock standards or from a premixed certified solution. Prepare these solutions in organic free reagent water. At least one of the calibration standards should correspond to a sample concentration at or below that necessary to meet the EQL. The remaining standards should correspond to the range of concentrations found in typical samples but should not exceed the working range of the GC/MS system. Each standard should contain each analyte of interest in the samples submitted.

10.24 A set of at least six different calibration standards is necessary. Calibration must be performed using the sample introduction technique that will be used for samples. For example Method 5030B, the purging efficiency for 5 mL of water is greater than for 25 mL. Therefore, the development of the standard curve is done with whichever volume of sample analyzed.

10.25 A set of at least six calibration standards containing the targeted analytes is needed. One calibration standard should contain each analyte at a concentration approaching but greater than the method detection limit for that compound; the other calibration standards should contain analytes at concentrations that define the range of the method. Prepare a calibration standard in a 50 mL syringe by adding an appropriate volume of intermediate solutions to 50 mL organic free water. Immediately transfer the contents of the syringe to a 40 mL vial. For soil method 5035, 5 mL of this calibration standard can be transferred to teflon screw-cap 40 ml VOA vial with a stir bar. For a methanol calibration curve, add 1 mL of methanol to 49 mL of water in to a 50 mL syringe.

10.26 The internal standards selected permit most of the components of interest in a chromatogram to have retention times of 0.80 to 1.20 minutes, relative to one of the internal standards. Use the base peak ion from the specific internal standard as the primary ion for quantitation. If interferences are noted, use the next most intense ion as the quantitation ion.

10.27 See Appendix B Table R for a summary of Internal Standards chosen and their retention times.

10.28 Tabulate the area response of the characteristic ions against the concentration for each target analyte and each internal standard. Calculate response factors (RF) for each target analyte relative to one of the internal standards. The internal standard selected for the calculation of the RF for a target analyte should be the internal standard that has a retention time closest to the analyte being measured.

10.29 See Appendix B Table R for a summary of the characteristic ions by analyte.

10.30 The RF is Calculated as follows:

$$RF = \frac{A_s \times C_{is}}{A_{is} \times C_s}$$

where:

A_s = Peak area(or height) of the analyte or surrogate.
A_{is} = Peak area (or height) of the internal standard.
C_s = Concentration of the analyte or surrogate.
C_{is} = Concentration of the internal standard.

10.31 System Performance Check Compounds (SPCCs) - Calculate the mean RF for each target analyte using the five RF values calculated from the initial (6 point) calibration curve. A system performance check should be made before this calibration curve is used.

10.32 Five compounds (the System Performance Check Compounds, or SPCCs) are checked for a minimum average response factor. These compounds are chloromethane, 1,1-Dichloroethane, Bromoform, Chlorobenzene, and 1,1,2,2-Tetrachloroethane. These compounds are used to check compound instability and to check for degradation caused by contaminated lines or active sites in the system. Example problems include:

Chloromethane is the most likely compound to be lost if the purge flow is too fast.

Bromoform is one of the compounds most likely to be purged very poorly if the purge flow is too slow. Cold spots and/or active sites in the transfer lines may adversely affect response. Response of the quantitation ion (m/z173) is directly affected by the tuning of BFB at ions m/z 174/176. Increasing the m/z 174/176 ratio relative to m/z 95 may improve Bromoform response.

Tetrachloroethane and 1,1-Dichloroethane are degraded by contaminated transfer lines in purge and trap systems and/or active sites in trapping materials.

10.33 The minimum mean response factors for the volatile SPCCs are as follows:

Chloromethane	0.10
1,1-Dichloroethane	0.10
Bromoform	0.10
Chlorobenzene	0.30
1,1,2,2-Tetrachloroethane	0.30

10.34 Calibration Check Compounds (CCCs) are used to evaluate the calibration from the standpoint of the integrity of the system. High variability for these compounds may be indicative of system leaks or reactive sites on the column. Meeting the CCC criteria is not a substitute for successful calibration of the target analytes.

10.35 Calculate the standard deviation (SD) and relative standard deviation (RSD) of the response factors for all target analytes from the initial calibration, as follows:

$$SD = \sqrt{\frac{\sum_{i=1}^{n}(RF_i - \overline{RF})^2}{n-1}} \qquad RSD = \frac{SD}{\overline{RF}} \times 100$$

where:

RF_i	=	RF for each of the calibration standards
$\overline{RF}$	=	mean RF for each compound from the initial calibration
n	=	Number of calibration standards, e.g., 5

The RSD should be less than or equal to 15% for each target analyte. However, the RSD for each individual Calibration Check Compound (CCC) must be equal or less than 30%.

10.36 The CCCs are:

1,1-Dichloroethene	Toluene
Chloroform	Ethyl benzene
1,2-Dichloropropane	Vinyl chloride

10.37 If an RSD of greater than 30% is measured for any CCC, then corrective action to eliminate a system leak and/or column reactive sites is necessary before reattempting

calibration.

10.38 Evaluation of retention times - The relative retention times of each target analyte in each calibration standard should agree within 0.10 relative retention time units. Late eluting compounds usually have much better agreement.

10.39 Linearity limit of target analytes is an RSD of 15% or less. At 15% or less the response factor is assumed to be constant over the calibration range, and the average response factor may be used for quantitation.

10.40 If the average response factor varies more than 15 % for a particular compound, a linear regression calculation for reporting may be performed for that particular compound in the analysis if the correlation coefficient is greater than 0.99. A new calibration curve can be performed to re-assess the use of average response factors at this time.

10.41 When the RSD exceeds 15%, the plotting and visual inspection of a calibration curve can be a useful diagnostic tool. The inspection may indicate analytical problems, including errors in standard preparation, the presence of active sites in the chromatographic system, analytes that exhibit poor chromatographic behavior, etc.

10.42 See Appendix B Table I for a summary of frequency and acceptance criteria.

10.43 See Appendix B Table O for a summary of Corrective Actions when linearity is not achieved.

10.44 The initial calibration curve for each compound of interest is verified, once every time clock or 12 hours, whichever is more frequent, prior to sample analysis, using the introduction technique used for samples. This is accomplished by analyzing a calibration standard at a concentration near the midpoint concentration for the calibrating range of the GC/MS. Continuing Calibration Check standards must be prepared daily.

10.45 See Appendix B Table I for a summary of frequency and acceptance criteria.

11.0 PROCEDURE:

11.1 Verify the instrument chromatographic conditions:

General:

Injector temperature:	220°C
Transfer line temperature:	280°C

Column:

Carrier gas (He) flow rate:	0.5 mL/min
Initial temperature:	40°C and hold for 3 minutes

Temperature program:	8°C/min. to 110°C
	20°C/min. to 220°C
Final temperature:	220°C hold 1 minute

Note: Direct inject to the DB624 column. 17 ml/min split/splitless flow with a column flow rate of 0.5 ml/min.

11.2 Establish the GC/MS operating conditions, using the following as guidance:

Mass range:	35 - 300 amu
Scan time:	0.6 - 2 sec/scan

Source temperature: According to manufacturer's specifications

Note: Condition a new trap by baking for 30 minutes at 270°C. To keep the trap in performance, condition the trap daily for 7 minutes at 270°C while backflushing.

11.3 Verify GC/MS calibration

11.4 Calibration verification consists of the steps listed below and are performed at the beginning of each 12-hour analytical shift.

11.5 Prior to the analysis of samples or calibration standards, inject or introduce 50 ng of the 4-Bromofluorobenzene standard into the GC/MS system. The resultant mass spectra for the BFB must meet the criteria given in Table 1 before sample analysis begins. These criteria must be demonstrated each 12 hour shift during which samples are analyzed.

11.6 The initial calibration curve for each compound of interest must be verified once every 12 hours prior to sample analysis, using the introduction technique used for samples. This is accomplished by analyzing a calibration standard at a concentration near the midpoint concentration for the calibrating range of the GC/MS.

11.7 See Appendix B Table I for a summary of frequency and acceptance criteria.

11.8 See Appendix B Table O for a summary of Corrective Actions when linearity is not achieved.

NOTE: The BFB and calibration verification standard may be combined into a single standard as long as both tuning and calibration verification acceptance criteria for the project can be met without interferences.

11.9 A method blank must be analyzed after the calibration standard to ensure that the total system (introduction device, transfer lines and GC/MS system) is free of contaminants. If the method blank indicates contamination, then it may be appropriate to analyze another method blank to demonstrate that the contamination is not a result of carryover from standards or samples.

11.10 If the second blank confirms contamination, the analyst must stop the analytical sequence determine the source of the contamination, eliminate the contamination if it involves a compound of interest, and begin the 12 hour sequence again.

11.11 See Appendix B Table I for a summary of frequency and acceptance criteria.

11.12 See Appendix B Table O for a summary of Corrective Actions when system in not free of contaminants is not achieved.

11.13 A system performance check must be made during every 12-hour analytical shift. Each SPCC compound in the calibration verification standard must meet its minimum response factor. This is the same check that is applied during the initial calibration.

11.14 If the minimum response factors are not met, the system must be evaluated, and corrective action must be taken before sample analysis begins. Possible problems include standard mixture degradation, injection port inlet contamination, contamination at the front end of the analytical column, and active sites in the column or chromatographic system. This check must be met before sample analysis begins.

11.15 After the system performance check is met, the CCCs are used to check the validity of the initial calibration. Use percent difference when performing the average response factor model calibration.

11.16 If the percent difference or drift for each CCC is less than or equal to 30%, the initial calibration is assumed to be valid. If the criterion is not met (i.e., greater than 30% difference or drift), for any one CCC, then corrective action must be taken prior to the analysis of samples. If the CCC's are not included in the list of analytes for a project, and therefore not included in the calibration standards, then all analytes must meet the 30% difference or drift criterion.

> Note: Problems similar to those listed under SPCCs could affect the CCCs. If the problem cannot be corrected by other measures, a new six point initial calibration must be generated. The CCC criteria must be met before sample analysis begins.

11.17 Internal standard retention time - The retention times of the internal standards in the calibration verification standard must be evaluated. If the retention time for any internal standard changes by more than 30 seconds from the that in the mid-point standard level of the most recent initial calibration sequence, then the chromatographic system must be inspected for malfunctions and corrections must be made, as required. When corrections are made, reanalysis of samples analyzed while the system was malfunctioning is required.

11.18 Internal standard response - If the EICP area for any of the internal standards in the calibration verification standard changes by a factor of two (50% to + 100%) from that in the mid-point standard level of the most recent initial calibration sequence, the mass spectrometer must be inspected for malfunctions and corrections must be made, as appropriate. When corrections are made, reanalysis of samples analyzed while the system was malfunctioning is required.

11.19 The experience of the analyst performing GC/MS analyses is invaluable to the success of the methods. Each day that analysis is performed, the calibration verification standard should be evaluated to determine if the chromatographic system is operating properly. Questions that should be asked are: Do the peaks look normal? Is the response obtained comparable to the response from previous calibrations? Careful examination of the standard chromatogram can indicate whether the column is still performing acceptably, the injector is leaking, the injector septum needs replacing, etc. If any changes are made to the system (e.g., the column changed), recalibration of the system must take place.

11.20 GC/MS analysis of Aqueous samples

11.21 Screening of the sample prior to purge-and-trap analysis will provide guidance on whether sample dilution is necessary and will prevent contamination of the purge-and-trap system. The Automated static headspace technique is used for the prescreening of volatile organics from solid and aqueous samples (Method 5021) using a gas chromatograph (GC) equipped with a photo ionization detector (PID)before introduction into the GC/MS system. It is highly recommended that the sample be screened to minimize contamination of the GC/MS system from unexpectedly high concentrations of organic compounds. When used only for screening purposes, the quality control requirements in the methods may be reduced as appropriate. Sample screening is particularly to protect a particularly expensive investment in equipment.

Note: BFB tuning criteria and GC/MS calibration verification criteria must be met before analyzing samples.

11.22 All samples and standard solutions must be allowed to warm to ambient temperature before analysis.

11.23 The process of taking an aliquot destroys the validity of remaining volume of an aqueous sample for future analysis. Therefore, if only one VOA vial is provided to the laboratory, the analyst should prepare two aliquots for analysis at this time, to protect against possible loss of sample integrity. This second sample is maintained only until such time when the analyst has determined that the first sample has been analyzed properly. For aqueous samples, one 20-mL syringe or vial could be used to hold two 5-mL aliquots although a dilution is required into a 40 mL vial to place in the auto sampler for proper operation. If the

second aliquot is to be taken from the syringe or vial, it must be analyzed within 24 hours. Care must be taken to prevent air from leaking into the syringe.

11.24 Adjust the purge gas (helium) flow rate to 40 mL/min on the purge-and-trap device. Optimize the flow rate to provide the best response for chloromethane and Bromoform, if these compounds are analytes. Excessive flow rate reduces chloromethane response, whereas insufficient flow reduces Bromoform response (see Section 6.3.5). Purge flow should be checked periodically.

11.25 Place 40 mL sample vials on the Archon or Dynatech auto sampler. Fill the standard syringe with working IS or IS/SS standard mixture. The concentration of this standard is 250 ppm, so that the amount of all components injected will be equivalent to 50 ug/L. Fill

the rinse water bottle with OFW, and discard the waste water bottle.

11.26 The following procedure is appropriate for diluting purgeable samples. All steps must be performed without delay until the diluted sample is in a 40 mL teflon screw-cap vial.

11.27 Dilutions are made in 50 mL syringes. Intermediate dilutions may be necessary for extremely large dilutions.

11.28 Calculate the approximate volume of OFW reagent water to be added to the 50 mL syringe. Appropriate dilution aliquot will be directly injected in the 50 mL syringe. Invert the syringe a minimum of three times, and then transfer to 40 ml teflon screw-cap vial. The sample vials will be placed on the Dynatech/Archon auto sampler.

11.29 The Dynatech or Archon system will drawn 5 mL aliquot from 40 mL sample vial, and combined approximately 1 ul of IS or IS/SS from standard syringe into 5 mL sparge tube.

11.30 Sample desorption - occurs at 250°C.

11.31 After the 10 minute purge, Dynatech/Archon steps into desorption and starts the GC/MS program.

11.32 The Mass Spectrometer starts data acquisition after a 1 minute solvent delay. (Desorption mode)

11.33 The trapped materials are introduced to the GC column by rapidly heating the trap to 250°C while back flushing the trap with an inert gas for 2 minutes.

11.34 Hold the column temperature at 40°C for 3 minutes, then a program increase the temperature at 8°C per minute to a temperature of 110°C, then increase to 220 °C at a rate of 20°C per minute and hold for 1 minute.

11.35 After desorbing the sample for 2 minutes, bake the trap at 270°C for 7 minutes.

11.36 During bake time, the sparge tube will be flushed with DI water 3 times.

11.37 When the trap is cool, the next sample can be purged.

11.38 If the initial analysis of sample or a dilution of the sample has a concentration of analytes that exceeds the initial calibration range or if the sample is suspected to be high, the sample must be reanalyzed at a higher dilution. Secondary ion quantitation is allowed only when there are sample interferences with the primary ion. When a sample is analyzed that has saturated ions from a compound, this analysis must be followed by a blank organic-free reagent water analysis. If the blank analysis is not free of interferences, the system must be decontaminated. Sample analysis may not resume until the blank analysis is demonstrated to be free of interferences.

11.39 For matrix spike analysis, add appropriate amount of the matrix spike to the 40 mL of sample vial. Disregarding any dilutions, this is equivalent to a concentration of 50 ug/L of each matrix spike standard.

11.40 All dilutions should keep the response of a major constituents (previously saturated peaks) near the upper half of the linear range of the curve, exception a larger dilution may be report if the chromatogram is very complex or late elution peaks are present.

11.41 Water-miscible liquids

11.42 Water-miscible liquids are analyzed as water samples after first diluting them at least 50 fold with OFW reagent water.

11.43 Initial and serial dilutions can be prepared by injecting with a gas tight syringe, 1 mL of the sample to a 50 mL DI water syringe. Transfer immediately to a 40 mL vial.

11.44 GC/MS analysis of Sediment/Soil and Waste samples

11.45 Screening of the sample prior to purge-and-trap analysis will provide guidance on whether sample dilution is necessary and will prevent contamination of the purge-and-trap system. The Automated static headspace technique is used for the prescreening of volatile organics from solid and aqueous samples (Method 5021) using a gas chromatograph (GC) equipped with a photo ionization detector (PID)before introduction into the GC/MS system. It is highly recommended that the sample be screened to minimize contamination of the GC/MS system from unexpectedly high concentrations of organic compounds. When used only for screening purposes, the quality control requirements in the methods may be reduced as appropriate. Sample screening is particularly important to protect particularly expensive investment in equipment. Use the screening data to determine whether to use the low-concentration method (< 200 ug/Kg) or the high-concentration method (> 200 ug/Kg).

Note: BFB tuning criteria and GC/MS calibration verification criteria must be met before analyzing samples.

11.46 All samples and standard solutions must be allowed to warm to ambient temperature before analysis.

11.47 Low Compound concentration method - This is designed for samples containing individual purgeable compounds of < 200 ug/Kg. It is limited to sediment/soil samples and waste that is of a similar consistency (granular and porous). The low-concentration method is based on purging a heated sediment/soil sample mixed with organic-free reagent water containing the surrogate and internal standards

11.48 Use a 5 g sample if the expected concentration is < 200 ug/Kg or a 1 g sample for expected concentrations between 200 and 1000 ug/Kg.

11.49 A heated purge calibration curve must be prepared and used for the quantitation of all samples analyzed with the low-concentration method. Follow the initial and daily calibration instructions, except for the addition of a 40°C purge temperature.

11.50 The sample consists of the entire contents of the sample container. Do not discard any supernatant liquids. Do not mix the contents of the sample container with a narrow metal spatula. Weigh the amount as determined in screening into a tarred 40ml VOA vial, 5ml of OFW, and a stir bar. Note and record the actual weight to the nearest 0.1 g. It is preferable to obtain a representative sample without mixing if possible, and proceed.

11.51 For samples received in a 5 gram En Core sampler, prior to extruding the sample, an effervescence check is made to determine which preservation technique is used. This step will have been completed and the volatiles area will have received either 2-40ml VOA vial with a stir bar and 1 gram of Sodium Bisulfate and 5 mls of DI water or 2-40ml VOA vial with a stir bar OFW if sample effervescence was indicated.

11.52 A third 5g or 25g sample preserved in methanol at a 1:1 ratio is also provided for high level analysis. During the preparation of the sample, noted and recorded electronically are the actual weights to the nearest 0.1 g for use in later calculations.

11.53 Determination of the percent solids found in the samples is accomplished by the procedures as listed in the SOP using the determinative method SM2540G. Waste samples are to be reported on a "wet-weight" basis in order to minimize the contamination to ovens used for more than one task.

11.54 Heat the sample to 40°C +/- 1°C and purge the sample for 10.0 +/- 0.1 minutes. Be sure the trap is cooler than 25°C.

11.55 For matrix spike analysis, add appropriate amount of the matrix spike to the 40 mL of sample vial. Disregarding any dilutions, this is equivalent to a concentration of 50 ug/kg of each matrix spike standard.

11.56 All dilutions should keep the response of a major constituents (previously saturated peaks) near the upper half of the linear range of the curve, exception a larger dilution may be report if the chromatogram is very complex or late elution peaks are present.

11.57 For low-concentration sediment/soils, add 2.5uL of the 100ppm matrix spike solution to the 5 mL of organic-free reagent water. The concentration for a 5 g sample would be equivalent to 50 ug/Kg of each matrix spike standard. Additionally, spike 5ml of DI water and add it to a 40ml VOA vial with a stir bar for the LCS/LCSD. If additional samples were not submitted for matrix spiking, then spike the same above amounts for a LCS/LCSD- pair.

11.58 High-concentration method - The method is based on extracting the sediment/soil with methanol. A waste sample is either extracted or diluted, depending on its solubility in methanol. An aliquot of the extract is added to OFW. This is purged at ambient temperature. All samples with an expected concentration of >200 ug/Kg should be analyzed by this method.

11.59 The sample consists of the entire contents of the sample container. Do not discard any supernatant liquids. Do not mix the contents of the sample container with a narrow metal spatula.

11.60 For waste that is soluble in methanol, weigh 20 g (wet weight) into a tarred 2 oz. jar.

11.61 Add 20 ml of methanol and an appropriate amount of surrogate-20 µl of 2500ppm surrogate mixture.

11.62 Cap and shake for 2 min and then sonicate for 20 minutes.

Note: The procedures above must be performed rapidly and without interruption to avoid loss of volatile organics. These steps must be performed in a laboratory free of solvent fumes, especially Methylene Chloride.

11.63 These extracts are stored at 4°C, prior to analysis.

11.64 Determine the volume of solvent extract to add to the 50mL of OFW for analysis. Use the estimated concentration from the screening data run to determine the appropriate volume of solvent extract. Otherwise, estimate the concentration range of the sample from the low concentration analysis to determine the appropriate volume. If the sample was submitted as a high-concentration sample, start a low µl amount and work up to a maximum of 1mL, while keeping a major peak near the upper half of the linear range of the curve. Exceptionally larger dilutions may be reported if chromatogram is complex or if late eluting peaks are present. This will protect the system from running a sample with high levels of target and non-target compounds.

11.65 Remove the plunger from a 50 mL Luerlock type syringe, fill until overflowing with OFW. Replace the plunger and compress the water to vent trapped air. Adjust the volume to 49 mL or Pull the plunger back to 50 mL to allow volume for the addition of the sample extract. Then add the volume of solvent extract determined in Section 6.6.5.6 and a volume of extraction or dissolution solvent to total 1mL. Invert the syringe twice, and transfer to 40 mL vial.

11.66 Place 40 mL vial on the Dynatech/Archon auto sampler.

11.67 Proceed with the analysis. Analyze all blanks on the same instrument as that used for the samples.

11.68 For a matrix spike in the high-concentration sediment/soil samples, add 10 mL of methanol, 10 µL of surrogate spike solution, and 50 µL of matrix spike solution as in section 6.6.5.5. Add a 1 mL aliquot of this extract to 49 mL of organic-free reagent water for purging. Due to varying high level sample concentrations it is sometimes necessary to run a LCS/LCSD instead of a matrix spike as a matrix spike is project specific and may not indicate effects associated with other samples from differing sources. This allows the reporting of precision and accuracy data when requested.

12.0 CALCULATIONS:

12.1 Qualitative Analysis

12.2 The qualitative identification of each compound determined by this method is based on retention time, and on comparison of the sample mass spectrum, after background correction, with characteristic ions in a reference mass spectrum. The reference mass spectrum must be generated by the laboratory using the conditions of this method. The characteristic ions from the reference mass spectrum are defined to be the three ions of greatest relative intensity, or any ions over 30% relative intensity if less than three such ions occur in the reference spectrum. Compounds are identified as present when the following criteria are met.

12.3 The intensities of the characteristic ions of a compound maximize in the same scan or within one scan of each other. Selection of a peak by a data system target compound search routine where the search is based on the presence of a target chromatographic peak containing ions specific for the target compound at a compound specific retention time will be accepted as meeting this criterion.

12.4 The relative retention time (RRT) of the sample component is within ± 0.10 RRT units of the RRT of the standard component.

12.5 The relative intensities of the characteristic ions agree within 30% of the relative intensities of these ions in the reference spectrum.

> Example: For an ion with an abundance of 50% in the reference spectrum, the corresponding abundance in a sample spectrum can range between 20% and 80%.

12.6 A "Q" value is evaluated along with the concentration. Q values below 80% should be looked at closely for the above criteria. Ion chromatograms of all positive hits should be examined in Target Review.

12.7 Structural isomers that produce very similar mass spectra should be identified as individual isomers if they have sufficiently different GC retention times. Sufficient GC resolution is achieved if the height of the valley between two isomer peaks is less than 25% of the sum of the two peak heights. Otherwise, structural isomers are identified as isomeric pairs.

12.8 Identification is hampered when sample components are not resolved chromatographically and produce mass spectra containing ions contributed by more than one analyte. When gas chromatographic peaks obviously represent more than one sample component (i.e., a broadened peak with shoulder(s) or a valley between two or more maxima), appropriate selection of analyte spectra and background spectra is important.

12.9 Examination of extracted ion current profiles of appropriate ions can aid in the selection of spectra, and in qualitative identification of compounds. When analytes co elute (i.e., only one chromatographic peak is apparent), the identification criteria may be met, but each analyte spectrum will contain extraneous ions contributed by the co eluting compound.

12.10 For samples containing components not associated with the calibration standards, a library search may be made for the purpose of tentative identification. The necessity to perform this type of identification will be determined by the purpose of the analyses being conducted. Data system library search routines should not use normalization routines that would misrepresent the library or unknown spectra when compared to each other. For example, the RCRA permit or waste delisting requirements may require the reporting of non-target analytes. Only after visual comparison of sample spectra with the nearest library searches may the analyst assign a tentative identification. Use the following guidelines for making tentative identifications:

(1) Relative intensities of major ions in the reference spectrum (ions greater than 10% of the most abundant ion) should be present in the sample spectrum.

(2) The relative intensities of the major ions should agree within ± 20%.

Example: For an ion with an abundance of 50% in the standard spectrum, the corresponding sample ion abundance must be between 30 and 70%).

(3) Molecular ions present in the reference spectrum should be present in the sample spectrum.

(4) Ions present in the sample spectrum but not in the reference spectrum should be reviewed for possible background contamination or presence of co eluting compounds.

(5) Ions present in the reference spectrum but not in the sample spectrum should be reviewed for possible subtraction from the sample spectrum because of background contamination or co eluting peaks. Data system library reduction programs can sometimes create these discrepancies.

(6) Computer generated library search routines should not use normalization routines that would misrepresent the library or unknown spectra when compared to each other. Only after visual comparison of sample with the nearest library searches will the mass spectral interpretation specialist assign a tentative identification.

12.11 Quantitative Analysis

12.12 When a compound has been identified, the quantitation of that compound will be based on the integrated abundance from the EICP of the primary characteristic ion. Quantitation will take place using the internal standard technique. The internal standard used shall be the one nearest the retention time of that of a given analyte.

12.13 If the RSD of a compound's response factors is 15% or less, then the concentration in the sample may be determined using the average response factor (RF)from initial calibration data.

12.14 Where applicable, the concentration of any non-target analytes identified in the sample should be estimated. The same formula should be used with the following modifications: The areas $A_{(x)}$ and $A_{(is)}$ should be from the total ion chromatograms, and the RF for the compound should be assumed to be 1.

12.15 The resulting concentration should be reported indicating:

(1) that the value is an estimate, and

(2) which internal standard was used to determine concentration. Use the nearest internal standard free of interferences.

12.16 Calculate the concentration of each identified analyte in the sample as follows:

Aqueous samples

$$\text{concentration (ug/L)} = \frac{(A_{(x)})(I_{(is)})}{(A_{(is)})(RF)(V_{(o)})}$$

where:

$A_{(x)}$ = Area of characteristic ion for compound being measured.
$I_{(is)}$ = Amount of internal standard injected (ng).
$A_{(is)}$ = Area of characteristic ion for the internal standard.
RF = Response factor for compound being measured (Sec.6.3.4).
$V_{(o)}$ = Volume of water purged (mL), taking into consideration any dilutions made.

Soil and Solid samples

$$\text{concentration (ug/Kg)} = \frac{(A_{(x)})(I_{(is)})(V_{(t)})}{(A_{(is)})(RF)(V_{(i)})(W_{(s)})(D)}$$

where:

$A_{(x)}$, $I_{(is)}$, $A_{(is)}$, RF, = Same as for water.
$V_{(t)}$ = Volume of total extract (uL) (use 10,000 uL or a factor of this when dilutions are made).
$V_{(i)}$ = Volume of extract added (uL) for purging.
$W_{(s)}$ = Weight of sample extracted or purged (g).
D = % dry weight of sample/100, or 1 for a wet-weight basis.

13.0 METHOD PERFORMANCE:

13.1 The method detection limit (MDL) is defined as the minimum concentration of a substance that can be measured and reported with 99% confidence that the value is above zero. The MDL actually achieved in a given analysis will vary depending on instrument sensitivity and matrix effects.

13.2 This method was tested using purge-and-trap with water fortified with all target compounds at concentrations of 0.5 ug/L to 5 ug/L, depending on the response of the particular analyte. A narrow-bore column was used in the analyses.

13.3 See Appendix B Table K for a Initial Demonstration of Capability summary.

14.0 POLLUTION PREVENTION:

14.1 Pollution prevention encompasses any technique or procedure that reduces or eliminates the quantity or toxicity of waste at the point of generation. Laboratory staff should order where possible acceptable non-toxic alternative supplies and prepare only those quantities of reagents or standards that will be used prior to the expiration date. Other appropriate measures to minimize waste generation should be brought to the attention of laboratory management. All laboratory waste shall be handled as directed by the Laboratory Waste Management Plan and Hazardous Waste Contingency Plan.

15.0 DATA ASSESSMENT AND ACCEPTANCE CRITERIA FOR QUALITY CONTROL MEASURES:

15.1 At a minimum there are three levels of data review:

Analyst/Technician verification at the bench top are looking at instrument performance as it relates to initial calibration, calibration verification, cleanliness.

Supervisor/Analyst verification after analysis are looking for sample concentration versus linear range of the instrument, typical patterns resulting from the compounds or elements in question, and quality control measure criteria. Data is either accepted without qualification, accepted with qualification, or rejected with samples reprocessed.

Report Reviewer/Project Management personnel look at the presentation of the data on final reports. The reports are verified for holding time compliance, receipt conditions, method citation and reasonableness, which may include field duplicate analysis, analytical comments and qualifications presented by the previous reviews, and comparing results of similar analytical techniques as it relates to the project site information when available.

A fourth level of review , data validation is determined from a project or program scope of laboratory services and encompasses the generation Quality Control summaries in the media of "Form Generation",

15.2 See Appendix B Tables listed below for summaries of review:

Analyst/Technician Data Assessment	Table L
Analyst/Supervisor Data Assessment	Table M
Report Reviewer/Project Management Data Assessment	Table N
Corrective Actions	Table O
Contingencies for Out of Control Data	Table P

16.0 CORRECTIVE ACTIONS FOR OUT-OF-CONTROL DATA

16.1 Assessment of Quality Control measures is done to provide a level of confidence in the data generated. The measures provide documentation that the instrument conditions were reliable during the analysis.

16.2 See Table O for corrective actions taken in cases where statistical goals were not achieved

17.0 CONTINGENCIES FOR HANDLING OUT-OF-CONTROL OR UNACCEPTABLE DATA

17.1 During analysis, events occur specific to the physical and chemical characteristics of the environmental sample. When possible, with received sample volumes, data generated along with measures that do not meet statistical goals are re-analyzed again to see if the statistical goal can be achieved. When environmental samples do not meet statistical unacceptable data is generated. These events are different from those pertaining to instrument operating conditions. These events occur when the instruments are operating under ideal conditions. Contingencies for handling sample out-of-control quality control assessments are found in Table P. Data Qualifiers are found in Table Q. The tables list routine assessment and not those specific to a project/program where data validation is determined from a project or program scope of laboratory services and encompasses the generation Quality Control summaries in the media of "Form Generation",

18.0 WASTE MANAGEMENT

18.1 To minimize waste during sample preparation has two benefits. The first benefit is a cost savings to the lab in materials and supplies. The second is a benefit to the environment as less materials need to be disposed.

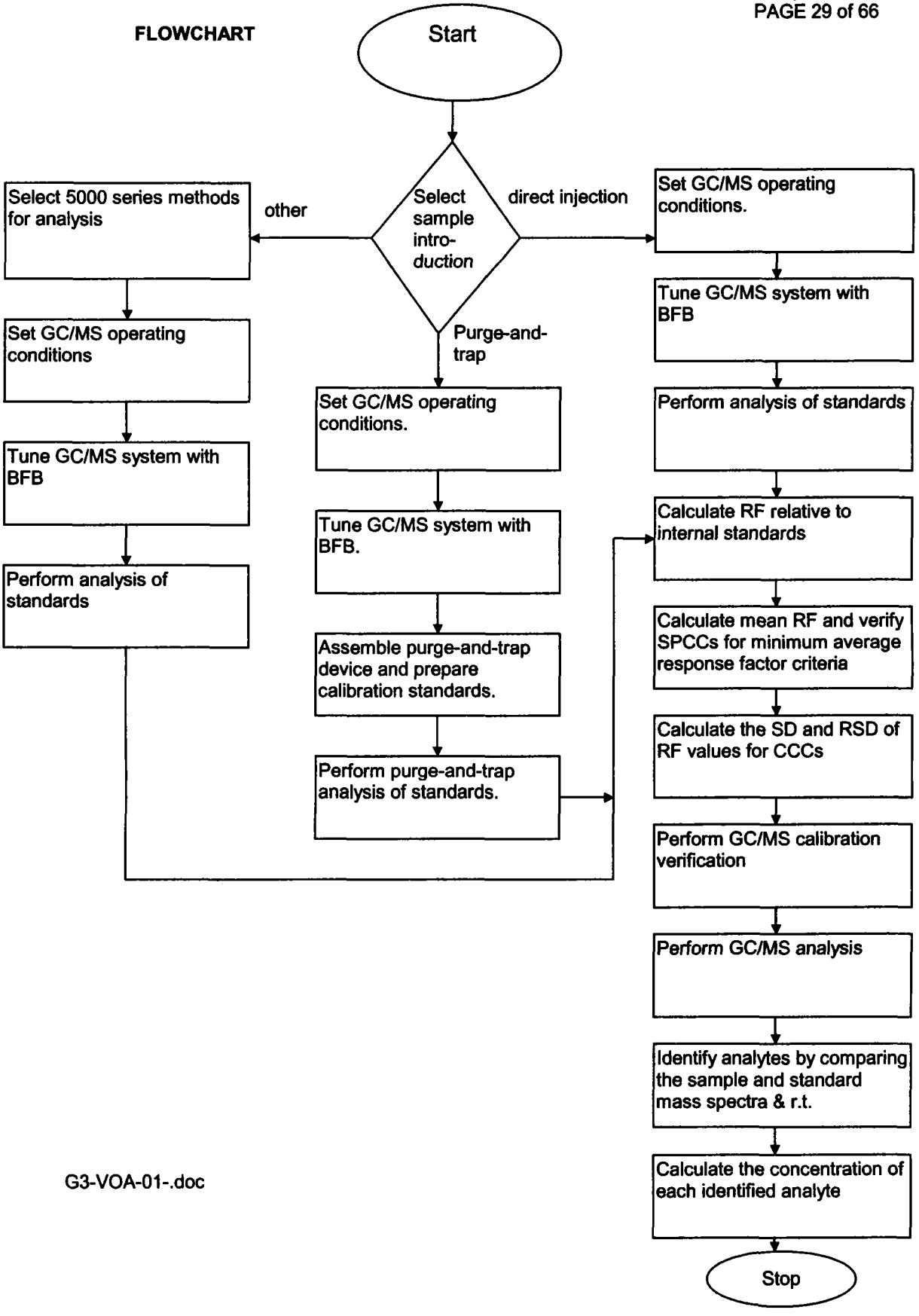
18.2 Use micro-syringes where ever possible, especially the 50 mL syringes for standards and dilutions. Spiking amounts are kept below the midpoint of the curve in order to eliminate carryover into the blank.

18.3 Solvent amount for cleaning syringes, standard preparation, are kept to a minimum.

REFERENCES:

40CFR Part 136
American Society for Quality Control (ASQC), Definitions of Environmental Quality Assurance Terms, 1996
American National Standards Institute (ANSI), Style Manual for Preparation of Proposed American National Standards, Eighth Edition, March 1991
ANSI/ASQC E4, 1994
ANSI N42.23-1995, Measurement and Associated Instrument Quality Assurance for
National Institute of Standards and Technology (NIST)
National Environmental Laboratory Accreditation Conference (NELAC), July 1998 Standards
Random House College Dictionary
US EPA Quality Assurance Division (QAD)
Webster's New World Dictionary of the American Language
EPA SW-846, Methods 8260B, December 1996
EPA SW-846, Methods 5035, December 1996
EPA SW-846, Methods 5030B, December 1996
EPA SW-846, Methods 8000B December 1996
40 CFR, Method EPA 624

FLOWCHART


Start
Select sample intro-duction
other
direct injection
Purge-and-trap
Select 5000 series methods for analysis
Set GC/MS operating conditions
Tune GC/MS system with BFB
Perform analysis of standards
Set GC/MS operating conditions.
Tune GC/MS system with BFB.
Assemble purge-and-trap device and prepare calibration standards.
Perform purge-and-trap analysis of standards.
Set GC/MS operating conditions.
Tune GC/MS system with BFB
Perform analysis of standards
Calculate RF relative to internal standards
Calculate mean RF and verify SPCCs for minimum average response factor criteria
Calculate the SD and RSD of RF values for CCCs
Perform GC/MS calibration verification
Perform GC/MS analysis
Identify analytes by comparing the sample and standard mass spectra & r.t.
Calculate the concentration of each identified analyte
Stop

VALIDATION DATA:

The laboratory must demonstrate initial proficiency with each combination of sample preparation and determinative methods utilized, by generating data of acceptable accuracy and precision for a reference sample containing the target parameters in a clean matrix. The laboratory must also repeat this demonstration whenever new staff are trained or significant changes in instrumentation are made. Analysts previously trained shall perform a continued demonstration of proficiency annually by performing "Yearly Demonstration of Capability" (YDC).

The values from the determinative method are presented as guidance only and are not intended as absolute acceptance criteria. En Chem generates data for method performance and are presented along with the performance values in Table R. Accuracy of parameters routinely analyzed by En Chem and not in Table R can be found in the Initial Demonstration Data Table K.

MANAGEMENT APPROVAL AND REVIEW OF SOPS - POLICY AND DOCUMENTATION

REVIEWED BY: _____ Section Supervisor — Date: 1/3/2002

REVIEWED BY: M. Subra _____ Quality Assurance Officer — Date: 01-02-2002

APPROVED BY: _____ Laboratory Manager — Date: 1/3/2002

Periodic Review Record

Review Date						
Initials						

APPENDICES

APPENDIX A	**DEFINITIONS**
APPENDIX B	**TABLES**

Appendix A

Acceptance Criteria: specified limits placed on characteristics of an item, process, or service defined in requirement documents. (ASQC)
Accuracy: the degree of agreement between an observed value and an accepted reference value. Accuracy includes a combination of random error (precision) and systematic error (bias) components which are due to sampling and analytical operations; a data quality indicator. (QAMS)
Aliquot: a measured portion of a sample, or solution, taken for sample preparation and/or analysis.
Analytical Detection Limit: the smallest amount of an analyte that can be distinguished in a sample by a given measurement procedure throughout a given (e.g., 0.95) confidence interval. (applicable only to radiochemistry)
Analyst: the designated individual who performs the "hands-on" analytical methods and associated techniques and who is the one responsible for applying required laboratory practices and other pertinent quality controls to meet the required level of quality. (NELAC)
Assessment Criteria: the measures established by NELAC and applied in establishing the extent to which an applicant is in conformance with NELAC requirements. (NELAC)
Blank: an analytical sample designed to assess specific sources of laboratory contamination.
4-Bromofluorobenzene (BFB): the compound chosen to establish mass spectral instrument performance for volatile (VOA) analyses. It is also used in the VOA fraction as a system monitoring compound (SMC) or surrogate standard.
Contamination: a component of a sample or an extract that is not representative of the environmental source of the sample. Contamination may stem from other samples, sampling equipment, while in transit, from laboratory reagents, laboratory environment, or analytical instruments.
Continuing Calibration: analytical standard run every 12 hours to verify the initial calibration of the system.
Data Reduction: the process of transforming raw data by arithmetic or statistical calculations, standard curves, concentration factors, etc., and collation into a more useable form. (EPA-QAD)
Deficiency: an unauthorized deviation from acceptable procedures or practices, or a defect in an item. (ASQC)
Demonstration of Capability: a procedure to establish the ability of the analyst to generate acceptable accuracy. (NELAC)
Detection Limit: the lowest concentration or amount of the target analyte that can be determined to be different from zero by a single measurement at a stated degree of confidence.
Document Control: the act of ensuring that documents (and revisions thereto) are proposed, reviewed for accuracy, approved for release by authorized personnel, distributed properly and controlled to ensure use of the correct version at the location where the prescribed activity is performed. (ASQC)
Duplicate Analyses: the analyses or measurements of the variable of interest performed identically on two sub-samples of the same sample. The results from duplicate analyses are used to evaluate analytical or measurement precision but not the precision of sampling, preservation or storage internal to the laboratory. (EPA-QAD)
Equipment Blank: a sample of analyte-free media which has been used to rinse common sampling equipment to check effectiveness of decontamination procedures. (NELAC)
estimate of Target analyte concentration is available. Matrix spikes are used, for example, to determine the effect of the matrix on a method's recovery efficiency. (QAMS)
Gas Chromatograph (GC): the instrument used to separate analytes on a stationary phase within a chromatographic column. The analytes are volatized directly from the sample (VOA water and low-soil), volatized from the sample extract (VOA medium soil). In VOA anlaysis, the

compounds are detected by a Mass Spectrometer (MS). In the screening procedure, the Photo Ionization Detector (PID) is used as the detector.

Initial Calibration: analysis of analytical standards for a series of different specified concentrations; used to define the linearity and dynamic range of the response of the mass spectrometer to the target compounds.

Internal Standards: compounds added to every standard, blank, matrix spike, matrix spike duplicate, sample (for volatiles), and sample extract (for medium soil) at a known concentration, prior to analysis. Internal standards are used as the basis for quantitation of the target compounds.

Instrument Blank: a blank designed to determine the level of contamination associated wit the analytical instruments.

m/z: Mass to charge ratio, synonymous with "m/e".

Matrix: the predominant material of which the sample to be analyzed is composed. For the purpose of this SOW, a sample matrix is either water or soil/sediment. Matrix is not synonymous with phase (liquid or solid).

Matrix Effect: in general, the effect of a particular matrix (water of soil/sediment) on the constituents with which it contacts. This is particularly pronounced for clay particles which may adsorb chemicals and catalyze reations. Matrix effects may prevent extraction of target analytes, and may affect surrogate recoveries. In addition, non-target analytes may be extracted from the matrix casusing interferences.

Matrix Spike Duplicate (spiked sample or fortified sample duplicate): a second replicate matrix spike prepared in the laboratory and analyzed to obtain a measure of the precision of the recovery for each analyte. (QAMS)

May: denotes permitted action, but not required action. (NELAC)

Method Blank: a sample of a matrix similar to the batch of associated samples (when available) that is free from the analytes of interest, which is processed simultaneously with and under the same conditions as samples through all steps of the analytical procedures, and in which no target analytes or interferences are present at concentrations that impact the analytical results for sample analyses. (NELAC)

Method Detection Limit: the minimum concentration of a substance (an analyte) that can be measured and reported with 99% confidence that the analyte concentration is greater than zero and is determined from analysis of a sample in a given matrix containing the analyte. (40 CFR Part 136, Appendix B)

Micro Bore Capillary Column: a gas chromatographic column with an internal diameter (ID) that is less than or equal to 0.18 mm. Columns with greater diameters are classified as narrow bore and mega bore capillary columns.

Must: denotes a requirement that must be met. (Random House College Dictionary)

National Institute of Standards and Technology (NIST): an agency of the US Department of Commerce's Technology Administration that is working with EPA, States, NELAC, and other public and commercial entities to establish a system under which private sector companies and interested States can be accredited by NIST to provide NIST-traceable proficiency testing (PT) to those laboratories testing drinking water and wastewater. (NIST)

Negative Control: measures taken to ensure that a test, its components, or the environment do not cause undesired effects, or produce incorrect test results. (NELAC)

(items), and services. The quality system provides the framework for planning, implementing, and assessing work performed by the organization and for carrying out required QA and QC. (ANSI/ASQC E-41994)

OFW: Organic-free water, ASTM Type II deionzed water, used in the laboratory.

Percent Moisture: an approximation of the amount of water in a soil/sediment sample made by drying an aliquot of the sample at 105°C. The percent moisture determined in this manner also includes contributions from all compounds that may volatilize at or below 105°C, including water.

Percent moisture may be determined from decanted samples and from samples that are not decanted.

Purge and Trap (Device): analytical technique (device) used to isolate volatile (purgeable) organics by stripping the compounds from water or soil by a stream of inert gas, trapping the compounds on an adsorbent such as a porous polymer trap, and thermally desorbing the trapped compounds onto the gas chromatographic column.

Quantitation Limits: the maximum or minimum levels, concentrations, or quantities of a target variable (e.g., target analyte) that can be quantified with the confidence level required by the data user. (NELAC)

Range: the difference between the minimum and the maximum of a set of values. (EPA-QAD)

Raw Data: any original factual information from a measurement activity or study recorded in a laboratory notebook, worksheets, records, memoranda, notes, or exact copies thereof that are necessary for the reconstruction and evaluation of the report of the activity or study. Raw data may include photography, microfilm or microfiche copies, computer printouts, magnetic media, *including dictated observations, and recorded data from automated instruments. If exact copies* of raw data have been prepared (e.g., tapes which have been transcribed verbatim, data and verified accurate by signature), the exact copy or exact transcript may be submitted. (EPA-QAD)

Reagent Blank (method blank): a sample consisting of reagent(s), without the target analyte or sample matrix, introduced into the analytical procedure at the appropriate point and carried through all subsequent steps to determine the contribution of the reagents and of the involved analytical steps. (QAMS)

Reagent Water: water in which an interferant is not observed at or above the minimum quantitation limit of the parameters of interest.

Reconstructed Ion Chromotogram (RIC): a mass spectral graphical representation of the separation achieved by a gas chromatograph; a plot of total ion current versus retention time.

Record Retention: The systematic collection, indexing and storing of documented information under secure conditions. (EPA-QAD)

Reference Material: a material or substance one or more properties of which are sufficiently well established to be used for the calibration of an apparatus, the assessment of a measurement method, or for assigning values to materials. (ISO Guide 30-2.1)

Reference Method: a method of known and documented accuracy and precision issued by an organization recognized as competent to do so. (NELAC)

Reference Standard: a standard, generally of the highest metrological quality available at a given location, from which measurements made at that location are derived. (VIM-6.08)

Relative Percent Difference (RPD): the comparison of two values; the relative percent difference is based on the mean of the two values, and is reported as an absolute value, i.e., always expressed as a positive number or zero.

Relative Response Factor (RRF): a measure of the relative mass spectral response of an analyte compared to it's internal standard. Relative Response Factors are determined by analysis of standards and are used in the calculation of concentrations of analytes in samples. See the "Calculations" section for the exact formula.

Relative Retention Time (RRT): the ratio of the tetention time of a compound to that of a standard (such as an internal standard).

$$RRT = \frac{RT_c}{RT_{is}}$$

Where,

Rt_c = Retention time for the volatile target or surrogate compound in continuing calibration.

Rt_{is} = Retention time for the internal standard in calibration standard or in a sample.

Replicate Analyses: the measurements of the variable of interest performed identically on two or more sub-samples of the same sample within a short time interval. (NELAC)

Requirement:. denotes a mandatory specification; often designated by the term "shall". (NELAC)

Resource Conservation and Recovery Act (RCRA): the enabling legislation under 42 USC 321 *et seq.* (1976), that gives EPA the authority to control hazardous waste from the "cradle-to-grave", including its generation, transportation, treatment, storage, and disposal. (NELAC)

Response: or Instrumental Response: a measurement of the output of the GC detector (MS, FID, or PID) in which the intensity of the signal is proportionate to the amount (or concentration) detected. Measured by peak area or peak height.

Retention Time (RT): the time a target analyte is retained on a GC column before elution. The identification of a target analyte is dependent on a target compound's retention time falling within the specified retention time window established for that compound. Retention time is dependent on the nature of the column's stationary phase, column diameter, temperature, flow rate, and other parameters.

Sample Duplicate: two samples taken from and representative of the same population and carried through all steps of the sampling and analytical procedures in an identical manner. Duplicate samples are used to assess variance of the total method including sampling and analysis. (EPA-QAD)

Selectivity: (Analytical chemistry) the capability of a test method or instrument to respond to a target substance or constituent in the presence of non-target substances. (EPA-QAD)

Sensitivity: the capability of a method or instrument to discriminate between measurement responses representing different levels (e.g., concentrations) of a variable of interest. (NELAC)

Shall: denotes a requirement that is mandatory whenever the criterion for conformance with the specification requires that there be no deviation. This does not prohibit the use of alternative approaches or methods for implementing the specification so long as the requirement is fulfilled. (ANSI)

Should: denotes a guideline or recommendation whenever noncompliance with the specification is permissible. (ANSI)

Spike: a known mass of target analyte added to a blank sample or sub-sample; used to determine recovery efficiency or for other quality control purposes. (NELAC)

Standard: the document describing the elements of laboratory accreditation that has been developed and established within the consensus principles of NELAC and meets the approval requirements of NELAC procedures and policies. (ASQC)

Standard Operating Procedures (SOPs): a written document which details the method of an operation, analysis or action whose techniques and procedures are thoroughly prescribed and which is accepted as the method for performing certain routine or repetitive tasks. (QAMS)

Standardized Reference Material (SRM): a certified reference material produced by the U.S. National Institute of Standards and Technology or other equivalent organization and characterized for absolute content, independent of analytical method. (EPA-QAD)

Supervisor (however named): the individual(s) designated as being responsible for a particular area or category of scientific analysis. This responsibility includes direct day-to-day supervision of technical employees, supply and instrument adequacy and upkeep, quality assurance/quality control duties and ascertaining that technical employees have the required balance of education, training and experience to perform the required analyses. (NELAC)

Surrogate: a substance with properties that mimic the analyte of interest. It is unlikely to be found in environment samples and is added to them for quality control purposes. (QAMS)
Systems Audit (also Technical Systems Audit): a thorough, systematic, qualitative on-site assessment of the facilities, equipment, personnel, training, procedures, record keeping, data validation, data management, and reporting aspects of a total measurement system. (EPA-QAD)
Test: a technical operation that consists of the determination of one or more characteristics or performance of a given product, material, equipment, organism, physical phenomenon, process or service according to a specified procedure. The result of a test is normally recorded in a document sometimes called a test report or a test certificate. (ISO/IEC Guide 2-12.1, amended)
Test Method: an adoption of a scientific technique for a specific measurement problem, as documented in a laboratory SOP. (NELAC)
Tolerance Chart: A chart in which the plotted quality control data is assessed via a tolerance level (e.g. +/- 10% of a mean) based on the precision level judged acceptable to meet overall quality/data use requirements instead of a statistical acceptance criteria (e.g. +/- 3 sigma) (applies to radiobioassay laboratories). (ANSI)
Toxic Substances Control Act (TSCA): the enabling legislation in 15 USC 2601 *et seq.*, (1976), that provides for testing, regulating, and screening all chemicals produced or imported into the United States for possible toxic effects prior to commercial manufacture. (NELAC)
Traceability: the property of a result of a measurement whereby it can be related to appropriate standards, generally international or national standards, through an unbroken chain of comparisons. (VIM-6.12)
Twelve Hour Time Clock: the twelve (12) hour time period for GC/MS system instrument performance check, standards calibration (initial or continuing calibration), and method blank analysis begins at the moment of injection of the BFB analysis that the laboratory submits as documentation of instrument performance. The time period ends after 12 hours have elapsed according to the system clock.
United States Environmental Protection Agency (EPA): the federal governmental agency with responsibility for protecting public health and safeguarding and improving the natural environment (i.e., the air, water, and land) upon which human life depends. (US-EPA)
Validation: the process of substantiating specified performance criteria. (EPA-QAD)
Verification: confirmation by examination and provision of evidence that specified requirements have been met. (NELAC)NOTE: In connection with the management of measuring equipment, verification provides a means for checking that the deviations between values indicated by a measuring instrument and corresponding known values of a measured quantity are consistently smaller than the maximum allowable error defined in a standard, regulation or specification peculiar to the management of the measuring equipment. The result of verification leads to a decision either to restore in service, to perform adjustment, to repair, to downgrade, or to declare obsolete. In all cases, it is required that a written trace of the verification performed shall be kept on the measuring instrument's individual record.
Volatile Compounds: compounds amenable to analysis by the purge and trap technique. Used synonymously with purgeable compounds.
Work Cell: a well-defined group of analysts that together perform the method analysis. The members of the group and their specific functions within the work cell must be fully documented. (NELAC)

Appendix B

Tables

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Table A

COMPOUND NAME	AQUEOUS (ug/L)		SOLIDS (ug/Kg)			
			Low Level		Medium Level	
	MDL	EQL	MDL	EQL	MDL	EQL
Dichlorodifluoromethane	0.12	1	0.27	5	17	50
Chloromethane	0.42	1	0.30	5	22	50
Vinyl Chloride	0.19	1	0.25	5	19	50
Bromomethane	0.62	1	0.55	5	25	50
Chloroethane	0.46	1	0.36	5	21	50
Dichlorofluoromethane	0.23	1	0.39	5	20	50
Trichlorofluoromethane	0.31	1	0.24	5	21	50
Diethyl Ether	0.57	1	0.44	5	33	50
Acrolein	1.38	5	1.24	5	94	50
1 1-Dichloroethene	0.29	1	0.47	5	24	50
1 1 2-Trichlorotrifluoroethane	0.54	1	0.35	5	28	50
Acetone	2.84	5	4.58	5	137	250
Iodomethane	0.23	1	0.21	5	17	50
Carbon Disulfide	0.24	1	0.94	5	18	50
Allyl Chloride	0.85	1	0.46	5	23	50
Methylene Chloride	0.36	1	0.39	5	22	50
Acrylonitrile	0.72	5	1.30	5	69	250
Trans-1 2-Dichloroethene	0.35	1	0.35	5	25	50
Methyl T-Butyl Ether	0.20	1	0.17	5	20	50
N-Hexane	0.38	1	0.34	5	22	50
1 1-Dichloroethane	0.17	1	0.33	5	23	50
Vinyl Acetate	1.95	5	1.67	5	250	250
Diisopropyl Ether	0.23	1	1.22	5	11	50
2 2-Dichloropropane	0.28	1	0.18	5	23	50
Cis-1 2-Dichloroethene	0.27	1	0.25	5	22	50
2-Butanone	3.01	5	1.98	5	86	250
Bromochloromethane	0.56	1	0.37	5	21	50
Tetrahydrofuran	1.57	5	0.83	5	67	250
Chloroform	0.29	1	0.31	5	21	50
1 1 1-Trichloroethane	0.21	1	0.26	5	13	50
Carbon Tetrachloride	0.22	1	0.33	5	21	50
1 1-Dichloropropene	0.46	1	0.36	5	24	50
Benzene	0.29	1	0.28	5	12	50
1 2-Dichloroethane	0.21	1	0.30	5	22	50
Diethoxymethane	1.09	1	0.20	5	250	50
Trichloroethene	0.32	1	0.16	5	19	50
1 2-Dichloropropane	0.23	1	0.23	5	18	50

COMPOUND NAME	AQUEOUS (ug/L)		SOLIDS (ug/Kg)			
			Low Level		Medium Level	
	MDL	EQL	MDL	EQL	MDL	EQL
Dibromomethane	0.36	1	0.26	5	24	50
Bromodichloromethane	0.30	1	0.19	5	21	50
2-Chloroethyl Vinyl Ether	0.96	1	0.66	5	45	50
Cis-1 3-Dichloropropene	0.23	1	0.33	5	13	50
4-Methyl-2-Pentanone	1.06	5	0.42	5	48	250
Toluene	0.13	1	0.21	5	11	50
Trans-1 3-Dichloropropene	0.21	1	0.23	5	12	50
1 1 2-Trichloroethane	0.33	1	0.40	5	17	50
Tetrachloroethene	0.33	1	0.38	5	23	50
1 3-Dichloropropane	0.35	1	0.24	5	17	50
2-Hexanone	1.38	5	1.97	5	27	250
Dibromochloromethane	0.25	1	0.95	5	12	50
1 2-Dibromoethane	0.42	1	0.14	5	13	50
Chlorobenzene	0.19	1	0.16	5	16	50
1 1 1 2-Tetrachloroethane	0.30	1	0.18	5	14	50
Ethyl Benzene	0.19	1	0.24	5	16	50
M- P-Xylene	0.60	2	0.45	10	27	100
O-Xylene	0.28	1	0.42	5	13	50
Styrene	0.26	1	0.27	5	12	50
Bromoform	0.34	1	0.37	5	16	50
Isopropylbenzene	0.19	1	0.18	5	14	50
Trans-1 4-Dichloro-2-Butene	1.41	5	1.53	5	25	250
Bromobenzene	0.35	1	0.25	5	17	50
1 1 2 2-Tetrachloroethane	0.22	1	0.26	5	13	50
1 2 3-Trichloropropane	0.89	1	0.42	5	24	50
Cis-1 4-Dichloro-2-Butene	1.49	5	2.45	5	33	250
N-Propylbenzene	0.17	1	0.28	5	13	50
2-Chlorotoluene	0.48	1	0.25	5	12	50
4-Chlorotoluene	0.21	1	0.25	5	17	50
1 3 5-Trimethylbenzene	0.29	1	0.29	5	13	50
Tert-Butylbenzene	0.23	1	0.32	5	13	50

COMPOUND NAME	AQUEOUS (ug/L)		SOLIDS (ug/Kg)			
			Low Level		Medium Level	
1 2 4-Trimethylbenzene	0.34	1	0.26	5	14	50
Sec-Butylbenzene	0.20	1	0.22	5	14	50
1 3-Dichlorobenzene	0.12	1	0.23	5	14	50
P-Isopropyltoluene (Cymene)	0.25	1	0.19	5	12	50
1 4-Dichlorobenzene	0.31	1	0.27	5	13	50
1 2-Dichlorobenzene	0.22	1	0.26	5	13	50
N-Butylbenzene	0.28	1	0.25	5	24	50
Hexachloroethane	0.55	1	0.44	5	22	50
1 2-Dibromo-3-Chloropropane	0.69	1	0.24	5	21	50
1 2 4-Trichlorobenzene	0.24	1	0.36	5	20	50
Hexachlorobutadiene	0.43	1	0.32	5	24	50
Naphthalene	0.27	1	0.22	5	10	50
1 2 3-Trichlorobenzene	0.30	1	0.28	5	22	50
2-Methylnaphthalene	0.45	5	0.32	5	29	250

Table B

REQUIRED SAFETY EQUIPMENT

Item:	Safety Apparel Description	Mandatory for Procedure	Optional
1	Safety Goggles or Glasses		✔
2	Lab Coat		✔
3	Gloves		✔
4	Face Shield		✔

Table C

EQUIPMENT

Equipment	Manufacturer	Model(s)	Serial #	Date In Service
GCMS#1	Hewlett-Packard	5972//5890A	3306A00154//3235A46437	5/93
GCMS#2	Hewlett-Packard	5972//5890E	3341A01324//3336A53122	5/94
GCMS#3	Hewlett-Packard	5972//5890E	3341A01384//3336A53586	8/94
GCMS#4	Hewlett-Packard	5972//5890E	3341A01395//3336A53831	9/94
GCMS#6	Hewlett-Packard	5972//5890E	3501A02346//3336A59437	8/95
P&T#1	Tekmar	3000	94104001	12/2000
P&T#2	Tekmar	3000	94067011	5/94
P&T#3	Tekmar	3000	94104006	8/94
P&T#4	Tekmar	3000	98197002	7/96
P&T#6	Tekmar	3000	95164011	8/95
Archon#1	Dynatech	5100A	11943-696A	5/95
PTA30#2	Dynatech	PTA30-W/S	11156-593E	5/94
PTA30#3	Dynatech	PTA30-W/S	11153-593E	8/94
PTA30#4	Dynatech	PTA30-W/S	11646-1194	9/94
Archon#6	Dynatech	5100	11768-695	8/95
Nanopure DI System	Barnstead-Thermoline	D 8961-Infinity	896991200089	2/2000
Balance, top loading	Sartorius	Sartorius Basic	11203133	5/93
Sonicator	Lab-line instru.Inc.	9333	0194-03	5/93

Table D

SUPPLIES

Supplies	Manufacturer	Vendor	Catalog #
10ul Gastight 1701	Hamilton	Fisher Scientific	14-815-1
25ul Gastight 1702	Hamilton	Fisher Scientific	14-815-29
50ul Gastight 1705	Hamilton	Fisher Scientific	14-824-30
100ul Gastight 1710	Hamilton	Fisher Scientific	13-684-100
250ul Gastight 1725	Hamilton	Fisher Scientific	13-684-102
500ul Gastight 1750	Hamilton	Fisher Scientific	13-684-106
1ml Gastight 1001	Hamilton	Fisher Scientific	14-824-25
5ml Gastight 1005	Hamilton	Fisher Scientific	13-684-96
50ml Gastight 1050	Hamilton	Fisher Scientific	14-815-195
DB-624 Capillary column, 20mX0.18 mm i.d.X1.0 um	J&W Scientific	VWR Scientific	121-1324
K-Trap, Vocarb3000, Tekmar3000	Supelco	Supelco	24920-U
Fritless 5-ml Sparge Tube	Supelco	Supelco	22780
IceBlue Septa, 11mm	Restek	Restek	22392
Single Gooseneck Injection port liners (4mm)	Restek	Restek	20799
Gold-plated inlet seals	Restek	Restek	21306
Viton O-rings	Restek	Restek	20377
0.4mm Vespel/Graphite ferrules	Restek	Restek	20211
GCMS Filaments	Agilent Technologies	Agilent Technologies	05972-60053
Stir Bar	Fisher Brand	Fisher Brand	14-511-60A
40 ml VOA vials	QEC	QEC	3112-40ml
10 ml volumetric	Kimax Brand	Fisher Scientific	10-212AA
25 ml volumetric	Kimax Brand	Fisher Scientific	10-212BB
50 ml volumetric	Kimax Brand	Fisher Scientific	10-212A
100 ml volumetric	Kimax Brand	Fisher Scientific	10-212B
200 ml volumetric	Kimax Brand	Fisher Scientific	10-212C
500 ml volumetric	Kimax Brand	Fisher Scientific	10-218D
Pasteur Pipettes	Fisher Scientific	Fisher Scientific	13-678-20A
Pipette bulb	Fisher Scientific	Fisher Scientific	14-065B
0.1-2.5 ml Repipetter	Brinkmann	Fisher Scientific	13-688-130
1-5 ml Repipetter	Brinkmann	Fisher Scientific	13-688-131
2-10 ml Repipetter	Brinkmann	Fisher Scientific	13-688-133
5-25 ml Repipetter	Brinkmann	Fisher Scientific	13-688-134
1.8 ml amber vials & caps	Restek	Restek	24637
10 ml graduate cylinder	Kimex Brand	Fisher Scientific	08-554B "to deliver"
40 ml VOA vials HCl preserved	QEC	QEC	3112-40HCL
2 oz. jars with teflon lids	QEC	QEC	2114-0002
spatulas	Fisher Scientific	Fisher Scientific	14-511-60A

Table E

REAGENTS

Reagent	Conc.	Purity	Manufacturer	Vendor	Catalog #
Methanol	100%	Purge and Trap grade	Burdick & Jackson	VWR Scientific	232-1
Sodium Bisulfate	granular	Certified grade	Fisher Scientific	Fisher Scientific	S-240-3
Helium gas		99.999%	Michigan Airgas	Michigan Airgas	
DFW		ASTM Type II	Barnstead-Nanopure	VWR Scientific	D8961
4-BFB Tuning Standard	5000 ug/mL	99%	Restek	Restek	30003-520
Ottawa Sand, 20-30 mesh		ASTM C190	Fisher Scientific	Fisher Scientific	S23-3
8260 Internal Standard	2500 ug/mL	99%	Restek	Restek	30173
8260 Surrogate Standard	2500 ug/mL	99%	Restek	Restek	30174
VOA Matrix Spike	2500 ug/mL	99%	Restek	Restek	30005-520
Custom VOC Mix Rev. 4/99	2000 ug/mL	99%	Restek	Restek	56241
Custom Acrolein Mix	20000 ug/mL	99%	Restek	Restek	56242
VOA Calibration Mix #1	5000 ug/mL	99%	Restek	Restek	30006
502.2 Cal 2000 Megamix	2000 ug/mL	99%	Restek	Restek	30431
2-Chloroethylvinyl Ether	2000 ug/mL	99%	Restek	Restek	30265
502.2 Calibration Mix#1	2000 ug/mL	99%	Restek	Restek	30042
Custom Cyclohexanes/Methyl Acetate Mix	10000 ug/mL	99%	Restek	Restek	58314
1,4-Dioxane Standard	2000 ug/mL	99%	Restek	Restek	30287
Diethoxymethane	neat	99%	Aldrich Chemical	Aldrich Chemical	16,6421
Vinyl Acetate	neat	99+%	Aldrich Chemical	Aldrich Chemical	V150-3

Table F

STANDARDS

Standard	Acronym	Concentration	Direction found in Section:	Alias
Level 1 Calibration Standard	VSTD001	1.0 ug/L	Table J	Cal Stnd
Level 2 Calibration Standard	VSTD005	5.0 ug/L	Table J	Cal Stnd
Level 3 Calibration Standard	VSTD020	20 ug/L	Table J	Cal Stnd
Level 4 Calibration Standard	VSTD050	50 ug/L	Table J	Cal Stnd
Level 5 Calibration Standard	VSTD100	100 ug/L	Table J	Cal Stnd
Level 6 Calibration Standard	VSTD200	200 ug/L	Table J	Cal Stnd
Independent Calibration Verification	ICV050	50 ug/L	Table J	Check Stnd
Calibration Verification Standard	VSTD050	50 ug/L	Table J	Check Stnd
Surrogate Standard	SS	50 ug/L	Table J	Surrogate
Internal Standard	IS	50 ug/L	Table J	Internal
Method Blank	MB	< Reporting Limit		Reagent Blank
BFB	Vtunxxx	50 ng injection	Table J	Tune Check
Matrix Spike	MS	50 ug/L	Table J	Matrix Spike
Matrix Spike Duplicate	MSD	50 ug/L	Table J	MSD
Blank Spike	BS	50 ug/L	Table J	Blank Spike
Blank Spike Duplicate	BSD	50 ug/L	Table J	BSD
Extraction Blank	EBLK	< Reporting Limit		Extraction Blank (MeOH)

Table G

SAMPLE COLLECTION, PRESERVATION, SHIPMENT AND STORAGE

Matrix	Prep Method	Container(s)	Preservation	Shipment Conditions	Lab Storage Conditions
Aqueous	SW846 5030B	40 ml VOC Vials (3)	0.5 ml of 18 % HCL	On ice 4+/- 2° Celsius	Refrigerate 4+/- 2° Celsius
Solid	SW846 5035	Encore samplers (two 5 gram samplers and one 25 gram sampler) or 40 ml unpreserved VOC vial	If sample exhibits effervescence, then 5 mls OFW. Non-effervescence = 5 mls OFW containing 1 g Sodium Bisulfate. Medium level with MeOH	On ice 4+/- 2° Celsius	• Refrigerate 4+/- 2° Celsius • Freeze within 48 hours of sampling, thaw, preserve with OFW or Sodium Bisulfate based on effervescence, then analyze
Solid	SW846 5030B	4 oz ZH packed tight	Water and/or MeOH	On ice 4+/- 2° Celsius	Refrigerate 4+/- 2° Celsius

Table H

QUALITY CONTROL

Preparation Method ⇨ Quality Control Measure ⇩	**SW846 5030**	**SW846 5030B**	**SW846 5035**	**EPA 624**	**WI Modified Soils & Waters**
Initial Calibration	Minimum of five levels; lowest level near but above MDL	Minimum of five levels; lowest level near but above MDL	Minimum of five levels; lowest level near but above MDL	Minimum of five levels; lowest level near but above MDL	Minimum of five levels; lowest level near but above MDL
Method Blank	One per batch of samples, up to 20 environmental samples, whichever is more frequent.	One per batch of samples, up to 20 environmental samples, whichever is more frequent.	One per batch of samples, up to 20 environmental samples, whichever is more frequent.	One per batch of samples, up to 20 environmental samples, whichever is more frequent.	One per batch of samples, up to 20 environmental samples, whichever is more frequent.
Continuing Calibration Verification	Mid-level calibration standards run at the beginning of each 12 hour time clock.	Mid-level calibration standards run at the beginning of each 12 hour time clock.	Mid-level calibration standards run at the beginning of each 12 hour time clock.	Mid-level calibration standards run at the beginning of each 12 hour time clock.	Mid-level calibration standards run at the beginning of each 12 hour time clock.
Independent Calibration Verification	Mid-level calibration standard from an independent source to verify initial calibration curve.	Mid-level calibration standard from an independent source to verify initial calibration curve.	Mid-level calibration standard from an independent source to verify initial calibration curve.	Mid-level calibration standard from an independent source to verify initial calibration curve.	Mid-level calibration standard from an independent source to verify initial calibration curve.
Laboratory Control Spike and Duplicate	One pair per batch of samples, up to 20 environmental samples, whichever is more frequent.	One pair per batch of samples, up to 20 environmental samples, whichever is more frequent.	One pair per batch of samples, up to 20 environmental samples, whichever is more frequent.	One pair per batch of samples, up to 20 environmental samples, whichever is more frequent.	One pair per batch of samples, up to 20 environmental samples, whichever is more frequent.
Matrix Spike and Duplicate	One pair per batch of samples, up to 20 environmental samples, whichever is more frequent.	One pair per batch of samples, up to 20 environmental samples, whichever is more frequent.	One pair per batch of samples, up to 20 environmental samples, whichever is more frequent.	One pair per batch of samples, up to 20 environmental samples, whichever is more frequent.	One pair per batch of samples, up to 20 environmental samples, whichever is more frequent.
Internal Standards	Added to every sample.	Added to every sample.	Added to every sample.	Added to every sample.	Added to every sample.
Surrogate Standards	Added to every sample.	Added to every sample.	Added to every sample.	Added to every sample.	Added to every sample.

Table I

CALIBRATION AND STANDARDIZATION

Analytical Method Acceptance Criteria⇒ Calibration Measure ⇓	Frequency	Accpetance Criteria
Initial Calibration	• Installation of new column or trap • Creation of new analytical method • CVS fails criteria • BFB tuning criteria fails and GCMS is retuned • GCMS is retuned	• % RSD ≤ 15% for every compound; 30% for CCCs • RF SPCCs = 0.300 for: Chlorobenzene, 1,1,2,2-Tetrachloroethane • RF= 0.100 for: Chloromethane, 1,1-Dichloroethane, Bromoform • Use of Average Response factors if: %RSD is ≤15% for all reportable compounds or the mean % RSD of all calibrated compounds is ≤15%. Otherwise, use Linear Regression or Quadratic Fit and Correlation Coefficient is ≥ 0.9900.
Independent Calibration Verification Standard	• After every valid initial calibration	• RF of ICVS must be ≤ 20% from initial calibration.
Continuing Calibration Verification	• At the beginning of every 12 hour time clock or analytical sequence.	• RF of CCV must be ≤ 20% from initial calibration.
Laboratory Control Spike and Duplicate	• One pair per batch of samples, up to 20 environmental samples, whichever is more frequent. Fortify with 5 classic spikes	• Recovery must be within 85-115 % for all compounds.
Matrix Spike and Duplicate	• One pair per batch of samples, up to 20 environmental samples, whichever is more frequent. Fortify with all target compounds.	• In house limits determined. See Table in Appendix F for compound criteria.
Internal Standards	• Added to every sample.	• Retention Time must be ± 30 seconds from the last CVS • Area of IS must be -50% to +100%
Surrogate Standards	• Added to every sample.	• In house limits determined. See Table in Appendix F for compound criteria.

Table J

PREPARATION OF INTERMEDIATE STANDARDS

Standard	Acronym	Concentration of Intermediate	Reagents Used	Final Volume
4-Bromofluorobenzene	BFB	50 ug/mL	100µL of 5000 ug/mL BFB into methanol	10 mL
Internal Standard	IS	250 ug/mL	5000 µL of 2500 ug/mL IS into methanol	50 mL
Surrogate Standard	SS	250 ug/mL	5000 µL of 2500 ug/mL SS into methanol	50 mL
Internal/Surrogate Stnd.	IS/SS	250 ug/mL	5000 µL of 2500 ug/mL IS/SS into MeOH	50 mL
100 ug/mL Calibration Stnd.	CAL	100 ug/mL	1250 µL of 2000 ug/mL Custom Voa Mix 1250 µL of 2000 ug/mL 502.2 Cal 2000 1250 µL of 2000 ug/mL 2-Chloroethylvinyl ether 1250 µL of 2000 ug/mL 502.2 Cal. Mix #1 125 µL of 20000 ug/mL Custom Acrolein 500 µL of 5000 ug/mL VOA Cal. Mix #1 263 µL of 9520 ug/mL Vinyl Acetate 246 µL of 10168 ug/mL Diethoxymethane	25 mL
Vinyl Acetate	VA	9520 ug/mL	0.238 mg of neat Vinyl Acetate	25 mL
Diethoxymethane	DEM	10168 ug/mL	0.254 mg of neat Diethoxymethane	25 mL
100 ug/mL Matrix Spike (2nd lot #) - Water	MS	100 ug/mL	1250 µL of 2000 ug/mL Custom Voa Mix 1250 µL of 2000 ug/mL 502.2 Cal 2000 1250 µL of 2000 ug/mL 2-Chloroethylvinyl ether 1250 µL of 2000 ug/mL 502.2 Cal. Mix #1 125 µL of 20000 ug/mL Custom Acrolein 500 µL of 5000 ug/mL VOA Cal. Mix #1 263 µL of 9520 ug/mL Vinyl Acetate 246 µL of 10168 ug/mL Diethoxymethane	25 mL
100 ug/mL Blank Spike	BS	100 ug/mL	1000 µL of 2500 ug/mL CLP Matrix Spike	25 mL
500 ug/mL Matrix Spike - MeOH Extracts	MS#1	500 ug/mL	2500 µL of 2000 ug/mL 502.2 Cal. Mix #1 2500 µL of 2000 ug/mL 2-Chloroethylvinyl ether 2500 µL of 2000 ug/mL 502.2 Cal 2000	10 mL
500 ug/mL Matrix Spike - MeOH Extracts	MS#2	500 ug/mL	250 µL of 20000 ug/mL Custom Acrolein 2500 µL of 2000 ug/mL Custom Voa Mix 1000 µL of 5000 ug/mL VOA Cal. Mix #1 105 µL of 9520 ug/mL Vinyl Acetate 94 µL of 10168 ug/mL Diethoxymethane	10 mL

Table K

En Chem Initial Demonstration of Capability

Compound/Element	Acceptable Recov. from Authoritative Method 8260B	Acceptable Recov. from Authoritative Method 624	En Chem Initial Demonstration Recovery
Dichlorodifluoromethane	80-120%		88.4%
Chloromethane	80-120%	D-273%	77.2%
Vinyl Chloride	80-120%	D-251%	81.2%
Bromomethane	80-120%	D-242%	84.5%
Chloroethane	80-120%	D-230%	81.0%
Dichlorofluoromethane	80-120%		88.7%
Trichlorofluoromethane	80-120%	17-181%	93.5%
Diethyl Ether	80-120%		82.9%
Acrolein	40-160%		131.7%
1 1-Dichloroethane	80-120%	D-234%	86.8%
1 1 2-Trichlorotrifluoroethane	80-120%		88.7%
Acetone	40-160%		80.4%
Iodomethane	80-120%		89.0%
Carbon Disulfide	80-120%		91.3%
Allyl Chloride	80-120%		83.5%
Methylene Chloride	80-120%	D-221%	88.4%
Acrylonitrile	40-160%		81.0%
Trans-1 2-Dichloroethene	80-120%	54-156%	89.9%
Methyl T-Butyl Ether	80-120%		88.5%
N-Hexane	80-120%		70.8%
1 1-Dichloroethane	80-120%	59-155%	88.2%
Vinyl Acetate	40-160%		84.6%
Diisopropyl Ether	80-120%		83.0%
2 2-Dichloropropane	80-120%		71.5%
Cis-1 2-Dichloroethene	80-120%		88.6%
2-Butanone	40-160%		70.2%
Bromochloromethane	80-120%		94.7%
Tetrahydrofuran	40-160%		78.5%
Chloroform	80-120%	51-138%	91.8%
1 1 1-Trichloroethane	80-120%	52-162%	90.9%
Carbon Tetrachloride	80-120%	70-140%	93.2%
1 1-Dichloropropene	80-120%		80.9%
Benzene	80-120%	37-151%	86.5%
1 2-Dichloroethane	80-120%	49-155%	98.0%
Diethoxymethane	80-120%		84.2%
Trichloroethene	80-120%	71-157%	91.9%

Compound/Element	Acceptable Recov. from Authoritative Method 8260B	Acceptable Recov. from Authoritative Method 624	En Chem Initial Demonstration Recovery
1 2-Dichloropropane	80-120%	D-210%	90.2%
Dibromomethane	80-120%		96.6%
Bromodichloromethane	80-120%	35-155%	96.5%
2-Chloroethyl Vinyl Ether	80-120%	D-305%	89.5%
Cis-1 3-Dichloropropene	80-120%	D-227%	85.8%
4-Methyl-2-Pentanone	40-160%		85.3%
Toluene	80-120%	47-150%	94.5%
Trans-1 3-Dichloropropene	80-120%	17-183%	89.7%
1 1 2-Trichloroethane	80-120%	52-150%	97.5%
Tetrachloroethene	80-120%	64-148%	96.5%
1 3-Dichloropropane	80-120%		97.4%
2-Hexanone	40-160%		73.2%
Dibromochloromethane	80-120%	53-149%	100.5%
1 2-Dibromoethane	80-120%		101.4%
Chlorobenzene	80-120%	37-160%	101.3%
1 1 1 2-Tetrachloroethane	80-120%		97.2%
Ethyl Benzene	80-120%	37-162%	93.6%
M- P-Xylene	80-120%		97%
O-Xylene	80-120%		98.0%
Styrene	80-120%		101.0%
Bromoform	80-120%	45-169%	97.7%
Isopropylbenzene	80-120%		94.5%
Trans-1 4-Dichloro-2-Butene	80-120%		86.0%
Bromobenzene	80-120%		101.4%
1 1 2 2-Tetrachloroethane	80-120%	46-157%	99.7%
1 2 3-Trichloropropane	80-120%		97.4%
Cis-1 4-Dichloro-2-Butene	80-120%		87.7%
N-Propylbenzene	80-120%		91.5%
2-Chlorotoluene	80-120%		96.4%
4-Chlorotoluene	80-120%		97.7%
1 3 5-Trimethylbenzene	80-120%		95.8%
Tert-Butylbenzene	80-120%		93.9%
1 2 4-Trimethylbenzene	80-120%		96.3%
Sec-Butylbenzene	80-120%		90.2%
1 3-Dichlorobenzene	80-120%	59-156%	99.0%
P-Isopropyltoluene (Cymene)	80-120%		91.9%
1 4-Dichlorobenzene	80-120%	18-190%	100.4%
1 2-Dichlorobenzene	80-120%	18-190%	100.6%
N-Butylbenzene	80-120%		88.1%

Compound/Element	Acceptable Recov. from Authoritative Method 8260B	Acceptable Recov. from Authoritative Method 624	En Chem Initial Demonstration Recovery
Hexachloroethane	80-120%		88.9%
1 2-Dibromo-3-Chloropropane	80-120%		91.7%
1 2 4-Trichlorobenzene	80-120%		96.0%
Hexachlorobutadiene	80-120%		93.8%
Naphthalene	80-120%		94.9%
1 2 3-Trichlorobenzene	80-120%		99.4%
2-Methylnaphthalene	80-120%		85.7%
Total 1,2-Dichloroethene	80-120%		89.2%
Methyl Methacrylate			88.4%
Ethyl Methacrylate			89.2%

D-Detectable Recovery

Table K1
Surrogate Recovery Limits

Preparation Method ⇨ Effective Date: June 2001	SW846 5030		SW846 5030B		SW846 5035		WI Modified Waters		WI Modified Soils		EPA 624	
Surrogate Spike Recovery Parameters ⇩	LCL	UCL	LCL	UCL	LCL	UCL	LCL	UCL	LCL	UCL	LCL	UCL
Dibromofluoromethane	67	112	68	125	67	112	68	125	63	131	68	125
Toluene-d8	76	114	70	128	76	114	70	128	68	131	70	128
4-Bromofluorobenzene	75	113	61	127	75	113	61	127	65	124	61	127

Table K2
Matrix Spike Recovery Limits

Preparation Method ⇨ Effective Date: June 2001	SW846 5030			SW846 5030B			SW846 5035			WI Modified Waters			WI Modified Soils[b]			EPA 624		
Matrix Spike Recovery Parameters ⇩	LCL	UCL	RPD	LCL	UCL	RPD	LCL	UCL	RPD	LCL	UCL	RPD	LCL	UCL	RPD	LCL	UCL	RPD
Chloromethane	49	135	12	36	141	11	49	135	12	36	141	11	-	-	-	36	141	11
Bromomethane	51	147	14	44	128	12	51	147	14	44	128	12	-	-	-	44	128	12
Dichlorodifluoromethane	10	160	13	3	151	17	10	160	13	3	151	17	-	-	-	3	151	17
Vinyl Chloride	55	137	12	53	140	10	55	137	12	53	140	10	-	-	-	53	140	10
Chloroethane	62	137	11	68	127	10	62	137	11	68	127	10	-	-	-	68	127	10
Methylene Chloride	58	137	18	71	118	10	58	137	18	71	118	10	-	-	-	71	118	10
Trichlorofluoromethane	68	138	11	72	133	10	68	138	11	72	133	10	-	-	-	72	133	10
Acetone	17	269	26	54	129	19	17	269	26	54	129	19	-	-	-	54	129	19
1,1-Dichloroethene[a]	65	138	11	77	123	10	65	138	11	77	123	10	-	-	-	77	123	10
Trans-1,2-Dichloroethene	66	131	11	77	119	11	66	131	11	77	119	11	-	-	-	77	119	11
Methyl Tert-butyl ether	57	131	27	77	109	10	57	131	27	77	109	10	-	-	-	77	109	10
1,1-Dichloroethane	68	129	11	81	120	10	68	129	11	81	120	10	-	-	-	81	120	10
2-Butanone	16	206	17	55	125	21	16	206	17	55	125	21	-	-	-	55	125	21
Di-isopropyl ether	70	126	10	77	112	10	70	126	10	77	112	10	-	-	-	77	112	10
Cis-1,2-Dichloroethene	67	127	10	81	113	15	67	127	10	81	113	15	-	-	-	81	113	15
2,2-Dichloropropane	67	133	11	79	127	10	67	133	11	79	127	10	-	-	-	79	127	10
Bromochloromethane	67	125	10	83	107	10	67	125	10	83	107	10	-	-	-	83	107	10
Chloroform	66	129	10	83	118	10	66	129	10	83	118	10	-	-	-	83	118	10
1,1,1-Dichloroethane	71	131	12	82	126	10	71	131	12	82	126	10	-	-	-	82	126	10
Carbon Tetrachloride	72	131	12	75	132	10	72	131	12	75	132	10	-	-	-	75	132	10
1,1-Dichloropropene	68	135	10	87	121	10	68	135	10	87	121	10	-	-	-	87	121	10
Bromodichloromethane	65	126	10	84	113	10	65	126	10	84	113	10	-	-	-	84	113	10
1,2-Dichloropropane	69	126	10	86	113	10	69	126	10	86	113	10	-	-	-	86	113	10
Dibromomethane	65	125	10	79	114	10	65	125	10	79	114	10	-	-	-	79	114	10
Trichloroethene[a]	69	132	10	88	114	10	69	132	10	88	114	10	-	-	-	88	114	10
Benzene[a]	59	133	12	89	111	10	59	133	12	89	111	10	-	-	-	89	111	10
1,2-Dichloroethane	68	126	10	75	123	10	68	126	10	75	123	10	-	-	-	75	123	10
1,1,2-Trichloroethane	59	130	12	82	108	10	59	130	12	82	108	10	-	-	-	82	108	10
1,2-Dibromoethane	52	132	10	82	110	10	52	132	10	82	110	10	-	-	-	82	110	10
Toluene[a]	63	131	10	91	110	10	63	131	10	91	110	10	-	-	-	91	110	10
Chlorobenzene[a]	64	131	13	90	111	10	64	131	13	90	111	10	-	-	-	90	111	10
1,1,1,2-Tetrachloroethane	71	127	15	84	117	10	71	127	15	84	117	10	-	-	-	84	117	10
Cis-1,3-Dichloropropene	70	125	13	84	120	10	70	125	13	84	120	10	-	-	-	84	120	10
Trans-1,3-Dichloroprene	63	128	13	83	121	10	63	128	13	83	121	10	-	-	-	83	121	10
Tetrachloroethene	71	142	15	87	123	10	71	142	15	87	123	10	-	-	-	87	123	10
1,3-Dichloropropane	68	129	14	84	119	10	68	129	14	84	119	10	-	-	-	84	119	10
Dibromochloromethane	66	127	14	80	118	10	66	127	14	80	118	10	-	-	-	80	118	10
Ethylbenzene	66	135	14	92	118	10	66	135	14	92	118	10	-	-	-	92	118	10
Styrene	54	137	12	91	116	10	54	137	12	91	116	10	-	-	-	91	116	10

Preparation Method ⇨	SW846 5030			SW846 5030B			SW846 5035			WI Modified Waters			WI Modified Soils			EPA 624		
Matrix Spike Recovery Parameters ⇩	LCL	UCL	RPD	LCL	UCL	RPD	LCL	UCL	RPD	LCL	UCL	RPD	LCL	UCL	RPD	LCL	UCL	RPD
Bromoform	48	136	15	75	113	10	48	136	15	75	113	10	-	-	-	75	113	10
M/P-Xylene	54	145	13	93	118	10	54	145	13	93	118	10	-	-	-	93	118	10
O-Xylene	66	133	14	91	117	10	66	133	14	91	117	10	-	-	-	91	117	10
Isopropylbenzene	60	148	14	87	125	10	60	148	14	87	125	10	-	-	-	87	125	10
Bromobenzene	60	135	15	85	116	10	60	135	15	85	116	10	-	-	-	85	116	10
1,2,3-Trichloropropane	64	138	15	61	128	22	64	138	15	61	128	22	-	-	-	61	128	22
1,1,2,2-Tetrachloroethane	60	137	14	68	129	11	60	137	14	68	129	11	-	-	-	68	129	11
2-Chlorotoluene	60	145	15	88	119	14	60	145	15	88	119	14	-	-	-	88	119	14
N-propylbenzene	61	152	12	85	127	10	61	152	12	85	127	10	-	-	-	85	127	10
4-Chlorotoluene	57	144	13	85	121	10	57	144	13	85	121	10	-	-	-	85	121	10
1,3,5-Trimethylbenzene	45	163	16	88	123	10	45	163	16	88	123	10	-	-	-	88	123	10
Tert-Butylbenzene	68	141	15	88	123	10	68	141	15	88	123	10	-	-	-	88	123	10
1,2,4-Trimethylbenzene	65	139	14	88	122	10	65	139	14	88	122	10	-	-	-	88	122	10
Sec-butylbenzene	56	152	14	87	126	10	56	152	14	87	126	10	-	-	-	87	126	10
1,3-Dichlorobenzene	58	133	14	84	115	10	58	133	14	84	115	10	-	-	-	84	115	10
1,4-Dichlorobenzene	54	133	14	83	114	10	54	133	14	83	114	10	-	-	-	83	114	10
P-isopropyltoluene	51	153	15	88	124	10	51	153	15	88	124	10	-	-	-	88	124	10
1,2-Dichlorobenzene	52	133	14	83	118	10	52	133	14	83	118	10	-	-	-	83	118	10
N-Butylbenzene	51	148	20	84	132	10	51	148	20	84	132	10	-	-	-	84	132	10
1,2-Dibromo-3-chloropropane	41	148	24	54	145	14	41	148	24	54	145	14	-	-	-	54	145	14
1,2,4-Trichlorobenzene	24	142	12	82	115	10	24	142	12	82	115	10	-	-	-	82	115	10
Naphthalene	21	142	27	67	134	11	21	142	27	67	134	11	-	-	-	67	134	11
Hexachlorobutadiene	27	155	20	68	129	11	27	155	20	68	129	11	-	-	-	68	129	11
1,2,3-Trichlorobenzene	22	143	14	80	118	10	22	143	14	80	118	10	-	-	-	80	118	10
2-Methylnaphthalene	Dr	151	27	36	186	15	Dr	151	27	36	186	15	-	-	-	36	186	15

a - For preparation methods SW8466 5030; SW8466 5030E or SW8466 5035 the 5 classic spikes are used to dertermined compliance unless project specific requirements are requested. It is recommended that if project specific requirements are needed in-house limits as listed are used.

b - This prepatory method does not include hte use of matrix spikes, However, it does rely on a Laboratory Control Spike pair to determine extraction effeciency, accuracy and precision

Dr - Detectable recovery. Poor purging compound.

Table L
ANALYST/TECHNICIAN DATA ASSESSMENT

Analytical Method Acceptance Criteria⇨ Data Assessment Measure ⇩	Method Citation SW If these conditions are not achieved ⇒	Method Citation EPA If these conditions are not achieved ⇒
Initial Calibration	• 1	• 1
Initial Calibration Verification	• 1	• 1
Calibration Blank	• 1	• 1
Method Blank	• 2	• 2
Internal Standard Area	• 3	• 3
Continuing Calibration Verifications	• 4	• 4
Surrogate Recovery	• 5	• 5
Accuracy & Precision Laboratory Control Spikes	• 6	• 6
Accuracy & Precision Matrix Spikes	• 7	• 7

1. Check instrumentation/equipment condition; enter maintenance in instrument log; perform another initial calibration. No data can be reported. Generate on Non-Conformance Memo.

2. In the absence of project specific requirements, sample detects greater than 20 times the method blank contamination level are reported with those not exceeding 20 times re-analyzed or qualified. Generate on Non-Conformance Memo.

3. In the absence of project specific requirements, check to be sure that there are no errors in the calculations. If errors are found, recalculate the data accordingly. Examine chromatograms for interfering peaks and integrated peak areas. Re analyze sample if volume allows. Generate on Non-Conformance Memo.

4. In the absence of project specific requirements, immediately after the flawed CCV analyze another CCV or re-analyzed all samples set after the last acceptable CCV. Generate on Non-Conformance Memo.

5. In the absence of project specific requirements, check to be sure that there are no errors in the calculations. If errors are found, recalculate the data accordingly. Examine chromatograms for interfering peaks and integrated peak areas. If the program allows report the concentrations noting the surrogate recovery is most likely due to co-elution with target compounds. Some samples may require dilution in order to bring one or more target analytes within the calibration range or to overcome significant interferences with some analytes. Re analyze sample if volume allows. If the surrogate recoveries are available from a less-diluted or undiluted aliquot of the sample or sample extract, those recoveries may be used to demonstrate that the surrogates were within the in-house QC limits, and no further action is required. If, upon re-analysis the recovery is again not within limits, report the data as an "estimated concentration." If the recovery is within the limits in the re-analysis, provide the re-analysis data to the data user. Generate on Non-Conformance Memo.

6. In the absence of project specific requirements, the LCS and LCSD are spiked with the same analytes at the same concentrations as the matrix spike. When the results of the matrix spike analysis indicate a potential problem due to the sample matrix itself, the LCS results are used to verify that the laboratory can perform the analysis in a clean matrix. If the LCS and LCSD recoveries do not meet those limits as required in the method or program, the samples shall be re-analyzed. If sample volume does not allow re-analysis the entire prep/analytical batch of samples shall be flagged to reflect the deficiencies. Generate on Non-Conformance Memo.

7. In the absence of project specific requirements, when the results of the matrix spike analysis indicate a potential problem due to the sample matrix itself, the LCS results are used to verify that the laboratory can perform the analysis in a clean matrix. If sample volume allows re-analysis, it shall be performed to verify the matrix effect. When sample volume does not re-analysis, the parent sample and samples from that project site shall be flagged to reflect the deficiencies. Generate a Non- Conformance Memo.

Table M
ANALYST/SUPERVISOR DATA ASSESSMENT

Analytical Method Acceptance Criteria⇨ Data Assessment Measure ⇩	**Method Citation SW If these conditions are not achieved ⇒**	**Method Citation EPA If these conditions are not achieved ⇒**
Initial Calibration	• 1	• 1
Initial Calibration Verification	• 1	• 1
Calibration Blank	• 1	• 1
Method Blank	• 2	• 2
Internal Standard Area	• 3	• 3
Continuing Calibration Verifications	• 4	• 4
Surrogate Recovery	• 5	• 5
Accuracy & Precision Laboratory Control Spikes	• 6	• 6
Accuracy & Precision Matrix Spikes	• 7	• 7

1. Check instrumentation/equipment condition; enter maintenance in instrument log; perform another initial calibration. No data can be reported. Generate on Non-Conformance Memo.

2. In the absence of project specific requirements, sample detects greater than 20 times the method blank contamination level are reported with those not exceeding 20 times re-analyzed or qualified. Generate on Non-Conformance Memo.

3. In the absence of project specific requirements, check to be sure that there are no errors in the calculations. If errors are found, recalculate the data accordingly. Examine chromatograms for interfering peaks and integrated peak areas. Re analyze sample if volume allows. Generate on Non-Conformance Memo.

4. In the absence of project specific requirements, immediately after the flawed CCV analyze another CCV or re-analyzed all samples set after the last acceptable CCV. Generate on Non-Conformance Memo.

5. In the absence of project specific requirements, check to be sure that there are no errors in the calculations. If errors are found, recalculate the data accordingly. Examine chromatograms for interfering peaks and integrated peak areas. If the program allows report the concentrations noting the surrogate recovery is most likely due to co-elution with target compounds. Some samples may require dilution in order to bring one or more target analytes within the calibration range or to overcome significant interferences with some analytes. Re analyze sample if volume allows. If the surrogate recoveries are available from a less-diluted or undiluted aliquot of the sample or sample extract, those recoveries may be used to demonstrate that the surrogates were within the in-house QC limits, and no further action is required. If, upon re-analysis the recovery is again not within limits, report the data as an "estimated concentration." If the recovery is within the limits in the re-analysis, provide the re-analysis data to the data user. Generate on Non-Conformance Memo.

6. In the absence of project specific requirements, the LCS and LCSD are spiked with the same analytes at the same concentrations as the matrix spike. When the results of the matrix spike analysis indicate a potential problem due to the sample matrix itself, the LCS results are used to verify that the laboratory can perform the analysis in a clean matrix. If the LCS and LCSD recoveries do not meet those limits as required in the method or program, the samples shall be re-analyzed. If sample volume does not allow re-analysis the entire prep/analytical batch of samples shall be flagged to reflect the deficiencies. Generate on Non-Conformance Memo.

7. In the absence of project specific requirements, when the results of the matrix spike analysis indicate a potential problem due to the sample matrix itself, the LCS results are used to verify that the laboratory can perform the analysis in a clean matrix. If sample volume allows re-analysis, it shall be performed to verify the matrix effect. When sample volume does not re-analysis, the parent sample and samples from that project site shall be flagged to reflect the deficiencies. Generate a Non- Conformance Memo.

Table N
REPORT REVIEWER/PROJECT MANAGEMENT DATA ASSESSMENT

Analytical Method Acceptance Criteria⇨ Data Assessment Measure ⇩	**Method Citation SW If these conditions are not achieved ⇒**	**Method Citation EPA If these conditions are not achieved ⇒**	**Method Citation EPA If these conditions are not achieved ⇒**
Holding Time Compliance	• 1	• 1	•
Sample Receipt Conditions and Preservation	• 2	• 2	•
Method Citation	• 3	• 3	•
Reasonableness: Field Duplicate	• 4	• 4	•
Reasonableness: Analytical Comments Qualifications	• 5	• 5	•

1. Flag results with an H and the number of days past hold in parenthesis e.g. H(5).

2. In prose describe the receipt conditions as they relate to the acceptance criteria list in Table G

3. Compare to regulatory program in QAPjP or Chain of Custody.

4. Are duplicate with 40% agreement. If not, internally flag for review.

5. Do related suites of tests agree or are reasonable.

Table O
CORRECTIVE ACTIONS

Analytical Method Acceptance Criteria⇒ Quality Control Measure ⇓	Method Citation SW If these conditions are not achieved ⇒	Corrective Action (Key below)	Method Citation EPA If these conditions are not achieved ⇒	Corrective Action (Key below)
Initial Calibration		• 1	•	• 1
Method Blank		• 2	•	• 2
Continuing Calibration Verification		• 3	•	• 3

1. Check instrumentation/equipment condition; enter maintenance in instrument log; perform another initial calibration

2. Samples detects greater than 20 times the method blank contamination level are reported with those not exceeding 20 times re-analyzed or qualified.

3. Immediately after the flawed CCV analyze another CCV or re-analyzed all samples set after the last acceptable CCV.

Table P
CONTINGENCIES FOR HANDLING OUT-OF-CONTROL OR UNACCEPTABLE DATA

Analytical Method Acceptance Criteria⇨ Quality Control Measure ⇩	**Method Citation SW If these conditions are not achieved ⇒**	**Corrective Action (Key below)**	**Method Citation EPA If these conditions are not achieved ⇒**	**Corrective Action (Key below)**
Matrix Spike and Duplicate	•	• 1	•	• 1
Internal Standards	•	• 2	•	• 2
Surrogate Standards	•	• 2	•	• 2

1. If Lab Control Spikes pass acceptance criteria, flag parent sample and samples from the entire project with a QC batch associated with analysis indicated matrix effects.

2. When sample volume is available reanalyze the samples. If the second analysis indicates the same internal standard recoveries add analytical comments and effects on data.

Table Q
DATA QUALIFIERS

B	Analyte is present in the method blank. Method blank criteria is evaluated to the laboratory method detection limit. Additionally, method blank acceptance may be based on project specific criteria or determined from analyte concentrations in the sample and are evaluated on a sample by sample basis.
C	Elevated detection limit (see Sample Narrative).
D	Analyte value from diluted analysis.
E	Analyte concentration exceeds calibration range (see Sample Narrative).
F	Surrogate results outside control criteria or not available due to sample dilution.
H(n)	Extraction or analysis performed n days past holding time.
J	Qualitative evidence of analyte present: concentration detected is greater than the method detection limit but less than the reporting limit.
K	Detection limit may be elevated due to the presence of an unrequested analyte (see Sample Narrative).
N	Spiked sample recovery not within control limits.
P	The relative percent difference between the two GC columns for detected concentrations was greater than 40%.
Q	The analyte has been detected between the limit of detection (LOD) and limit of quantitation (LOQ). The results are qualified due to the uncertainty of analyte concentrations within this range.
U	The analyte was not detected above the reporting limit.
W	Sample received with headspace.
X	See Sample Narrative.
&	Laboratory Control Spike recovery not within control limits.
*	Duplicate analyses not within control limits.

Table R
Chromatographic Retention Times and Characteristic Masses (m/z)

Compound	Retention Time	Primary Ion	Secondary Ions
Dichlorodifluoromethane	1.64	85	87
Chloromethane	1.85	50	52
Vinyl Chloride	1.98	62	64
Bromomethane	2.39	94	96
Chloroethane	2.53	64	66
Trichlorofluoromethane	2.87	151	101,153
Acrolein	3.47	56	55,58
Iodomethane	3.84	142	127,141
Acetonitrile**	---	41	40,39
Carbon Disulfide	3.93	76	78
Methyl-t-butyl ether	5.05	73	57
Allyl Chloride	4.23	76	41,39,78
Methylene Chloride	4.45	84	86,49
1,1-Dichloroethene	3.63	96	61,63
Diethyl Ether	3.37	74	45,59
Acetone	3.73	58	43
trans-1,2-Dichloroethene	4.93	96	61,98
Acrylonitrile	4.87	53	52,51
1,1-Dichloroethane	3.63	63	65,83
Vinyl Acetate	5.78	43	86
2,2-Dichloropropane	6.60	77	97
2-Butanone	6.66	72	43
cis-1,2-Dichloroethene	6.62	96	61,98
Propionitrile**(ethyl cyanide)	---	54	52,55,40
Compound	**Retention Time**	**Primary Ion**	**Secondary Ions**

Chloroform	7.17	83	85
Bromochloromethane	6.99	128	49,130
Methacrylonitrile**	---	41	67,39,52,66
1,1,1-Trichloroethane	7.44	97	99,61
Carbon Tetrachloride	7.71	117	119
1,1-Dichloropropene	7.72	75	110,77
Benzene	8.04	78	-
1,2-Dichloroethane	8.07	62	98
Trichloroethene	9.11	95	97,130,132
1,2-Dichloropropane	8.07	63	112
Bromodichloromethane	9.93	83	85,127
Dibromomethane	9.63	93	95,174
Methyl Methacrylate	9.83	69	41,100,39
1,4-Dioxane**	---	88	58,43,57
2-Chloroethylvinyl ether	10.46	63	65,106
4-Methyl-2-pentanone	10.94	100	43,58,85
Toluene	11.21	92	91
trans-1,3-Dichloropropene	11.60	75	77,39
cis-1,3-Dichloropropene	17.90	75	77,39
1,1,2-Trichloroethane	11.89	83	97,85
Ethyl Methacrylate	11.91	69	41,99,86,114
2-Hexanone	12.34	43	58,57,100
Tetrachloroethene	12.14	164	129,131,166
1,3-Dichloropropane	12.16	76	78
Dibromochloromethane	12.54	129	127
1,2-Dibromoethane	12.70	107	109,188
Chlorobenzene	13.56	112	77,114
Compound	**Retention Time**	**Primary Ion**	**Secondary Ions**
1,1,1,2-Tetrachloroethane	13.72	131	133,119

Ethylbenzene	13.78	91	106
m/p-Xylene	13.99	106	91
o-Xylene	14.66	106	91
Styrene	14.68	104	78
Bromoform	14.97	173	175,254
Isopropylbenzene(Cumene)	15.32	105	120
cis-1,4-Dichloro-2-butene	15.94	75	53,77,124,89
1,1,2,2-Tetrachloroethane	15.84	83	131,85
Bromobenzene	15.80	156	77,158
1,2,3-Trichloropropane	15.89	75	77
n-Propylbenzene	16.05	91	120
2-Chlorotoluene	16.17	91	126
trans-1,4-Dichloro-2-butene	15.42	53	88,75
1,3,5-Trimethylbenzene	16.38	105	120
4-Chlorotoluene	16.36	91	126
Pentachloroethane**	---	167	130,132,165,169
1,2,4-Trimethylbenzene	17.03	105	120
sec-Butylbenzene	17.34	105	134
tert-Butylbenzene	16.94	119	91,134
p-Isopropyltoluene	17.61	119	134,91
1,3-Dichlorobenzene	17.49	146	111,148
1,4-Dichlorobenzene	17.65	146	111,148
n-Butylbenzene	18.34	91	92,134
1,2-Dichlorobenzene	18.30	146	111,148
1,2-Dibromo-3-chloropropane	19.69	75	155,157
1,2,4-Trichlorobenzene	21.21	180	182,145
Compound	**Retention Time**	**Primary Ion**	**Secondary Ions**
Hexachlorobutadiene	21.58	225	223,227
Hexachloroethane	18.90	201	166,199,203

Naphthalene	21.63	128	-
1,2,3-Trichlorobenzene	22.08	180	182,145
Internal Standards/Surrogates			
Pentafluorobenzene	7.55	168	-
1,4-Difluorobenzene	8.71	114	-
Chlorobenzene-d5	13.51	117	-
1,4-Dichlorobenzene-d4	17.61	152	-
4-Bromofluorobenzene	15.56	95	174,176
Dibromofluoromethane	7.42	113	-
Toluene-d8	11.11	98	-

En Chem, Inc.

Quality Assurance Document

SET No: 1

STANDARD OPERATING PROCEDURE

DEPARTMENT: Inorganic – Metals

INSTRUMENT TYPE: Inductively Coupled Plasma – Atomic Emission Spectroscopy

APPLICATION: Dissolved waters. Digested sample matrices including water, soils, biota, industrial wastes, sludges, sediments, solid wastes, and leaching extracts for the determination of metals.

REFERENCES: USEPA Test Methods for Evaluating Solid Wastes SW-846, 3rd Edition, Method 6010B Revision 2 (December 1996)
EPA Method 200.7 Rev. 4.4

PROCEDURE SUMMARY:

Prior to analysis, samples are digested using appropriate sample preparation method (See SW 846 Methods 3005A, 3010A, 3050B, 3015, and 3051). Samples are introduced by a peristaltic pump, nebulized, and the resulting aerosol introduced to the plasma torch. In the plasma, element specific atomic line emission spectra are produced. A grating disperses the spectra and the intensities of the lines are measured by photomultiplier tube. Background correction is required for trace metal analysis. The background points selected may be on either or both sides of the analyte line of interest and should be in an area free of spectral interference.

Detection Limits:
See Appendix 1.

Range of Measurement:
See Appendix 1.

APPROVED BY: Tim Gramling — 1-4-2002
Tim Gramling, Metals Technical Advisor — Date

Julie A. Trivedi — 1-4-2002
Julie A. Trivedi, Quality Assurance Officer — Date

Glen A. Coder — 1-4-2002
Glen A. Coder, Laboratory Manager — Date

Annual Review

Date:					
Initials:					

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DEFINITIONS:

Laboratory Water – eighteen mega-ohm water.

Calibration Blank (CAL BLK) – a volume of laboratory water containing five-percent nitric acid and five-percent hydrochloric acid volume/volume used in calibrating the instrument.

Calibration Standard – a solution prepared from single and/or mixed stock standards in laboratory water and acid used to calibrate the instrument.

Initial Calibration Verification (ICV) – a solution prepared in laboratory water and acid from a second independent source used to verify the calibration.

Initial Calibration Blank (ICB) – a solution prepared in laboratory water and acid used to validate the calibration.

Continuing Calibration Verification (CCV) – a solution prepared in laboratory water and acid used to verify the calibration.

Continuing Calibration Blank (CCB) - a solution prepared in laboratory water and acid used to validate the calibration.

Reagent Water – acidified laboratory water used as a diluent in the preparation of sample dilutions and serial dilutions.

Internal Standard – a fixed concentration standard used to adjust for variances in matrix and operating conditions.

Instrument Detection Limit (IDL) – the concentration equivalent to a signal, due to the analyte, which is equal to three times the average of the standard deviation of a series of seven replicates done on three non-consecutive days.

Method Detection Limit (MDL) – the concentration equivalent to a signal, due to analyte, which is equal to three times the standard deviation of a series of seven replicates.

Instrument Profile – the process of optimizing an analytical peak.

Linear Range (LR) – the concentration range over which the instrument response is linear.

Method Blank – a laboratory water that is treated in the same manner as a group of samples.

Laboratory Control Sample (LCS) – laboratory water spiked at known concentration and treated in the same manner as a group of samples.

Laboratory Control Sample Duplicate (LCSD) - laboratory water spiked at known concentration and treated in the same manner as a group of samples.

Matrix Spike (MS) – a sample spiked at known concentration and treated in the same manner as a group of samples to determine accuracy.

Matrix Spike Duplicate (MSD) - a sample spiked at known concentration and treated in the same manner as a group of samples to determine accuracy and precision.

Stock Standard – a single or mixed element standard purchased from vendors.

Duplicate (DUP) – a second aliquot of sample treated in the same manner as a group of samples used to determine precision.

Analytical Post Spike (PS) – a post-digestion spike performed on a sample in a digestion group.

Serial Dilution – a test performed on a sample in a digestion group or analytical batch comparing the sample result with a 1/5 dilution of the same sample.

Sensitivity Check (CRI) – a low level standard used to evaluate the calibration curve.

Interelement Correction Factor (IEC) – a factor used to compensate for spectral interferences.

Background Correction Point - an offline point selected to correct background.

Interference Check Sample (ICS) – a group of solutions containing both interferents and analyte elements of known concentration used to evaluate background and interelement correction factors.

Initial Demonstration of Capacity (IDC) – testing performed for each digestion method by every analyst.

Reporting Limit – a limit below which analytes are reported as not detected. Normally referred to as Estimated Quantitation Limit (EQL), MDL, or Project Specific Reporting Limit (PSRL).

SAMPLE HANDLING AND PREPARATION:

Aqueous samples are collected in plastic 250-mL, 500-mL, or 1000-mL bottles and preserved by adding HNO_3 to obtain a pH < 2. Once preserved, the sample holding time may not exceed 6 months. Solid samples may be collected in plastic or glass containers and refrigerated at 4°C.

Samples are prepared for ICP analysis using EPA analytical digestion methods. For recoverable metals see Method 3005A. For total metals see Method 3010A or 3015. For solid or biota samples see Method 3050Bor 3051. Dissolved metals do not need digestion if they were field filtered and acidified at the time of collection.

APPARATUS and EQUIPMENT:

Thermo-Jarrell Ash 61E Trace, Inductively Coupled Argon Plasma Spectrophotometer
50-mL, 100-mL, 200-mL and 500-mL class A volumetric flasks
Pipettes, adjustable and fixed
Liquid Argon – high purity
Liquid Nitrogen

INTERFERENCES:

Spectral interferences are caused by overlap of a spectral line from another element, background noise contribution, stray light from the line emission of high concentration elements and overlap from molecular band spectra. An interelement interference table (see current table) has been

developed to help the analyst identify potential spectral interferences. Selecting background correction points adjacent to the analyte line can compensate for stray light and background noise contribution. Spectral overlaps can be compensated by equations that correct for interelement contributions.

Physical interferences are effects associated with sample introduction and nebulization. Changes in viscosity, especially in samples with high dissolved solids and high acid concentrations, can cause significant inaccuracies during analysis. Use of a peristaltic pump and an internal standard minimize these effects.

Chemical interferences include molecular compound formation, ionization effects, and solute vaporization effects. Normally, these effects are not significant with ICP. They can be reduced by matrix matching and standard additions if observed.

REAGENTS:

Hydrochloric Acid (HCL), trace metal grade.

Nitric Acid (HNO_3), trace metal grade.

Reagent Water – in a 500-mL volumetric flask add 200-mLs laboratory water, 25-mLs nitric acid, 25-mLs hydrochloric acid and bring to volume with laboratory water.

Yttrium Intermediate standard – in a 500-mL flask add 200-mLs laboratory water, 5-mLs nitric acid, 50-mLs 1000-ppm Yttrium stock standard and bring to volume with laboratory water.

Internal standard solution – in a 500-mL flask add 200-mLs laboratory water, 25-mLs nitric acid, 25-mLs hydrochloric acid, 25-mLs Yttrium Intermediate standard, 30-mLs 10,000-ppm Lithium standard and bring to volume with laboratory water.

STANDARDS:

GENERAL NOTES:

All standards less than 1 ppm are made daily.
Standards used for Calibration are made from stock standards purchased from High Purity.
Standards used to prepare ICV/CCV solutions are prepared using stock standards purchased for Inorganic Ventures.
All metals standards are prepared in class A volumetrics.
Standards are prepared using fixed and variable pipettors. Please refer to LAB-1 for the pipettor calibration procedure.
Standards are not used past the vendors' expiration date listed on the stock standard.
All prepared standards are recorded in the ICP Standard Logbook.

STOCK STANDARDS:

QCS-19 is purchased from High Purity. It contains 19 elements at 100-ppm each.

IV-19 is purchased from Inorganic Ventures. It contains 19 elements at 100-ppm each.

QCS-7 is purchased from High Purity. It contains 7 elements.

ICL500-6 is purchased from Environmental Express. It contains Al, Ca, and Mg at 5000-ppm, and Fe at 2000-ppm.

Single element stock standards are purchased in 1000-ppm and 10,000-ppm concentrations. All stock standards are logged into the Metals Stock Standard Logbook.

ANALYTICAL STANDARDS:

IV Calibration Standard – in a 200-mL flask add 100-mLs of laboratory water, 10-mLs nitric acid, 2.0-mLs of QCS-7, 0.98-mLs 10,000-ppm sodium, and 0.30-mLs 10,000-ppm potassium. Bring to volume with laboratory water.

HP Calibration Standard - in a 200-mL flask add 100-mLs of laboratory water, 10-mLs nitric acid, 10-mLs hydrochloric acid, and 2.0-mLs of QCS-19. Bring to volume with laboratory water.

STD3 Calibration Standard - in a 200-mL flask add 100-mLs of laboratory water, 10-mLs nitric acid, and 4.0-mLs each of 10,000-ppm single stock standards of calcium, aluminum, iron and magnesium. Bring to volume with laboratory water.

SNNA Calibration Standard - in a 200-mL flask add 100-mLs of laboratory water, 10-mLs nitric acid, 0.2-mLs of 10,000-ppm tin, and 0.5-mLs 10,000-ppm sodium. Bring to volume with laboratory water.

MG Calibration Standard - in a 200-mL flask add 100-mLs of laboratory water, 10-mLs nitric acid, and 4.0-mLs of 10,000-ppm magnesium. Bring to volume with laboratory water.

ICVSP/CCVSP Standard - in a 200-mL flask add 100-mLs of laboratory water, 10-mLs nitric acid, 1.0-mLs of IV-19, 0.2-mLs 10,000-ppm potassium, 1.98-mLs 10,000-ppm calcium and 1.98-mLs 10,000-ppm magnesium. Bring to volume with laboratory water.

ICVQC/CCVQC Standard - in a 200-mL flask add 100-mLs of laboratory water, 10-mLs nitric acid, 0.1-mLs each of tin, barium, boron and silver; 2.0-mLs each of 10,000-ppm aluminum and iron; and 0.5-mLs 10,000-ppm sodium. Bring to volume with laboratory water.

Sensitivity Check (CRI-EQL) – in a 50-mL flask add 20-mLs laboratory water, 2.5-mLs nitric acid, 0.5-mL 1-ppm antimony, 0.5-mLs ICP CRI EQL intermediate, and 0.25-mLs 1-ppm silver. Bring to volume with laboratory water.

ICSA - in a 200-mL flask add 100-mLs of laboratory water, 10-mLs nitric acid, 10-mLs hydrochloric acid, and 20-mLs ICL500-6. Bring to volume with laboratory water.
ICSAB – in a 100-mL flask add 100-mLs of laboratory water, 10-mLs nitric acid, 10-mLs hydrochloric acid 0.050-mLs each of 1000-ppm tin, nickel, cadmium and zinc; 10-mLs ICL500-6, 0.5-mLs IV-19 or QCS-19, and 0.5-mLs QCS-7. Bring to volume with laboratory water.

Solution C is used for analytical post spikes, and LCS, LCSD, MS and MSD spiked during sample preparation. The solution is verified before use. To test, dilute with reagent water and analyze as a sample.
In a 500-mL flask add 50-mLs of laboratory water, 5-mLs of nitric acid, and the following:

25-mLs 10,000-ppm sodium
23.75-mLs each of 10,000-ppm calcium and magnesium
12.5-mLs 10,000-ppm potassium
12.5-mLs each of 1000-ppm barium, boron and tin
1.25-mLs 10,000-ppm aluminum
1.25-mLs 1000-ppm silver
125-mLs 100-ppm QCS-19 or IV-19.

Bring to volume with laboratory water.

TCLP Spike Solution (TRTCP). This is used for spiking TCLP leachates. The solution is verified before use. To test, dilute with reagent water and analyze as a sample.
In a 200-mL flask add 30-mLs laboratory water, 2-mLs nitric acid 20-mLs each of 1000-ppm arsenic, lead, and chromium; 25-mLs 1000-ppm barium, 5.0-mLs each of 1000-ppm tin and boron; 0.5-mLs 1000-ppm silver, 0.5-mLs 10,000-ppm aluminum, and 50-mlS of IV-19 or QCS-19. Bring to volume with laboratory water.

INSTRUMENT OPERATION:

All ICP methods use a two-point calibration (a blank and a standard—see Appendix 2) for each analytical line. Calibration is verified prior to sample analysis by the use of Initial Calibration Verification (ICV) standard that is prepared from a source independent of the calibration standards. The instrument is calibrated after a thirty-minute warm-up and after profiling. Specific acceptance criteria for each QC standard are provided below. Details of instrument operation for profiling and calibration can be found in EN CHEM SOP 2-MET-42 "Inductively Coupled Plasma Emission Spectrometer", which is the Instrument Operating Procedure for the ICP.

SAMPLE ANALYSIS:

Samples are analyzed after the calibration is verified and the IEC corrections are checked. The calibration is verified by analyzing the ICV and ICB as noted in the QUALITY CONTROL section below. The ICSA and ICSAB solutions are analyzed to verify the IEC corrections factors.

NOTE: Any aqueous sample that is analyzed for selenium and is determined to have a detectable concentration present must be confirmed by GFAA. The only exception is TCLP extraction.

QUALITY CONTROL:

Initial Demonstration of Performance

Factory qualified service engineers install ICP's. The service engineer certifies that the instrument is functioning according to manufacturer's specifications prior to use by the laboratory. After certification by the service engineer, a qualified analyst prepares the analytical methods.

Aspirating a 1ppm Cd solution optimizes the nebulizer pressure. The signal to noise ratio is calculated at various pressures. The pressure that gives the best S/N ratio is entered into the analytical method for use.

Wavelength scans are performed on single element stock standards to determine where background correction points can be placed off line. TJA Trace ICP's have fixed background points for Se and Pb. After the background correction points are established, they are not changed.

Method Detection Limits (MDL)

MDLs are determined according to CFR 40 Pt.136, Appendix B. They are determined after initial installation, yearly thereafter, or whenever there are significant changes in analysts, operation or environmental conditions. Method Detection Limits are determined by preparing seven aliquots of laboratory water spiked at 3-5 times the estimated detection limit and processed through the entire digestion and analytical method. The instrument is calibrated and verified as in a normal analytical run.

Instrument Detection Limits (IDL)

IDLs are determined after initial installation, and quarterly thereafter. IDLs are determined by preparing seven aliquots of reagent water spiked at 3-5 times the estimated detection limit. The aliquots are analyzed on three non-consecutive days. The average of the three standard deviations is computed and multiplied by three to determine the IDL.

Linear Ranges

Linear ranges are determined after the instrument is calibrated and verified. Single element or mixed stock standards may be used. The upper range limit is an observed signal extrapolated from the calibration curve that is ± 10% of the true value. Linear ranges are verified at six-month intervals.

Interelement Correction Factors

Interelement Correction Factors are determined after setting background points and determining MDLs, EQLs, and Linear Ranges. Single element standards are analyzed after calibration and verification. The single element stock standards must be free of contamination or they will give false results. The single element standards are analyzed at a concentration at or below the Linear Range. Any signal greater that the absolute value of the EQL is subject to review as a possible interference. Interferences can be positive or negative depending on the interference. IEC's are adjusted as needed and reviewed every six months. See Appendix 3.

Initial Demonstration of Capability (IDC)

Each analyst for each digestion analyzes IDC solutions. The IDC solutions are carried through the entire digestion procedure. The results of the IDC's must be ± 20% of the true value and are kept on file for each analyst.

<u>Daily Quality Control</u>

Initial Calibration Verification (ICV)

The ICV is prepared from a source independent from the calibration standards and must be analyzed immediately after calibration. The ICV must meet the rejection criteria of ± 10% of the true value. Also, the relative standard deviation must be < 5 % from replicate (minimum of two) integrations. Recalibrate if the ICV fails. The concentration of the ICV should be near the mid-

point of the calibration curve. For EPA Method 200.7, the ICV must meet the rejection criteria of ± 5% of the true value.

Initial Calibration Blank (ICB)

The ICB must be analyzed after the ICV. The absolute value of the ICB must be ≤ 3 x IDL. If 3 times the IDL is less than 1/10 the EQL, 1/10 the EQL is used as the control limit. Also, if three times the IDL exceeds the EQL, the EQL is used as the control limit. In cases where 3 x the IDL criteria are consistently exceeded, the QC officer may determine a limit from historical data. Recalibrate if the ICB fails. However, if the ICB concentration is < 1/10 the concentration of the associated samples, the analysis need not be terminated. Current control limits for ICB are established quarterly and maintained on file in the laboratory.

Sensitivity Check (CRI)

The sensitivity check is normally run after the ICB. The concentration of the CRI will usually be at the EQL. Pass/fail criteria and CRI concentrations may vary due to client request.

Continuing Calibration Verification (CCV)

The CCV is analyzed after every 10 samples. Rejection criteria is ± 10 % of true value. The relative standard deviation must be < 5% from replicate (minimum of two) integrations. If the CCV fails, the problem must be corrected and the previous 10 samples between the CCV and last CCB must be reanalyzed. Concentration of the CCV should be near the mid-point of the calibration curve. For ICP analysis, if the Caves that bracket the samples to be reported for the analytes of interest are within the acceptable limit, those analytes may be reported.

Continuing Calibration Blank (CCB)

The CCB is analyzed after every CCV. The absolute value of the CCB should be ≤ 3 x IDL. If 3 times the IDL is less than 1/10 the EQL, 1/10 the EQL is used as the control limit. Also, if three times the IDL exceeds the EQL, the EQL is used as the control limit. In cases where 3 x the IDL criteria are consistently exceeded, the QC officer may determine a limit from historical data. Recalibrate if the CCB fails. However, if the CCB is < 1/10 the concentration of the associated samples, analysis need not be terminated. Current control limits for CCB are established quarterly and maintained on file in the laboratory.

Laboratory Control Sample (LCS/LCSD)

The LCS is carried through all preparation procedures and analyzed for each batch of 20 or fewer samples. See current QC Charts for control ranges. In cases where the LCS or LCSD is outside of acceptable ranges all samples prepared in that batch must be redigested and/or reanalyzed. In situations where the LCS or LCSD fails, and redigestion is not possible, the "&" flag is applied to the LCS, LCSD and ALL associated samples including method blanks, MS and MSD. LCS control limits are updated yearly based on historical data for each digestion method.

Method Blank (MB)

A MB is carried through the digestion procedure and analyzed with each batch of 20 or fewer samples. Rejection criteria for the MB are dependent upon the specific reporting limits for a project and any applicable regulatory limits. Appendix 4 to this SOP includes specific instructions for rejection of the MB or qualification of the associated data in accordance with various reporting limits.

Serial Dilution Test

A Serial Dilution Test is performed on each digestion group (batch). For dissolved samples that are not digested, one serial dilution is done per twenty samples. A 1/5 dilution is analyzed and compared to the original sample result. The serial dilution times 5 should agree within ± 10% of the original value. If the results do not agree, the sample is flagged with an "E". The sample is not qualified if the result is less than 50X the IDL. The serial dilution is used to determine if matrix interferences are present. It is left to the analyst to determine if additional corrective action is required.

Analytical Post Spike

An Analytical Post Spike is required at a frequency of 5 % or per sample digestion group. The control limits for a post-spike are 75-125%. If the post-spike recovery is out-of-control, dilute the corresponding sample and perform a post-spike on the diluted aliquot of sample, or use a more appropriate concentration if necessary. Dilute appropriately until an acceptable recovery is obtained. For EPA Method 200.7, the post-spike recovery must be within 85-115%.

ICSA / ICSAB

Analytes present in the ICSA and ICSAB solutions should be recovered within 80-120% of the true value. There is no criteria established for elements not present in the ICSA solution. However, if the result of an element not present in the solution is greater than ± EQL, investigate whether a problem exists. A concentration greater than ± EQL can indicate contamination, poor replicates, or an improperly adjusted IEC.

Trace elements such as Cd, Se, Pb, Sb, and Tl are the most likely to be out of control. The ICSA is composed of 500-ppm AL, Ca, Mg, and 200-ppm Fe. If the element has 2 or more correction factors both individual solutions should be analyzed to determine which is the source of the QC failure.

> Example: ICSA produces a Pb result of 5.7 µg/L. The EQL is 5.0 µg/L. Al and Fe are both interferents. Run a 500-ppm Al and a 200-ppm Fe solution to determine if one or both of the IECs needs to be adjusted. Adjust IEC as per IEC appendix. If individual solutions are in control, sample analysis may be started. Analyses of IEC test solutions are counted as samples when determining the number of samples between the ICV and CCV.

Linear Range (LR)

Any analyte or interfering element greater than the linear range must be diluted until it is less than the linear range. Any analytes that have interfering elements greater than the linear range require that the interfering element be within the linear range for the reported analyte to be valid.

For EPA Method 200.7, analytes that are within 10% of the linear range must be diluted.

Internal Standard

The internal standard counts for a given analysis must be within 30-120% of the internal standard counts of the Calibration Blank.

Accuracy

One matrix spike and matrix spike duplicate are digested and analyzed for each group of 20 or fewer samples that are similar in matrix at a frequency of 5%. For EPA Method 200.7, a matrix spike and matrix spike duplicate are performed at a frequency of 10% or one per sample batch, whichever is more frequent. Both QC samples must be calculated for accuracy. See current QC charts for control range.

$$\text{Spike Percent Recovery} = \frac{SSR - SR}{SA}$$

SSR = Spike Sample Result
SR = Sample Result
SA = Spike Added

If one or both spike recoveries are outside of the specified control limit, the corresponding parent sample and MS/MSD are to be flagged with an "N" qualifier. An analytical post spike is required if either the MS or MSD fail for accuracy.

If the analyte of interest is greater than 4 times the level of the spike concentration, accuracy calculations are not necessary and the "N" flag need not be applied.

If there is insufficient sample volume to perform a matrix spike and a matrix spike duplicate, an LCS and LCSD must be used in its place.

Precision

Matrix spike duplicate samples (MSD) or duplicates (DUP) are analyzed at a frequency of 5% or one per batch of samples that are similar in matrix. For EPA Method 200.7, a matrix spike duplicate of sample duplicate is performed at a frequency of 10% or one per sample batch, whichever is more frequent.

For precision, relative percent difference (RPD) is used to calculate compliance. See current QC charts for control limits.

Calculation:

$$RPD = \frac{MS - MSD}{(MS+MSD)/2} \times 100$$

MS = Method Spike Value
MSD = Method Spike Duplicate Value

If the RPD is outside of the acceptable control limits, the reported sample result is to be qualified with a "*" flag.

For duplicate samples that are less than 5 times the EQL, use +/- the EQL for acceptance limits.

SAMPLE RESULT CALCULATIONS:

Aqueous Sample Calculation:

Raw Data result (µg/L) X DF X MW DF(if applicable) = Final Result (µg/L)

where: MW DF = microwave dilution factor or 1.11 (correction for 45 mL initial volume to 50 mL final volume)
DF = dilution factor

Soil Sample Calculation:

$$\frac{\text{Raw Data result } (\mu g/L) \times FV\ (L) \times DF}{\text{Sample Weight } (g) \times \text{Dry Weight (decimal form)}} = \text{Final Result (mg/kg dry weight corrected)}$$

where: DF = Dilution Factor
FV = Final Volume

SAFETY:

The toxicity or carcinogenicity of each reagent used in this method has not been fully established. Each chemical should be regarded as a potential health hazard and exposure should be as low as reasonably achievable. Laboratory staff should observe all safety procedures as outlined in the Laboratory Health and Safety Manual. Staff should consult Materials Safety Data Sheets (MSDS) for information on specific chemicals.

POLLUTION PREVENTION and WASTE MANAGEMENT:

Pollution prevention encompasses any technique that reduces or eliminates the quantity or toxicity of waste at the point of generation. Laboratory staff should order and prepare only those quantities of reagents that will be used prior to the expiration date. Other appropriate measures to minimize waste generation should be brought to the attention of laboratory management. All laboratory waste shall be handled as directed by the Laboratory Waste Management Plan and the Hazardous Waste Contingency Plan.

APPENDIX 1

Method Analytes and Reporting Limits

		EQL			
Analyte	Wavelength	Water µg/L	Soil/Biota mg/Kg	TCLP µg/L	Linear Range
Silver	328.0	5.0	0.50	50	10,000
Aluminum	308.2	200	20	200	50,000
Arsenic	189.0	10	1.0	200	500,000
Boron	249.6	100	10	100	10,000
Barium	493.4	5.0	0.5	200	10,000
Beryllium	313.0	1.0	0.10	50	10,000
Calcium	317.9	100	50	1000	500,000
Cadmium	226.5	1.0	0.10	50	50,000
Chromium	267.7	3.0	0.30	50	50,000
Copper	324.7	10	2.0	50	50,000
Cobalt	228.6	3.0	0.50	50	50,000
Iron	259.9	50	10	100	50,000
Iron	271.4	100	10	100	50,000
Potassium	766.4	100	100/500	1000	50,000
Magnesium	279.0	30	30/50	1000	500,000
Manganese	257.6	2.0	0.20/0.50	50	30,000
Molybdenum	202.0	10	1.0	50	50,000
Sodium	330.2	500	200/1000	1000	200,000
Nickel	231.6	5.0	0.50	50	50,000
Lead	220.3	5.0	0.50	200	50,000
Antimony	206.8	10	1.0	50	50,000
Selenium	196.0	10	1.0	200	50,000
Thallium	190.8	10	1.0	50	50,000
Tin	189.9	20	5.0	50	50,000
Titanium	334.9	20	2.0	50	50,000
Vanadium	292.4	5.0	0.50	50	50,000
Zinc	206.2	20	2.0/5.0	200	50,000

** Control limits and Linear Ranges are updated periodically. Those that are in use at the time of analysis will be used and made available to data validators.

APPENDIX 2

ICP Standard Components and Concentrations, µg/L

Element	ICVSP/ CCVSP	ICVQC/ CCVQC	ICSA	ICSB (AB)	Cal Std	Cal Std Name	Linear Range (+/- 10)	Spike **
Ag		500		500	1000	IV	10000	100
As	500			500	1000	HP	50000	1000
Al		100000	500000	500000	200000	STD3	500000	1000
B		500		500	1000	IV	30000	1000
Ba		500		500	1000	IV	10000	1000
Be	500			500	1000	HP	10000	1000
Ca	100000		500000	500000	200000	STD3	500000	20000
Cd	500			1000	1000	HP	50000	1000
Co	500			500	1000	HP	50000	1000
Cr	500			500	1000	HP	50000	1000
Cu	500			500	1000	HP	50000	1000
Fe259	500		200000	200000	1000	HP	50000	1000
Fe271		100000	200000	200000	200000	STD3	500000	1000
K404		100000			200000	STD3	500000	10000
K766	10000				25000	IV	50000	10000
Mg	100000		500000	500000	200000	STD3	500000	20000
Mn	500			500	1000	HP	20000	1000
Mo	500			500	1000	HP	50000	1000
Na330		25000			50000	IV	200000	20000
Na588	10000				25000	SnNa	50000	20000
Ni	500			1000	1000	HP	50000	1000
Sb	500			500	1000	HP	50000	1000
Sn		500		500	1000	SnNa	50000	1000
Pb	500			500	1000	HP	50000	1000
Se	500			500	1000	HP	50000	1000
Tl	500			500	1000	HP	50000	1000
Ti	500			500	1000	HP	50000	1000
V	500			500	1000	HP	50000	1000
Zn	500			1000	1000	HP	50000	1000

** Spike level for LCS, MS, MSD. Also post spike level when solution C is used.

APPENDIX 3

Interelement Correction Factor Set-up for TJA ICAP61E Trace

Purpose:

The interelement Correction Factors (IEC's) are used to correct for false signals (positive and negative) produced when peaks overlap in the spectra.

Initial Setting of the IEC's:

B. Single element standards are run with background correction points in place. The standards are run at or below the Linear Range. Any signal greater than the absolute value of the EQL should be evaluated as a possible interference. Wavelength scans are useful in determining if contamination is present. A copy of the MIT Wavelength Tables is available for analyst reference.

C. Use Part A for all elements other than Se and Pb.

A. Elements with results above the detection limits are noted. The following calculation is performed:

$$\frac{\text{Element Result } (\mu g/L)}{\text{Interfering Element Result } (\mu g/L} = \text{IEC}$$

This ratio is entered in the IEC table in the method file.

B. Pb and Se have two analytical lines, which are combined to produce the analytical result. Each Pb and Se line must be dealt with separately. Ratios will be entered for both the 220/1 and 220/2 in the case of Pb.
Example: A 500-ppm Al standard produces a –171 µg/L on the Pb 220/1 line and a 58 µg/L result on the Pb 220/2 line. The Al reads 497,100 µg/L. Using the above calculation we get –0.000344 for the Pb 220/1 and 0.000117 for the Pb 220/2.

Entering the IEC's:

B. At the main menu select **Development** and press enter. Select **Methods** and press enter. At the Enter Method Name prompt; type the method name or select it from the list. Depress the enter key. Press **F5 (Element Info)** and select **F3 (IEC's)**. Move the cursor to the element you wish to enter an IEC for and press the *page down* key to access the wavelength of choice. Pressing the arrows keys will highlight the second wavelength if one is listed. Press the *page down* key to enter the IEC.

C. If the interfering element is not selected (listed) press **F6** (*edit*) and the **F6** (select IEC's) key. Choose the interfering element by entering the number corresponding to the element from the list. Press the enter key. Press **F9** (*done/keep*).

D. Then type the ratio under the k1 header in the appropriate row. Enter a negative before the ratio if it is a negative interference. Press **F9** (*done/keep*) as many times as necessary to return to the main menu and save the IEC's.

APPENDIX 4

Method Blank Qualification
1 of 3

Flowchart A
LOD/LOQ BLANK FLAGGING and REPORTING

BLANK POSITION> / Sample Position⇓	5	4	3	1	2	6
1	R	A(-x) Flag Report < LOD	Report < LOD	Report < LOD	Report < LOD	Report < LOD
2	R	A(-x) Flag Report result w/20X Rule	Report result	Report result	A(x) Flag Report result w/20X Rule	R
3	R	A(-x) Flag Report <LOD	Report <LOD	Report <LOD	Report <LOD	Report <LOD
4	R	A(-x) Flag Report <LOD	Report <LOD	Report <LOD	A(x) Flag Report result w/20X Rule	Report <LOD
5	R	R	R	R	R	R
6	R	A(-x) Flag Report result w/20X Rule	Report result	Report result	A(x) Flag Report result w/20X Rule	Report result w/20X Rule -or- R if result is not 20XMB

R= Analyst discretion to Re-run,Re-calculate, or Re-digest
X= Value in Method Blank (MB).

APPENDIX 4

Method Blank Qualification
2 of 3

Flowchart B
EQL or PRSL BLANK FLAGGING and REPORTING

BLANK POSITION> / Sample Position ⇓	5	4	3	1	2	6
1	R	Report < RL	Report < RL	Report < LOD	Report < RL	Report < RL
2	R	Report < RL	Report < RL	Report result	Report < RL	Report < RL
3	R	Report < RL	Report < RL	Report <LOD	Report < RL	Report < RL
4	R	Report < RL	Report < RL	Report < RL	Report < RL	Report < RL
5	R	R	R	R	R	R
6	R	Report result	Report result	Report result	Report result	Report result w/20X Rule -or- R if result is not 20XMB

R= Analyst discretion to Re-run, Re-calculate or Re-digest
X= Value in Method Blank (MB)
RL= Reporting Limit

Appendix 4

Method Blank Qualification
3 of 3

Flowchart C
EQL or PRSL BLANK ''B'' FLAGGING and REPORTING

BLANK POSITION> / Sample Position⇓	5	4	3	1	2	6
1	R	Report < RL	Report < RL	Report < RL	Report < RL	Report < RL
2	R	Report Result B - Flag A(-x) Flag W/ 20X Rule	Report Result B - Flag	Report Result B - Flag	Report Result A(x) Flag W/ 20X Rule	R
3	R	Report < RL	Report < RL	Report < RL	Report < RL	Report < RL
4	R	Report < RL	Report < RL	Report < RL	Report < RL	Report < RL
5	R	R	R	R	R	R
6	R	Report result A(-x) Flag W/ 20X Rule	Report result	Report result	Report Result A(x) Flag W/ 20X Rule	Report result A(x) Flag w/20X Rule -or- R if result is not 20XMB

R= Analyst discretion to Re-run,Re-calculate, or Re-digest
X= Value in Method Blank (MB).
RL= Reporting Limit

En Chem, Inc.

Quality Assurance Document

SET No: 1

ANALYTICAL METHOD

TITLE: Acid Digestion of Solid Samples

DEPARTMENT: Inorganic - Metals

APPLICATION: Samples may be analyzed by ICP, ICPMS, or GFAA.
ICP or ICPMS for the following metals (GFAA metals = *):

Al	Ca	Fe	Na	Sr
As *	Cd *	K	Ni	Ti
B	Co	Mg	Pb *	Tl *
Ba	Cr *	Mn	Se *	V
Be	Cu *	Mo	Sn	Zn

For sample digestion using SW846 3050B for Ag, Sb, or Hg; see SOP MET-7
Applicable Matrices: Soils, Sediments, Sludges and Solid Wastes

REFERENCE: EPA Manual SW-846, 3rd Edition: Method 3050B

PROCEDURE SUMMARY:

This method prepares solid samples for analysis by ICP, ICPMS, or GFAA. Digestion can reduce interferences by organic matter and convert metals to a form that can be determined by instrumentation. A representative (wet weight) portion of sample is weighed into a Hot Block digestion tube and digested in nitric acid and hydrogen peroxide. The digestate is then refluxed with hydrochloric acid for ICP and ICPMS. After cooling, the sample is filtered and taken to a volume of 100 mL.

REVIEWED BY: Jeffrey A. Gordon — Date: 10-20-00
Inorganic Section Supervisor

Gregory J. Graf — Date: 10-20-00
Quality Assurance Officer

APPROVED BY: Glen A. Coder — Date: 10-20-00
Laboratory Manager

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SAMPLE HANDLING AND PRESERVATION:

Solid and semi-solid samples may be collected in clean plastic or glass containers. Samples samples are refrigerated at 4°C to preserve integrity.

INTERFERENCES:

Sludge samples can contain diverse matrix types, each of which may present analytical challenges. Spiked samples and Laboratory Control Samples are important for determining digestion efficiency.

APPARATUS AND MATERIALS:

Top-loading balance
Spatulas
Hot block
Acid repipettor
Pipette: adjustable and fixed volume
Hot block digestion tubes, plastic
Hot block digestion tubes, glass
Watch glasses
Whatman #541 filter paper
D.I. water bottle
Specimen containers: 120 mL

NOTE: All glassware is nitric acid (HNO_3) and DI water rinsed before use.

REAGENTS:

Deionized (D.I.) water
1:1 redistilled nitric acid (HNO_3)
Nitric acid (HNO_3): concentrated, redistilled
30% hydrogen peroxide (H_2O_2)
Concentrated (37%) hydrochloric acid (HCl)

PROCEDURE:

<u>Quality Control</u>

Method Blank - one per digestion group, or per 20 samples, whichever is more frequent.
Laboratory Control Sample (LCS) - one per digestion group, or per 20 samples, whichever is more frequent.
Matrix Spike - one per digestion group, or per 20 samples, whichever is more frequent.
Matrix Spike Duplicate - one per digestion group, or per 20 samples, whichever is more frequent.

Sample Preparation

1. WEAR SAFETY GLASSES AND GLOVES.

2. Heat hot block to 90-95°C. Typically, a setting of 110 degrees will achieve this range. Check the temperature setting with a secondary thermometer daily and record in daily log .

3. Verify sample Station IDs and LIMS numbers on containers against LIMS backlog lists.

4. Lay out run in digestion log book. Identify QC samples at frequency stated above.

5. Collect samples near top-loading balance.

6. To the extent possible, mix and crush the sample in the sample container with a spatula to achieve homogeneity.

7. Weigh 1.00 gram of homogenized sample into a labeled digestion tube. Record weight to nearest 0.01 gram on digestion log. Do this for each sample in the digestion group. For samples with high liquid content, a larger sample size may be used, as long as the digestion is complete.

8. Prepare method blank by labeling empty digestion tube.

9. Prepare Laboratory Control Sample (LCS) by labeling an empty digestion tube, adding the appropriate LCS spiking solution (see Step 11, below) to the digestion tube.

   To Prepare Paint Chips for Digestion:

   A. Transfer paint chips into labeled drying tins. Dry samples at 103-105° C for 12-24 hours.
   B. Remove samples from oven and desiccate for 1 hour.
   C. Mix and crush samples with mortar and pestle to achieve homogeneity.
   D. Into labeled digestion tubes, weigh out 1.0 g of sample. (If less than 1.0 g use whatever sample is available.)

   NOTE: Due to the pre-drying of samples, results are always reported as mg/kg dry wt.

   CONTINUE DIGESTION IN HOOD.

10. Add 10 mL 1:1 redistilled HNO_3 using a calibrated repipettor.

11. Spike designated samples using a calibrated pipettor. (See SOP MET-56)

12. Cover with a watch glass and swirl gently to mix.

13. Heat in 90-95°C hot block, refluxing for 15 minutes.

    CAUTION: DO NOT BOIL AT ANY TIME DURING DIGESTION.
    This may result in metals volatilizing, which would require starting the digestion over.

14. Remove from hot block and cool in hood.

15. Add 5 mL concentrated redistilled HNO_3 using a calibrated repipettor.

16. Cover again with watch glass and swirl gently to mix.

17. Heat in 90-95°C hot block, refluxing for 30 minutes.

18. If brown fumes are generated, remove from hot block and cool. Repeat steps #'s 15-17 over and over again, until no brown fumes are given off. Once no brown fumes are generated continue with step 19.

19. Continue heating the samples at 95 °C (± 5 °C) for 2 hours. Do not allow the samples to boil. Maintain a covering of solution over the bottom of the tube at all times. Remove from hot block and cool in hood.

20. Add 2 mL of DI water and 3 mL 30% H_2O_2 using a calibrated hydrogen peroxide repipettor.

21. Cover with watch glass, swirl gently to mix, and heat in hot block to start peroxide reaction.

23. Heat until effervescence subsides, then cool in hood.

23. Continue to add 30% H_2O_2 in 1 mL aliquots with warming until effervescence is minimal or sample appearance is unchanged. (Do not add more than a total of 10 mL 30% H_2O_2.)

    CAUTION: DO NOT ALLOW LOSS OF SAMPLE DUE TO EXCESSIVE EFFERVESCENCE. If sample loss occurs, the sample digestion for that sample must be repeated.

24. Heat the samples to 95 °C (± 5 °) and maintain for 2 hours. Do not allow the samples to boil. Maintain a covering of solution over the bottom of the tube at all times. Remove from hot block and cool in hood.

<u>IF SAMPLE DIGESTION IS FOR GFAA OR ICPMS ANALYSIS, SKIP TO STEP # 29</u>

25. Add 10 mL concentrated (37%) HCl using a calibrated repipettor

26. Cover with watch glass and swirl gently to mix.

27. Heat in hot block, refluxing for 15 minutes.

28. Remove from hot block and cool in hood. Transfer to counter.

29. Rinse watch glass with D.I. water into digestion tube.

30. Filter through Whatman #541 filter paper or equivalent into a labeled 120 mL specimen cup.

31. Rinse digestion tube with D.I. water into filter paper.

32. Rinse filter paper with D.I. water into specimen cup.

33. Bring to final volume of 100 mL with D.I. water.

34. Mix sample well.

35. Ready for analysis.

SAFETY:

The toxicity or carcinogenicity of each reagent used in this method has not been fully established. Each chemical should be regarded as a potential health hazard and exposure should be as low as reasonably achievable. Laboratory staff should observe all safety procedures as outlined in the Laboratory Health and Safety Manual. Staff should consult Materials Safety Data Sheets (MSDS) for information on specific chemicals.

POLLUTION PREVENTION and WASTE MANAGEMENT:

Pollution prevention encompasses any technique that reduces or eliminates the quantity or toxicity of waste at the point of generation. Laboratory staff should order and prepare only those quantities of reagents that will be used prior to the expiration date. Other appropriate measures to minimize waste generation should be brought to the attention of laboratory management. All laboratory waste shall be handled as directed by the Laboratory Waste Management Plan and Hazardous Waste Contingency Plan.

ANALYTICAL METHOD

TITLE: Acid Digestion of Aqueous Samples for Total Metals - ICP

DEPARTMENT: Inorganic - Metals

APPLICATION: Samples may be analyzed by ICP for the following metals:

Al	Cd	Mg	Sn
As	Co	Mn	Se
Ba	Cr	Mo	Ti
Be	Cu	Na	Tl
B	Fe	Ni	V
Ca	K	Pb	Zn

Applicable to all aqueous matrices **except** Dissolved or Recoverable metals:
Reminder : This digestion is Not to be used for Ag, Sb, or Hg due to their volatility.

REFERENCE: EPA Manual SW-846, 3rd Edition, Method 3010A

PROCEDURE SUMMARY:

This method prepares samples for total metals analysis. Digestion can reduce interferences by organic matter and convert metals to a form that can be determined by instrumentation. The sample is heated with nitric acid and taken down to a low volume, then refluxed with nitric acid until the digestate is light or stable in color. Then refluxed with hydrochloric acid, cooled, and brought back to volume.

REVIEWED BY: Jeffrey A. Gordon — Date: 2.29.00
Inorganic Section Supervisor

Gregory J. Graf — Date: 2-28-2000
Quality Assurance Officer

APPROVED BY: Glen A. Coder — Date: 2-29-2000
Laboratory Manager

SAMPLE HANDLING AND PRESERVATION:

All samples are collected in plastic or glass containers. Samples are received by the laboratory acidified to a pH of < 2 with HNO_3 and stored at an ambient temperature.

INTERFERENCES:

The digestion is not suitable for samples to be analyzed by graphite furnace atomic absorption because hydrochloric acid can cause interferences during atomization.

APPARATUS AND MATERIALS:

Hot block
Acid repipetters
Pipette: adjustable and fixed volume
Hot block digestion tubes
Graduated cylinders: 50 mL
Hot block watchglasses
Whatman #541 filter paper
D.I. water bottle
Specimen cups: 120 mL

NOTE: All glassware is nitric acid (HNO_3) and DI water rinsed before use.

REAGENTS:

Deionized (D.I.) water
Nitric acid (HNO_3): concentrated, redistilled
Hydrochloric acid (HCl): 1:1

PROCEDURE:

Quality Control

Method Blank - one per digestion group, or per 20 samples, whichever is more frequent.

Laboratory Control Sample (LCS) - one per digestion group, or per 20 samples, whichever is more frequent.

Matrix Spike - one per digestion group, or per 20 samples, whichever is more frequent.
Matrix Spike Duplicate - one per digestion group, or per 20 samples, whichever is more frequent.

NOTE: For TCLP samples one MS is performed for each waste-stream. TCLP samples are spiked prior to preservation.

Sample Preparation

1. WEAR SAFETY GLASSES AND GLOVES.

2. Heat hot block to 90-95°C. Typically, setting the hot block at about 110 degrees will achieve the required temperature range. When stable, record temperature in daily log once per day.

3. Verify sample Station IDs and LIMs numbers on containers against LIMs worklists.

4. Lay out run in digestion log book. Identify QC samples at frequency stated above.

5. Prepare Laboratory Control Sample (LCS) by adding the appropriate LCS spiking solution to a 50 mL aliquot of D.I. water in a labeled digestion tube. See SOP MET-56

6. Prepare method blank by transferring a 50 mL aliquot of D.I. water into a labeled digestion tube using a graduated cylinder.

7. Transfer a 50 mL aliquot of well-mixed sample into a labeled digestion tube using a graduated cylinder. Do this for each sample in digestion group.

8. Spike designated samples using a calibrated pipettor. See SOP MET-56.

   CONTINUE DIGESTION IN HOOD.

9. Add 1.5 mL concentrated redistilled HNO_3 using a calibrated repipettor.

10. Swirl gently to mix.

11. Heat in 90-95°C hot block, slowly evaporating to 20-25 mL.

    CAUTION: DO NOT BOIL. DO NOT ALLOW TO GO DRY. IF SAMPLE GOES TO DRYNESS, DISCARD AND REPREPARE.

12. Remove from hot block and cool in hood.

13. Add 1.5 mL concentrated redistilled HNO_3 using a calibrated repipettor.

14. Cover with a watchglass and swirl gently to mix.

15. Continue heating, until digestion is light in color or does not change in appearance. (15 minutes)

    CAUTION: DO NOT ALLOW TO GO DRY.

16. Remove from hot plate and cool in hood.

17. Add 5 mL 1:1 HCl using a calibrated repipettor.

18. Cover again with watchglass and swirl gently to mix.

19. Heat 15 minutes to dissolve any residue.

20. Remove from hot block and cool in hood. Transfer to counter.

21. Rinse watchglass with D.I. water into digestion tube.

22. Filter samples with insoluble particles through Whatman #541 filter paper or equivalent into a labeled 120 mL specimen cup. OR use hot block digestion in-tube filters.

23. Bring to final volume of 50 mL.

24. Ready for analysis.

SAFETY:

The toxicity or carcinogenicity of each reagent used in this method has not been fully established. Each chemical should be regarded as a potential health hazard and exposure should be as low as reasonably achievable. Laboratory staff should observe all safety procedures as outlined in the Laboratory Health and Safety Manual. Staff should consult Materials Safety Data Sheets (MSDS) for information on specific chemicals.

POLLUTION PREVENTION and WASTE MANAGEMENT:

Pollution prevention encompasses any technique that reduces or eliminates the quantity or toxicity of waste at the point of generation. Laboratory staff should order and prepare only those quantities of reagents that will be used prior to the expiration date. Other appropriate measures to minimize waste generation should be brought to the attention of laboratory management. All laboratory waste shall be handled as directed by the Laboratory Waste Management Plan and Hazardous Waste Contingency Plan.

ANALYTICAL METHOD

TITLE: Mercury Analysis of Compositional Samples by CVAA

DEPARTMENT: Inorganic - Metals

APPLICATION: Soils, sediments, sludges, and biological tissues

REFERENCE: USEPA Test Methods for Evaluating Solid Waste SW-846, 3rd Edition, Method 7471A, Revision 1, September 1994

PROCEDURE SUMMARY:

Cold vapor atomic absorption utilizes the volatile property of elemental mercury at the 253.7 nm wavelength. To release mercury from organic complexes, the sample is digested with oxidizing reagents and acids in a hot water bath. After digestion, the oxidizing reagents are neutralized. Stannous chloride is added to reduce ionic mercury to the ground state. The Flow Injection Analysis System sweeps the volatile elemental mercury out of the sample and into the cell of an atomic absorption spectrophotometer. The absorbance signal is proportional to the amount of mercury in the sample.

APPROVED BY: Michael J. Heimann — 12/27/01
Michael J. Heimann
Wet Chemistry Supervisor
Date

Julie Trivedi — 12/27/01
Julie A. Trivedi
Quality Assurance Officer
Date

Glen A. Coder — 12/31/01
Glen A. Coder
Laboratory Manager
Date

Annual Review

Date:					
Initials:					

SAMPLE HANDLING AND PRESERVATION:

Samples can be collected in either glass or plastic.

INTERFERENCES:

Potassium permanganate is added to eliminate possible interference from sulfide. *Concentrations as high as 20 mg/L of sulfide as NaS do not interfere with the recovery of added* inorganic mercury from D.I. water.

Copper has also been reported to interfere. However, copper concentrations as high as 10 mg/L had no effect on recovery of mercury from spiked samples.

Seawaters, brines and industrial effluents high in chlorides require additional permanganate, as much as 25 mL because, during the oxidation step, chlorides are converted to free chlorine, which also absorbs radiation at 253.7 nm. Care must therefore be taken to ensure that free chlorine is absent before the mercury is reduced and swept into the cell. This may be accomplished by using an excess of hydroxylamine hydrochloride reagent, 6 mL.

Certain volatile organic materials that absorb at this wavelength may also interfere. A preliminary run without reagents would determine if this type of interference is present.

APPARATUS AND MATERIALS:

Balance
BOD bottles: 300 mL
Graduated cylinders: 50 mL, 100 mL
Glass beaker: 1000 mL
Volumetric flasks: 2-100 mL, 1-50 mL
Volumetric pipets: 0.1 mL - 5 mL
Eppendorf pipettors and tips
Acid and reagent repipettors
Water bath(s)
Stir bar
Stir plate
Spatulas
Plastic containers with screw caps
Plastic bottle racks

REAGENTS:

Deionized (D.I.) water
Certified standard solution: 1000 mg/L (1000 ppm)
Second Source Certified standard solution: 1000 mg/L (1000 ppm)
Sulfuric acid (H_2SO_4): concentrated, reagent grade
Hydrochloric acid (HCl); concentrated, reagent grade
Nitric acid (HNO_3): concentrated, distilled or mercury-free
Potassium permanganate, low mercury, 5% solution (w/v)

Sodium chloride
Hydroxylamine hydrochloride
Stannous chloride

Prepare 0.5 N Sulfuric Acid :

Slowly add 14.0 mL of concentrated H_2SO_4 to 500 mL of reagent water and dilute to 1 L with reagent water.

Prepare Potassium permanganate, 5% solution (w/v):

- Dissolve 100 grams potassium permanganate ($KMnO_4$) in 2 liters D.I. water.

Prepare Aqua-Regia Solution:

- Prepare immediately before use by carefully adding three volumes of concentrated HCL to one volume of concentrated HNO3. Prepare the solution in a glass vessel.

Prepare Sodium chloride - hydroxylamine hydrochloride solution:

- Dissolve 120 grams sodium chloride (NaCl) and 120 grams hydroxylamine hydrochloride ($NH_2OH \cdot HCL$) in 1 liter D.I. water.

NOTE: The solutions above must be prepared fresh monthly.

Prepare Stannous chloride Solution:

- Add 25 grams stannous chloride ($SnCL_2 \cdot 2\ H_20$) to 250 mL 0.5 N Sulfuric Acid.

NOTE: The stannous chloride must be stirred continuously during use and should be made fresh prior to use.

Prepare Intermediate Calibration Standard, 1 ppm (prepare daily):

- Acid rinse a 100 mL volumetric flask, first with 1:1 HNO_3, then with D.I. water.
- Add 50 - 70 mL D.I. water.
- Add 0.5 mL HNO_3. Wash down with D.I. water.
- Pipet 0.1 mL certified standard solution (1000 ppm) into the flask using a calibrated Eppendorf pipettor.
- Bring to volume with D.I. water, invert 6 times, and label as 1 ppm Hg intermediate standard.

Prepare Working Calibration Standard, 0.1 ppm (prepare daily):

- Acid rinse a 50 mL volumetric flask.
- Add 0.3 mL HNO_3 to flask. Rinse down with D.I. water and swirl.

- Pipet 5 mL 1 ppm Hg intermediate standard into the flask using a glass or calibrated Eppendorf pipettor.

- Bring to volume with D.I. water, invert 6 times, and label as 0.1 ppm Hg working standard.

NOTE: The following standards must be prepared with each sample batch.

Prepare Calibration Standards:

- Label a BOD bottle as a blank and add 10 mL of D.I. water.

- Label one bottle as a zero standard and one bottle each as 0.2, 1.0, 5.0, 10.0 ppb standards. Using a calibrated pipettor, transfer 0.2, 1.0, 5.0 and 10.0 mL, respectively, of 0.1 ppm working standard into each bottle.

- Fill with D.I. water so that the total volume in each bottle equals 10 mL. Use a graduated cylinder.

Prepare Laboratory Control Sample (LCS):

Transfer 100 ml of DI water into a BOD bottle using a graduated cylinder. Pipette 0.5 ml of Hg intermediate standard into the bottle using a calibrated auto-pipettor.

NOTE: For CLP work or for BIOTA samples, a SRM may be requested by the client for an LCS. For BIOTA analysis, weigh out 1.0 g of Tuna in two separate bottles. One will be spiked for the LCS and the second will be analyzed for background level use in calculating the LCS.

Prepare ICB, CCB and MB (Initial Calibration Blank, Continuing Calibration Blank and Method Blank):

- Transfer 10 mL of D.I. water into BOD bottles using a pipette. Label as ICB, CCB or MB.

Prepare ICV and CCV (Initial Calibration Verification and Continuing Calibration Verification):

- Acid rinse a 100 mL volumetric flask, first with 1:1 HNO_3, then with D.I. water.

- Add 50 - 70 mL D.I. water.

- Add 0.5 mL HNO_3. Wash down with D.I. water and swirl.

- Pipet 0.1 mL standard solution (1000 ppm) into the flask using a calibrated Eppendorf pipettor.

- Bring to volume with D.I. water, invert 6 times, and label as 1 ppm Hg intermediate standard.

- Pipet 0.4 mL 1 ppm Hg intermediate into a BOD bottle using a calibrated Eppendorf pipettor. Label as ICV - 4 ppb. Pipette 0.5 mL 1 ppm Hg intermediate. Label as CCV.

- Make final volume 10 mL.

PROCEDURE:

1. Heat water bath(s) to 95°C. Water level should reach above liquid in BOD bottles. Note water bath temperature in logbook.

2. Prepare reagents as necessary.

3. Write up run on digestion bench sheet and CVAA compositional bench sheet. Identify the samples for Quality Control at 5%.

4. Collect and label acid-washed BOD bottles and acid-washed glassware.

5. Collect samples. Mix thoroughly to achieve homogeneity.

6. Mix and crush a generous aliquot in disposable screw-top container with spatula.

7. Weigh out a 1.0 g homogeneous portion into BOD bottle. Record weight to nearest 0.1 g on benchsheet. If making a dilution, do not use a smaller weight of sample. Prepare and digest a blank to provide diluent.

CAUTION: CONTINUE DIGESTION IN HOOD. WEAR SAFETY GLASSES AND GLOVES.

NOTE: All reagents should be added in equal amounts to standards and samples.

8. Add 5 mL of DI water.

9. Add 5 mL of Aqua-Regia Solution and mix.

10. Spike designated samples and the LCS by pipetting 0.5 mL 1 ppm certified Hg intermediate standard into BOD bottles using a calibrated Eppendorf pipettor.

11. Heat 2 minutes in 95°C water bath. (Use plastic bottle racks). Remove from bath and cool to room temperature.

12. Add 50 mL D.I. water to each of the samples and the LCS. To the MB, ICB, CCB, ICV and CCV add 40 mL D.I. water.

13. Add 15 mL potassium permanganate solution using a calibrated repipettor. Mix, adding additional potassium permanganate crystals if necessary until purple color persists.

14. Heat BOD bottles in 95°C water bath for 30 minutes. (Check to ensure purple color is maintained. Add potassium permanganate crystals if needed. The same amount of crystals must be added to all samples, standards, and QC samples).

15. Cool bottles to room temperature before analysis.

16. Set up AA instrument for cold vapor analysis. (See EN CHEM SOP MET-57.)

17. Add 6 mL sodium chloride-hydroxylamine hydrochloride solution to each bottle using an repipettor. Swirl until no color persists.

1. Add 50 mL D.I. water to each bottle using a graduated cylinder.

CALIBRATION:

The instrument must be calibrated every time it is set up. The absorbance obtained for S0 (the calibration blank), is subtracted from all absorbances in the run. Least-squares linear regression (y-axis = concentration, x-axis = absorbance) is used to correlate blank-corrected absorbance with concentration.

The correlation coefficient, r, must be 0.995 or greater. The absolute value of the y-intercept must be less than the PQL.

QUALITY CONTROL:

1. Initial Demonstration of Performance

The initial demonstration of performance is used to characterize instrument performance (determination of LCRs and analysis of QCS) and laboratory performance (determination of MDLs) prior to performing analyses by this method.

a. Linear Calibration Range (LCR) – The LCR must be determined initially and verified annually or whenever a significant change in instrument response is observed or expected. The initial demonstration of linearity must use sufficient standards to insure that the resulting curve is linear. The verification of linearity must use a minimum of a blank and three standards. If any verification data exceeds the initial values by ± 10%, linearity must be reestablished.

b. Method Detection Limit (MDL) – MDLs must be established for all analytes, using reagent water fortified at a concentration of one to five times the estimated instrument detection limit. MDLs should be determined annually, when a new operator begins work or whenever there is a significant change in the background or instrument response.

2. Assessing Laboratory Performance

a. Correlation Coefficient (r-value)
The correlation coefficient, the measure of linearity of a standard curve, must be 0.995 or greater. If the value is less than 0.995 recalibrate the instrument.

b. Initial Calibration Verification (ICV)
The ICV is prepared from a source independent from the calibration standards and must be analyzed immediately after calibration. The ICV must meet the rejection criteria of ± 10% of the true value. Recalibrate if the ICV fails. The concentration of the ICV should be near the mid-point of the calibration curve.

c. Initial Calibration Blank (ICB)
The ICB must be analyzed after the ICV. The absolute value must be ≤ EQL. Recalibrate if it fails.

d. Continuing Calibration Verification (CCV)
The CCV is analyzed after every 10 samples. The acceptance criteria is ±10% of true value. If the CCV fails, the problem must be corrected and the previous 10 samples between the CCV and last CCB must be reanalyzed. Concentration of the CCV should be near the mid-point of the calibration curve.

e. Continuing Calibration Blank (CCB)
The CCB is analyzed after every CCV. The absolute value must be ≤ EQL. If the CCB fails, the problem must be corrected and the previous 10 samples between the last CCB and the CCV must be reanalyzed.

f. Laboratory Control Sample (LCS)
The LCS is carried through all preparation procedures and analyzed for each matrix type with a frequency of 5%. See current QC Charts for control ranges. In cases where the LCS is outside of acceptable ranges all samples prepared in that batch must be reprepared and reanalyzed.

g. Method Blank (MB)
A MB is carried through all prep procedures and analyzed with a frequency of 5%. Rejection criteria is <LOD. Other criteria may apply, such as regulatory limit and the analyte concentration in the samples.

h. Matrix Spike (MS) and Matrix Spike Duplicate (MSD)
One matrix spike and matrix spike duplicate are analyzed for each group of samples that are similar in matrix at a frequency of 5%. Both QC samples must be calculated for accuracy. See current QC charts for control range.

$$\text{Spike Percent Recovery} = \frac{SSR - SR}{SA}$$

SSR = Spike Sample Result
SR = Sample Result
SA = Spike Added

If the both spike recoveries are outside of the specified control limit, the corresponding parent sample is to be post-spiked and the reported result shall be flagged with the [N qualifier.

The control limits for a post-spike are 75-125%. If the post-spike recovery is out-of-control, dilute the corresponding sample and perform a post-spike on the diluted aliquot of sample. Dilute appropriately until an acceptable recovery is obtained. If only the matrix spike OR the matrix spike duplicate are out of control for accuracy, then the corresponding parent sample is flagged with the [MS qualifier.

If the analyte of interest is greater than the linear range, dilute appropriately and post-spike the sample; however, the [N qualifier is not required. Also, if the analyte of interest is greater than 4x the level of the spike concentration, accuracy calculations are not necessary.

If there is insufficient sample volume to perform a matrix spike and a matrix spike duplicate an LCS and an LCS DUP must be performed.

i. Relative percent difference (RPD) between the MS/MSD is used to calculate compliance. See current QC charts for control limits.

Calculation:

$$RPD = \frac{(MS - MSD) \times 100}{(MS + MSD) / 2}$$

MS = Method Spike Value
MSD = Method Spike Duplicate Value

If the RPD is outside of the acceptable control limits, the reported sample result is to be qualified with the [* flag.

3. Sample Result Calculations:

$$\frac{\text{Raw Data Result (ug/L)} \times \text{FV (L)} \times \text{DF}}{\text{Sample Weight (g)} \times \text{Dry Wt. (decimal form)}} = \text{Final Result (mg/kg dry weight corrected)}$$

DF = Dilution Factor
FV = Final Volume

SAFETY:

The toxicity or carcinogenicity of each reagent used in this method has not been fully established. Each chemical should be regarded as a potential health hazard and exposure should be as low as reasonably achievable. Laboratory staff should observe all safety procedures as outlined in the Laboratory Health and Safety Manual. Staff should consult Materials Safety Data Sheets (MSDS) for information on specific chemicals.

POLLUTION PREVENTION and WASTE MANAGEMENT:

Pollution prevention encompasses any technique that reduces or eliminates the quantity or toxicity of waste at the point of generation. Laboratory staff should order and prepare only those quantities of reagents that will be used prior to the expiration date. Other appropriate measures to minimize waste generation should be brought to the attention of laboratory management. All laboratory waste shall be handled as directed by the Laboratory Waste Management Plan and Hazardous Waste Contingency Plan.

Standard Operating Procedure

TITLE: Analysis of Polychlorinated Biphenyls (PCB's) by GC

MATRICES:

This SOP pertains to Aqueous and Solids Matrices

DETECTION LIMITS:

The Reporting Limits used in the absence of Project or State Specific Required Detection Limits are found Appendix B Table A.

1.0 **SCOPE AND APPLICATION:**

1.1 This SOP details procedures used in the Identification of PCB's as Aroclors; specifically Aroclors 1016, 1221, 1232, 1242, 1248, 1254, 1260, and their quantitation down to the ppb level, using gas chromatographs equipped with electron capture detectors.

Note: Soil and sediment sample results maybe corrected for moisture and reported on a dry weight basis if required by the Accrediting Authority.

2.0 **SUMMARY OF THE TEST METHOD:**

2.1 A volume of sample extract is injected into a gas chromatograph (GC) which is then split via a Y-splitter behind the injection port to a primary and confirmation column. Compounds are detected by dual electron capture detector (ECD). Results are reported in parts per billion (µg/kg or µg/L).

3.0 **DEFINITIONS:**

3.1 Terms used throughout this document are defined in Appendix A.

4.0 **INTERFERENCES:**

4.1 Solvents, reagents and glassware can interfere with sample analysis. En Chem purchases high quality solvents and reagents to eliminate this potential of contamination and require vendors to stand behind claims made on the solvents purchased. En Chem has adopted and implemented SOPs addressing proper glassware cleaning techniques, tailored to the department using the apparatus.

4.2 Solvents, reagents, glassware, and other sample processing may yield artifacts and /or interferences to sample analysis. All these materials are demonstrated to be free from interferences under the conditions of the analysis by analyzing method blanks. Interferences co-extracted from samples will vary considerably from source to source. If analysis of an extracted sample is prevented due to interferences, further clean-up of the sample may be necessary. Phthalate esters contaminate many types of products commonly found in laboratories. Plastics, in particular, must be avoided because phthalates are commonly used as plasticisers and are easily extracted from plastic materials.

4.3 Because of the concern of interferences affecting the quality of an analysis, all glassware and other materials that come into contact with the sample, directly or indirectly, are first rinsed with the extraction solvent.

5.0 **SAFETY:**

5.1 Safety is everyone's business at En Chem, Inc. and everyone is responsible for assisting in reducing unsafe and unhealthy working conditions or potential hazards. The company provides a safe place to work, but cooperation is needed to keep it safe. When something does not look safe, or someone is working in an unsafe manner, mention it to a Supervisor.

5.2 All department personnel shall be familiar with company safety policies as covered in the employee manual.

5.3 All samples should be treated as hazardous. Safety glasses, gloves, and lab coats are to be worn. The toxicity or carcinogenicity of each reagent used in this method has not been precisely defined. However, each chemical compound should be treated as a potential health hazard. Exposure to these chemicals must be reduced to the lowest possible level by a safe technique. A reference file of material safety data sheets should also be made available to all personnel involved in the chemical analysis.

5.4 PCBs have been tentatively classified as known or suspected human or mammalian carcinogens. Primary standards of these toxic compounds should be prepared in a hood.

5.5 MSDS sheets are kept by the department supervisor and are located in the sample receiving area on the bookshelf behind the sample log in computer. They should be consulted whenever there is a question related to material handling and safety. Also, read all chemical labels for special handling requirements, precautions, and warnings.

5.6 Required Safety Equipment is listed in Appendix B Table B

6.0 **EQUIPMENT AND SUPPLIES:**

6.1 See the following Tables in Appendix B for a summary:

Equipment	Table C
Supplies	Table D

7.0 **REAGENTS AND STANDARDS:**

7.1 See the following Tables in Appendix B for a summary:

Equipment	Table E
Supplies	Table F

8.0 **SAMPLE COLLECTION, PRESERVATION, SHIPMENT AND STORAGE:**

8.1 Soil samples are collected in 8 oz. amber jars with teflon lined caps; water samples are collected in 1 liter amber bottles with teflon caps. All samples are then transported on ice and stored at a temperature of 4 degrees Celsius. Soil samples must be extracted within 14 days from the sample date and water samples must be extracted within 7 days from the sample date..

8.2 Sample extracts are stored at 4 degrees Celsius. All extracts must be analyzed within 40 days from the extraction date and sample extracts are stored in the lab for 40 days after the date of sample analysis.

8.3 See Appendix B Table G for a summary.

9.0 **QUALITY CONTROL:**

9.1 Quality control procedures exist to ensure the proper production of data in all of the various sample preparation and/or sample analysis techniques. The laboratory must also maintain records to document the quality of the data generated.

9.2 An initial demonstration of capability (IDOC) is performed by analysts prior to analyzing samples.

9.3 To demonstrate capability, analyze at least four replicate aliquots of the well-mixed reference samples by the same procedures used to analyze actual samples.

9.4 Calculate the average recovery (x) in µg/L, and the standard deviation of the recovery (s) in µg/L, for each analyte of interest using the four results.

9.5 The data generated is compared to the single-laboratory performance in the authoritative sources .

9.6 Analytes that are outside source IDOC limits are reviewed as outlined below and IDOC are repeated.

9.7 Locate and correct the source of the problem. Document the changes made in the maintenance log.

9.8 Repeated failure, however, will confirm a general problem with the measurement system. If this occurs, locate and correct the source of the problem. Document the changes made in the maintenance log.

9.9 Repeat the test for all compounds of interest until resolved.

9.10 See Appendix B Table K for a summary of the method IDOC.

9.11 MDL studies are performed yearly. Detection, quantitation and reporting limits are set using the data generated from these studies. The studies conform to the processes as described Chapter One of SW846 and CFR 40 part 136.

9.12 See Appendix B Table A for a summary of detection limits.

9.13 Reference samples are purchased from a NIST certified vendor. Results of these studies are made available to analysts, management and accrediting authorities.

9.14 Retention time windows are crucial to the identification of target compounds. Retention time windows are established to compensate for minor shifts that occur as a result of sample loading and normal chromotographic variability. Establishment of the width of this window is of considerable importance. A window that is "tight" may lead in the reporting of both false negative and false positive results. Re- analysis occurs in this situation due to the non-identification of surrogate and spike components in the analysis. "Wide" retention times lead to false positive results that cannot be confirmed upon re-analysis.

9.15 Before establishing retention time windows, make sure that the chromatographic system is operating reliably and that the system conditions have been optimized for the target analytes and surrogates in the sample matrix to be analyzed. Make three injections of all single component standard mixtures over the course of a 72-hour period.

9.16 Record the retention time for each single component analyte and surrogate to three decimal places (e.g., 0.007). Calculate the mean and standard deviation of the three absolute retention times for each single component analyte and surrogate

9.17 If the standard deviation of the retention times for a target compound is much smaller than the default deviation (i.e., no difference between the absolute retention times), then the laboratory may use a default standard deviation of 0.01 minutes.

9.18 En Chem has in place procedures for documenting the effect of the matrix on method performance (precision and accuracy). Development of limits is done by randomly choosing points by matrix from the previous year. When new compounds are added for analysis, default limits are utilized until new limits can be established. The average percent recovery (p) and standard deviation (s) are calculated and the upper and lower warning and control limits are established by the following equations:

Warning Limits-

Upper Warning Limit (UWL) = p+2s
Lower Warning Limit (LWL) = p-2s

Control Limits-

Upper Control Limit (UCL) = p+3s
Lower Control Limit (LCL) = p-3s

Relative Percent Difference Limits -

%RPD = p+3s

where:

p = is the average relative percent difference
s = is the standard deviation of the relative percent difference.

NOTE: If %RPD control limits are calculated to be below 10%, the limit for that individual compound will be set at 10%. This limit allows for assessment of the data without compromising the known quality in a production setting. Most samples received are not for research.

9.19 Continuous charting for precision and accuracy is done. Limits are set yearly for Laboratory Control Spikes (LCS) and Matrix Spikes (MS) and surrogates.

9.20 Surrogate recoveries - The laboratory must evaluate surrogate recovery data from individual samples against the surrogate control limits developed by the laboratory. Development of limits is done by randomly choosing a minimum of 20 points by matrix from the previous year. The average percent recovery (p) and standard deviation (s) are calculated and the upper and lower control limits are established by the following equations:

Control Limits-

Upper Control Limit (UCL) = p+3s
Lower Control Limit (LCL) = p-3s

NOTE: Remember that even for a 95% confidence interval, 1out of every 20 observations is likely to fall outside of the control limits established.

9.21 For each analytical batch, not to exceed 20 environmental samples , a method blank, matrix spike and matrix spike duplicate/duplicate must be analyzed (the frequency of the spikes may be different for different monitoring programs but not to exceed 1 per 20 samples). The blank and spiked samples must be carried through all stages of the sample preparation and measurement steps.

9.22 A Laboratory Control Sample (LCS) and Duplicate (LCSD) also known as a Blank Spike (BS) and Duplicate (BSD) pair is also included with each analytical batch. The pair consists of an aliquot of a clean (control) matrix similar to the sample matrix and of the same weight or volume. The pair is spiked with the same analytes at the same concentrations as the matrix spike. When the results of the matrix spike analysis indicate a potential problem due to the sample matrix itself, these results are used to verify that the instrument was in control.

9.23 See Appendix B Table H for a summary of frequency by method citation.

9.24 When results of sample spikes indicate atypical method performance, if possible the sample is reanalyzed to confirm matrix interferences.

9.25 See Appendix B Tables listed below for limit summaries:

Surrogate Limits	Table K1
Matrix Spike Limits	Table K2

9.26 See Appendix B Tables listed below for summaries of review and corrective actions:

Analyst/Technician Data Assessment	Table L
Analyst/Supervisor Data Assessment	Table M
Report Reviewer/Project Management Data Assessment	Table N
Corrective Actions	Table O
Contingencies for Out of Control Data	Table P

10.0 **CALIBRATION AND STANDARDIZATION**:

10.1 Prior to running samples on any instrument, a 5 level calibration of Aroclors 1016,1260 and surrogate DCB and single level calibrations of Aroclors 1221,1232,1242,1248 and 1254 are performed and evaluated using linear regression. The resgression is done using the area of the standards. For a calibration to be valid it must have a correlation coefficient of 0.99 or greater and an initial calibration verification of 1016,1260, and DCB, ran right after the calibration, must exhibit recoveries of 85% to 115% of the true value. Arochlor identification uses 5 peaks for all Arochlors with the exception of Arochlor 1221 which will use 3 to five peaks.

10.2 A calibration check standard is run at the beginning and end of an analytical sequence of 20 or less samples or during each 12 hour time period whichever is more frequent and must exhibit recoveries of 85% to 115% of the true value.

10.3 To prepare the 5-level calibration standards10.0 ul each of 1000 ppm 1016 and 1260 and 5.0 ul of 200ppm DCB are diluted to 10.0 ml in hexane, to give a 1000 ppb 1660/100 ppb DCB standard. Then individual 800 ul, 500ul, 300ul, and 100ul aliquots of the 1000ppb standard is each further diluted in hexane to 1.0 ml to produce 800 ppb 1660/ 80 ppb DCB, 500 ppb 1660/ 50 ppb DCB, 300 ppb 1660/ 30 ppb DCB and 100 ppb 1660/ 10ppb DCB.

10.4 The stocks standards used for preparation of the 5-level calibration standards are from a different source than those used to prepare the initial calibration verification and calibration check standards. See Appendix B Table D for further information about stock standards.

10.5 Single level calibration standards are prepared for Aroclors 1221,1232,1242,1248,1254 at the 500 ppb level. The 500 ppb standards are prepared by individually diluting 5 ul of a 1000 ppm stock standard of the above Aroclors to 10 ml with hexane. These standards are used for pattern recognition of the above Aroclors and for quantitation should these Aroclors be found in the sample extract. See Appendix B Table D for further information about stock standards.

10.5 To prepare the initial calibration verification and calibration check standards 12.5 ul 1000ppm 1660 plus 6.25 ul of 200 ppm DCB are diluted to 25.0 ml with hexane to make a 500 ppb 1660 and 50 ppb DCB standard.

10.6 The stocks standards used for preparation of the initial calibration verification and calibration check standards are from a different source than those used to prepare the 5-level calibration standards. See Appendix B Table D for further information about stock standards.

11.0 PROCEDURE:

11.1 GC CONDITIONS:

Injection Volume		2.0 µL
GC Column GC1 (Dual column GC)	Column 1 -	DB-608 Capillary column, 30 m x 0.32mm I.D. (J&W Scientific).
	Column 2 -	DB-1701 Capillary column, 30 m x 0.32 mm I.D. (J&W Scientific).
GC 1 Column Conditions:		Carrier gas - Helium Flow rate - 2.0 mL/min. Make-up gas - Nitrogen Flow rate - 63 mL/min. Total column flow - 65 mL/min. Detector temp. -350° C Injector temp. - 200° C Splitless injection
GC1 Temperature Program:		Initial temp. - 150° C Initial time - 0.0 min. Rate (1) - 12° C/min. Final temp.-220°C Final time-1.0 min. Rate(2)- 3.0° C/min. Final temp.-245°C Final time-1.0 min. Rate(3)- 9.0° C/min. Final temp.-270°C Final time-6.0 min.

11.2 A method blank is injected to assure that the system is not contaminated. Prior to samples being injected a calibration check standard containing 1660 at the mid point concentration of the curve and again at the end of a sample run to prove the accuracy of the existing calibration. The calibration is determined to be valid if the calibration check standard values are within 85% to 115% of their true value. These checks are performed with each analytical batch of 20 or less samples or at the end of a 12 hour time period, whichever is more frequent. No data is reported from samples unless they are bracketed by passing calibration check standards.

11.3 An LCS/D pair is analyzed per analytical batch. If insufficient sample volume is supplied for a matrix spike, data from the LCS/D pair is used to prove the accuracy and the precision of the sample preparation and analytical process and enhance the reliability of data collected for samples.

11.4 An MS/D pair is analyzed per analytical batch when sufficient sample volume is supplied to the lab. This analysis is used to further prove the accuracy and the precision of the sample preparation and analytical process and determine if there are any matrix specific problems with the analytical process.

11.5 A method blank must be analyzed after the calibration standard to ensure that the total system (introduction device, transfer lines and GC system) is free of contaminants. If the method blank indicates contamination, then it may be appropriate to analyze another blank to demonstrate that the contamination is not a result of carryover from standards or samples.

11.6 If the second blank confirms contamination, the analyst must stop the analytical sequence determine the source of the contamination, eliminate the contamination if it involves a compound of interest, and begin the 12 hour sequence again.

11.7 All samples and QC are spiked with surrogate during sample preparation. Percent recoveries of the surrogates are analyzed to determine if the surrogate recoveries are within control limits. This is used to determine the effectiveness of the sample preparation process.

11.8 Samples are spiked with surrogate. Decachlorobiphenyl (DCB) recovery is assessed in each sample and QC standard against control limits. If no recognizable Aroclor pattern exists the sample is reported as non detect for the PCB Aroclors. Soil and sediment sample results are corrected for moisture and reported on a dry weight basis where required by the accrediting authority.

11.8 If interfering peaks are present in the chromatogram the extract, a Sulfur clean up must be performed prior to re analysis.

11.9 The GC makes a single injection and a y-splitter is used behind the injection port to split the injection onto two separate columns. The two columns have different stationary phases and cause the analytes to elute at different times. One of the columns is designated as the primary column and the other is designated as the confirmation column. A recognizable Aroclor pattern must be present on the primary and confirmation column before a positive ID of an Aroclor is made.

11.10 Samples with Aroclors present at concentrations above the range of the calibration are diluted within the range of the calibration standards and re analyzed.

11.11 If an Aroclor pattern exists on the primary column and is confirmed on the confirmation column then a positive ID for the Aroclor(s) are made and the concentration is reported. Soil and sediment sample results are corrected for moisture and reported on a dry weight basis where required by the accrediting authority.

12.0 CALCULATIONS:

Aqueous samples

$$\text{Concentration } (\mu g/L) = \frac{(A_x)(V_t)(D)}{(Cf)(V_i)}$$

Nonaqueous samples

$$\text{Concentration } (\mu g/kg) = \frac{(A_x)(V_t)(D)}{(Cf)(W)(S)}$$

where:
A_x = Area for selected peak.
V_t = Final volume of extract in liters
D = Dilution factor
Cf = calibration factor *
V_i = Initial sample volume (L)
W = Initial sample weight (grams)
S= % Solids/100

* Note: If Aroclor 1016 and/or 1260 is being quantified use the average calibration factor from the AR1660 curve. Surrogates are quantified based on the average calibration factors for DCB analyzed with the AR1660 curve.

Determination of the remaining 5 Arochlors is done by comparison against the area of a single 500 ppb calibration point.

13.0 METHOD PERFORMANCE:

13.1 The method performance is validated through the Method Detection Limit Study, Initial Validations and the Performance Testing Samples taken through this procedure.

13.2 See Appendix B Tables K for a Initial Demonstration of Capability summary.

14.0 POLLUTION PREVENTION:

14.1 Pollution prevention encompasses any technique or procedure that reduces or eliminates the quantity or toxicity of waste at the point of generation. Laboratory staff should order where possible acceptable non-toxic alternative supplies and prepare only those quantities of reagents or standards that will be used prior to the expiration date. Other appropriate measures to minimize waste generation should be brought to the attention of laboratory management. All laboratory waste shall be handled as directed by the Laboratory Waste Management Plan and Hazardous Waste Contingency Plan.

15.0 DATA ASSESSMENT AND ACCEPTANCE CRITERIA FOR QUALITY CONTROL MEASURES:

15.1 At a minimum there are three levels of data review:

15.2 Analyst/Technician verification at the bench top are looking at instrument performance as it relates to initial calibration, calibration verification, cleanliness.

15.3 Supervisor/Analyst verification after analysis are looking for sample concentration versus linear range of the instrument, typical patterns resulting from the compounds or elements in question, and quality control measure criteria. Data is either accepted without qualification, accepted with qualification, or rejected with samples reprocessed.

15.4 Report Reviewer/Project Management personnel look at the presentation of the data on final reports. The reports are verified for holding time compliance, receipt conditions, method citation and reasonableness, which may include field duplicate analysis, analytical comments and qualifications presented by the previous reviews, and comparing results of similar analytical techniques as it relates to the project site information when available.

15.5 A fourth level of review , data validation is determined from a project or program scope of laboratory services and encompasses the generation Quality Control summaries in the media of "Form Generation."

15.6 See En Chem SOP G3-SVO-18 Appendix B Table M through Table O for a summary of specific items cross checked.

16.0 CORRECTIVE ACTIONS FOR OUT-OF-CONTROL DATA:

16.1 Assessment of Quality Control measures is done to provide a level of confidence in the data generated. The measures provide documentation that the instrument conditions were reliable during the analysis

16.2 See Appendix B Table O for a summary.

17.0 CONTINGENCIES FOR HANDLING OUT-OF-CONTROL OR UNACCEPTABLE DATA:

17.1 During analysis, events occur specific to the physical and chemical characteristics of the environmental sample. When possible, with received sample volumes, data generated along with measures that do not meet statistical goals are re-analyzed again to see if the statistical goal can be achieved. When environmental samples do not meet statistical unacceptable data is generated. These events are different from those pertaining to instrument operating conditions. These events occur when the instruments are operating under ideal conditions.

17.2 If the blank fails surrogate recovery criteria, all of the associated samples, matrix spikes and laboratory control spikes will be evaluated and a corrective action will be determined.

17.3 If the blank contains any analyte of interest above the reporting limit, all of the associated samples, matrix spikes, and laboratory control spikes must be re-extracted unless the sample concentration is greater than 20 times the amount found in the blank or the analyte is not detected in an associated sample.

Note: For Wisconsin projects this criteria will be "Above the LOD".

17.4 If the laboratory control spike and duplicate do not meet the recovery criteria specified, the results of all QC performed with the samples will be evaluated by the analyst. Corrective actions include re-extraction of the samples or reanalysis of the extracts.

17.5 Sample matrix spike recoveries should fall within the specified limits . If a matrix spike recovery fails this criteria, the recovery of the other spiked sample in the MS/MSD pair should be evaluated. If recovery failures are duplicated then the sample matrix is confirmed as the problem and the data should be flagged and the failures discussed in the sample narrative. The LCS recoveries can be used to verify that the method was acceptable for the analysis in a clean matrix.

17.6 See Appendix B Table P for a summary.

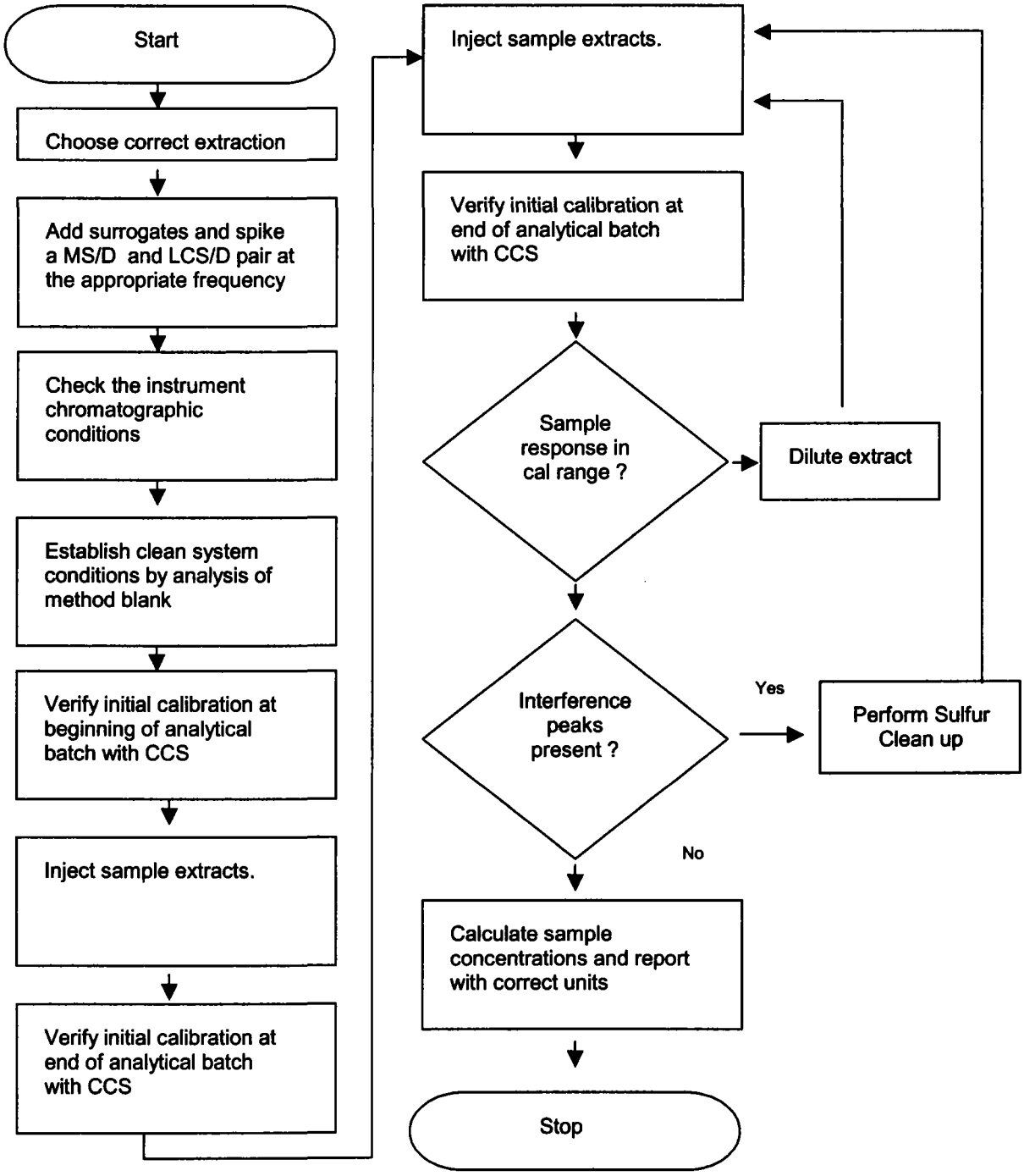
18.0 WASTE MANAGEMENT:

18.1 To minimize waste during sample preparation has two benefits. The first benefit is a cost savings to the lab in materials and supplies. The second is a benefit to the environment as less materials need to be disposed.

REFERENCES:

SW846 Method 8000B, December 1996
SW846 Method 8082, December 1996
40 CFR, EPA 608

FLOWCHART


Start
Choose correct extraction
Add surrogates and spike a MS/D and LCS/D pair at the appropriate frequency
Check the instrument chromatographic conditions
Establish clean system conditions by analysis of method blank
Verify initial calibration at beginning of analytical batch with CCS
Inject sample extracts.
Verify initial calibration at end of analytical batch with CCS
Inject sample extracts.
Verify initial calibration at end of analytical batch with CCS
Sample response in cal range ?
Dilute extract
Interference peaks present ?
Yes
Perform Sulfur Clean up
No
Calculate sample concentrations and report with correct units
Stop

VALIDATION DATA:

The laboratory must demonstrate initial proficiency with each combination of sample preparation and determinative methods utilized, by generating data of acceptable accuracy and precision for a reference sample containing the target parameters in a clean matrix. The laboratory must also repeat this demonstration whenever new staff are trained or significant changes in instrumentation are made. Analysts previously trained shall perform a continued demonstration of proficiency annually by performing "Yearly Demonstration of Capability" (YDC).

The values from the determinative method are presented as guidance only and are not intended as absolute acceptance criteria. En Chem generates data for method performance and are presented along with the performance values in Tables K and L.

MANAGEMENT APPROVAL AND REVIEW OF SOPS - POLICY AND DOCUMENTATION

REVIEWED BY: _____ Section Supervisor — Date: 01/07/2002

REVIEWED BY: M. Suha Quality Assurance Officer — Date: 01/07/2002

APPROVED BY: _____ Laboratory Manager — Date: 1/7/02

Periodic Review Record

Review Date	2/04/02					
Initials	MS/ARD					

APPENDICES

Appendix A

Acceptance Criteria: specified limits placed on characteristics of an item, process, or service defined in requirement documents. (ASQC)
Accuracy: the degree of agreement between an observed value and an accepted reference value. Accuracy includes a combination of random error (precision) and systematic error (bias) components which are due to sampling and analytical operations; a data quality indicator. (QAMS)
Analytical Detection Limit: the smallest amount of an analyte that can be distinguished in a sample by a given measurement procedure throughout a given (e.g., 0.95) confidence interval. (applicable only to radiochemistry)
Analyst: the designated individual who performs the "hands-on" analytical methods and associated techniques and who is the one responsible for applying required laboratory practices and other pertinent quality controls to meet the required level of quality. (NELAC)
Assessment Criteria: the measures established by NELAC and applied in establishing the extent to which an applicant is in conformance with NELAC requirements. (NELAC)
Data Reduction: the process of transforming raw data by arithmetic or statistical calculations, standard curves, concentration factors, etc., and collation into a more useable form. (EPA-QAD)
Deficiency: an unauthorized deviation from acceptable procedures or practices, or a defect in an item. (ASQC)
Demonstration of Capability: a procedure to establish the ability of the analyst to generate acceptable accuracy. (NELAC)
Detection Limit: the lowest concentration or amount of the target analyte that can be determined to be different from zero by a single measurement at a stated degree of confidence.
Document Control: the act of ensuring that documents (and revisions thereto) are proposed, reviewed for accuracy, approved for release by authorized personnel, distributed properly and controlled to ensure use of the correct version at the location where the prescribed activity is performed. (ASQC)
Duplicate Analyses: the analyses or measurements of the variable of interest performed identically on two sub-samples of the same sample. The results from duplicate analyses are used to evaluate analytical or measurement precision but not the precision of sampling, preservation or storage internal to the laboratory. (EPA-QAD)
Equipment Blank: a sample of analyte-free media which has been used to rinse common sampling equipment to check effectiveness of decontamination procedures. (NELAC)
estimate of Target analyte concentration is available. Matrix spikes are used, for example, to determine the effect of the matrix on a method's recovery efficiency. (QAMS)
Matrix Spike Duplicate (spiked sample or fortified sample duplicate): a second replicate matrix spike prepared in the laboratory and analyzed to obtain a measure of the precision of the recovery for each analyte. (QAMS)
May: denotes permitted action, but not required action. (NELAC)
Method Blank: a sample of a matrix similar to the batch of associated samples (when available) that is free from the analytes of interest, which is processed simultaneously with and under the same conditions as samples through all steps of the analytical procedures, and in which no target analytes or interferences are present at concentrations that impact the analytical results for sample analyses. (NELAC)
Method Detection Limit: the minimum concentration of a substance (an analyte) that can be measured and reported with 99% confidence that the analyte concentration is greater than zero and is determined from analysis of a sample in a given matrix containing the analyte. (40 CFR Part 136, Appendix B)
Must: denotes a requirement that must be met. (Random House College Dictionary)
National Institute of Standards and Technology (NIST): an agency of the US Department of Commerce's Technology Administration that is working with EPA, States, NELAC, and other

public and commercial entities to establish a system under which private sector companies and interested States can be accredited by NIST to provide NIST-traceable proficiency testing (PT) to those laboratories testing drinking water and wastewater. (NIST)

Negative Control: measures taken to ensure that a test, its components, or the environment do not cause undesired effects, or produce incorrect test results. (NELAC)

(items), and services. The quality system provides the framework for planning, implementing, and assessing work performed by the organization and for carrying out required QA and QC. (ANSI/ASQC E-41994)

Quantitation Limits: the maximum or minimum levels, concentrations, or quantities of a target variable (e.g., target analyte) that can be quantified with the confidence level required by the data user. (NELAC)

Range: the difference between the minimum and the maximum of a set of values. (EPA-QAD)

Raw Data: any original factual information from a measurement activity or study recorded in a laboratory notebook, worksheets, records, memoranda, notes, or exact copies thereof that are necessary for the reconstruction and evaluation of the report of the activity or study. Raw data may include photography, microfilm or microfiche copies, computer printouts, magnetic media, including dictated observations, and recorded data from automated instruments. If exact copies of raw data have been prepared (e.g., tapes which have been transcribed verbatim, data and verified accurate by signature), the exact copy or exact transcript may be submitted. (EPA-QAD)

Reagent Blank (method blank): a sample consisting of reagent(s), without the target analyte or sample matrix, introduced into the analytical procedure at the appropriate point and carried through all subsequent steps to determine the contribution of the reagents and of the involved analytical steps. (QAMS)

Record Retention: The systematic collection, indexing and storing of documented information under secure conditions. (EPA-QAD)

Reference Material: a material or substance one or more properties of which are sufficiently well established to be used for the calibration of an apparatus, the assessment of a measurement method, or for assigning values to materials. (ISO Guide 30-2.1)

Reference Method: a method of known and documented accuracy and precision issued by an organization recognized as competent to do so. (NELAC)

Reference Standard: a standard, generally of the highest metrological quality available at a given location, from which measurements made at that location are derived. (VIM-6.08)

Replicate Analyses: the measurements of the variable of interest performed identically on two or more sub-samples of the same sample within a short time interval. (NELAC)

Requirement:. denotes a mandatory specification; often designated by the term "shall". (NELAC)

Resource Conservation and Recovery Act (RCRA): the enabling legislation under 42 USC 321 *et seq.* (1976), that gives EPA the authority to control hazardous waste from the "cradle-to-grave", including its generation, transportation, treatment, storage, and disposal. (NELAC)

Sample Duplicate: two samples taken from and representative of the same population and carried through all steps of the sampling and analytical procedures in an identical manner. Duplicate samples are used to assess variance of the total method including sampling and analysis. (EPA-QAD)

Selectivity: (Analytical chemistry) the capability of a test method or instrument to respond to a target substance or constituent in the presence of non-target substances. (EPA-QAD)

Sensitivity: the capability of a method or instrument to discriminate between measurement responses representing different levels (e.g., concentrations) of a variable of interest. (NELAC)

Shall: denotes a requirement that is mandatory whenever the criterion for conformance with the specification requires that there be no deviation. This does not prohibit the use of alternative approaches or methods for implementing the specification so long as the requirement is fulfilled.

(ANSI)
Should: denotes a guideline or recommendation whenever noncompliance with the specification is permissible. (ANSI)
Spike: a known mass of target analyte added to a blank sample or sub-sample; used to determine recovery efficiency or for other quality control purposes. (NELAC)
Standard: the document describing the elements of laboratory accreditation that has been developed and established within the consensus principles of NELAC and meets the approval requirements of NELAC procedures and policies. (ASQC)
Standard Operating Procedures (SOPs): a written document which details the method of an operation, analysis or action whose techniques and procedures are thoroughly prescribed and which is accepted as the method for performing certain routine or repetitive tasks. (QAMS)
Standardized Reference Material (SRM): a certified reference material produced by the U.S. National Institute of Standards and Technology or other equivalent organization and characterized for absolute content, independent of analytical method. (EPA-QAD)
Supervisor (however named): the individual(s) designated as being responsible for a particular area or category of scientific analysis. This responsibility includes direct day-to-day supervision of technical employees, supply and instrument adequacy and upkeep, quality assurance/quality control duties and ascertaining that technical employees have the required balance of education, training and experience to perform the required analyses. (NELAC)
Surrogate: a substance with properties that mimic the analyte of interest. It is unlikely to be found in environment samples and is added to them for quality control purposes. (QAMS)
Systems Audit (also Technical Systems Audit): a thorough, systematic, qualitative on-site assessment of the facilities, equipment, personnel, training, procedures, record keeping, data validation, data management, and reporting aspects of a total measurement system. (EPA-QAD)
Test: a technical operation that consists of the determination of one or more characteristics or performance of a given product, material, equipment, organism, physical phenomenon, process or service according to a specified procedure. The result of a test is normally recorded in a document sometimes called a test report or a test certificate. (ISO/IEC Guide 2-12.1, amended)
Test Method: an adoption of a scientific technique for a specific measurement problem, as documented in a laboratory SOP. (NELAC)
Tolerance Chart: A chart in which the plotted quality control data is assessed via a tolerance level (e.g. +/- 10% of a mean) based on the precision level judged acceptable to meet overall quality/data use requirements instead of a statistical acceptance criteria (e.g. +/- 3 sigma) (applies to radiobioassay laboratories). (ANSI)
Toxic Substances Control Act (TSCA): the enabling legislation in 15 USC 2601 *et seq.*, (1976), that provides for testing, regulating, and screening all chemicals produced or imported into the United States for possible toxic effects prior to commercial manufacture. (NELAC)
Traceability: the property of a result of a measurement whereby it can be related to appropriate standards, generally international or national standards, through an unbroken chain of comparisons. (VIM-6.12)
United States Environmental Protection Agency (EPA): the federal governmental agency with responsibility for protecting public health and safeguarding and improving the natural environment (i.e., the air, water, and land) upon which human life depends. (US-EPA)
Validation: the process of substantiating specified performance criteria. (EPA-QAD)
Verification: confirmation by examination and provision of evidence that specified requirements have been met. (NELAC)NOTE: In connection with the management of measuring equipment, verification provides a means for checking that the deviations between values indicated by a measuring instrument and corresponding known values of a measured quantity are consistently smaller than the maximum allowable error defined in a standard, regulation or specification peculiar to the management of the measuring equipment. The result of verification leads to a decision either to restore in service, to perform adjustment, to repair, to downgrade, or

to declare obsolete. In all cases, it is required that a written trace of the verification performed shall be kept on the measuring instrument's individual record.

Work Cell: a well-defined group of analysts that together perform the method analysis. The members of the group and their specific functions within the work cell must be fully documented. (NELAC)

Appendix B

Tables

Table A	**Reporting limits**
Table B	**Required Safety Equipment**
Table C	**Equipment**
Table D	**Supplies**
Table E	**Reagent**
Table F	**Standards**
Table G	***Sample Collection and Preservation***
Table H	**Quality Control**
Table J	**Calibration and Standardization**
Table K	**Initial Demonstration of Capability**
Table K1	**Surrogate Limits**
Table K2	**Matrix Spike Limits**
Table L	**Analyst/Technician Data Assessment**
Table M	**Analyst/Supervisor Data Assessment**
Table N	**Report Reviewer/Project Management Data Assessment**
Table O	**Corrective actions**
Table P	**Contingencies for handling out of control or unacceptable data**

Table A

REPORTING LIMITS

Parameter	Aqueous Reporting Limit	Soil Reporting Limit
AROCLOR 1016	1.0	50
AROCLOR 1221	1.0	50
AROCLOR 1232	1.0	50
AROCLOR 1242	1.0	50
AROCLOR 1248	1.0	50
AROCLOR 1254	1.0	50
AROCLOR 1260	1.0	50

Units = ug/L Units = ug/kg

Table B

REQUIRED SAFETY EQUIPMENT

Item:	Safety Apparel Description	Mandatory for Procedure	Optional
1	Safety Goggles or Glasses		x
2	Lab Coat		x
3	Gloves		x
4	Face Shield		x

Table C

EQUIPMENT

Equipment	Manufacturer	Model(s)	Serial #	Date In Service
PCB 1 gas chromatograph	Hewlett Packard	5890 series II	921A23066	January 2000
PCB 1 auto injector	Hewlett Packard	7673A	837A1029	January 2000
PCB 1 controller module	Hewlett Packard	7683A	303A10177	January 2000
PCB 1 autosampler tray	Hewlett Packard	7683A	18A07338	January 2000
PCB 1 computer	Compaq	PC 350	13BK62E764	January 2000
PCB 1 monitor	Princeton	E050	72055349	January 2000

Table D

SUPPLIES

Supplies	Manufacturer	Vendor	Catalog #
PCB 1016	Restek	Restek	32006
PCB 1221	Restek	Restek	32007
PCB 1232	Restek	Restek	32008
PCB 1242	Restek	Restek	32009
PCB 1248	Restek	Restek	32010
PCB 1254	Restek	Restek	32011
PCB 1260	Restek	Restek	32012
PCB 1016	Supelco	Supelco	48097
PCB 1260	Supelco	Supelco	44809
PCB surrogate (TCMX; DCB)	Restek	Restek	32000
9" disposable pipets	Fisher Scientific	Fisher Scientific	13-678-20-C
pH indicator strips (0-14 range)	Whatman	VWR	EM-9590-3
Disposable culture tubes	VWR	VWR	60825-618
25 mL adjustable repipetor	Brinkman	VWR	
Lab notebooks	VWR	VWR	51280-108
Turbovap concentrator tube (200 mL; 1.0 mL endpoint)	Zymark	Zymark	45817
600 mL beakers	Kimble	Fisher Scientific	13910-145
250 mL beakers	Kimble	Fisher Scientific	14005-250
150 mL beakers	Kimble	Fisher Scientific	02539J
5 3/4" disposable pipets	Fisher Scientific	Fisher Scientific	13-678-20-A
Amber jars 9 oz.	QEC	VWR	2124-0009
heater block	Kontes	VWR	720000-0000
ebullator	Kontes	VWR	569401
filters	Dionex	Dionex	049458
collection vials	I-Chem		405531
1 liter Amber bottles; level 3 certified; with Teflon lined cap	QEC	QEC	3121-0033
25 uL gastight syringe	Hamilton	Fisher Scientific	24559

Supplies	Manufacturer	Vendor	Catalog #
50 uL gastight syringe	Hamilton	Fisher Scientific	111111
100 uL gastight syringe	Hamilton	Fisher Scientific	13-684-100
250 uL gastight syringe	Hamilton	Fisher Scientific	81100
500 uL gastight syringe	Hamilton	Fisher Scientific	81217
1000 uL gastight syringe	Hamilton	Fisher Scientific	1482425

Table E

REAGENTS

Reagent	Purity	Manufacturer	Vendor	Catalog #
Methylene Chloride	Pesticide Quality	Burdick & Jackson	Fisher Scientific	CS300-200
Nitrogen	99.99%	---	---	---
Di-ionized Water	Type I ASTM	---	---	---
Sodium sulfate (anhydrous; 10-60 mesh)	ASC grade	Fisher Scientific	Fisher Scientific	MK802406
Acetone	Pesticide Quality	Burdick & Jackson	Fisher Scientific	010-4
Hexane	Pesticide Quality	Burdick & Jackson	Fisher Scientific	217-4

Table F

STANDARDS

Standard	Acronym	Concentration	Direction found in Section:	Alias
Intial calibration verification	ICV	500ppb 1660; 50ppb DCB	Calibration and standardization	NA
Calibration check standard	CCS	500ppb 1660; 50ppb DCB	Calibration and standardization	Chkstd
100 ppb calibration standard	100 ppb	100ppb 1660; 10ppb DCB	Calibration and standardization	Level 1 calibration Std
300 ppb calibration standard	300 ppb	300ppb 1660; 30ppb DCB	Calibration and standardization	Level 2 calibration Std
500 ppb calibration standard	500 ppb	500ppb 1660; 50ppb DCB	Calibration and standardization	Level 3 calibration Std
800 ppb calibration standard	800 ppb	800ppb 1660; 80ppb DCB	Calibration and standardization	Level 4 calibration Std
1000 ppb calibration standard	1000 ppb	1000ppb 1660; 100ppb DCB	Calibration and standardization	Level 5 calibration Std
500 ppb 1221 calibration standard	500 ppb 1221	500 ppb	Calibration and standardization	Single level 1221
500 ppb 1232 calibration standard	500 ppb 1232	500 ppb	Calibration and standardization	Single level 1232
500 ppb 1242 calibration standard	500 ppb 1242	500 ppb	Calibration and standardization	Single level 1242
500 ppb 1248 calibration standard	500 ppb 1248	500 ppb	Calibration and standardization	Single level 1248
500 ppb 1254 calibration standard	500 ppb 1254	500 ppb	Calibration and standardization	Single level 1254

Table G

SAMPLE COLLECTION, PRESERVATION, SHIPMENT AND STORAGE

Matrix	Prep Method	Container(s)	Preservation	Shipment Conditions	Lab Storage Conditions
Aqueous	SW-846 3510	Amber bottle 1 liter	None	On ice 4+/- 2° Celsius	Refrigerate 4+/- 2° Celsius
Aqueous	EPA 608	Amber bottle 1 liter Add Sodium thiosulfate if residual chlorine is present	None	On ice 4+/- 2° Celsius	Refrigerate 4+/- 2° Celsius
Soil	SW-846 3545	Amber jars 9 oz	None	On ice 4+/- 2° Celsius	Refrigerate 4+/- 2° Celsius
Soil	SW-846 3545	Amber jars 9 oz	None	On ice 4+/- 2° Celsius	Refrigerate 4+/- 2° Celsius
Soil	EPA 608	Amber jars 9 oz	None	On ice 4+/- 2° Celsius	Refrigerate 4+/- 2° Celsius

Table H

QUALITY CONTROL

Preparation Method ⇨ Quality Control Measure ⇩	**SW846 8082**	**EPA 608**
Initial Calibration	Minimum of five levels; lowest level near but above MDL.	Minimum of five levels; lowest level near but above MDL.
Method Blank	One per batch of samples, up to 20 samples, whichever is more frequent.	One per batch of samples, up to 20 environmental samples, whichever is more frequent.
Calibration check Standard (CCS)	One at the beginning of a 12 hour time clock or more frequent.	One at the beginning of a 12 hour time clock or more frequent.
Independent Calibration Verification	Independent source run immediately after the calibration curve.	Independent source run immediately after the calibration curve.
Laboratory Control Spike and Duplicate	One pair per batch of samples, up to 20 environmental samples, whichever is more frequent.	One pair per batch of samples, up to 20 environmental samples, whichever is more frequent.
Matrix Spike and Duplicate	One pair per batch of samples, up to 20 environmental samples, whichever is more frequent.	One pair per batch of samples, up to 20 environmental samples, whichever is more frequent.
Method Validation	Annually	Annually
MDL	Annually	Annually
Surrogate Standards	Added to every sample.	Added to every sample.

Table J

CALIBRATION AND STANDARDIZATION

Analytical Method Acceptance Criteria⇒ Calibration Measure ⇓	**Frequency**	**Acceptance Criteria**
Initial Calibration	• Installation of new column • Creation of new analytical method • CVS fails criteria	• Linear Regression and Correlation Coefficient is > 0.9900.
Independent Calibration Verification Standard	• After every calibration	• 85% TO 115% of the true value
Calibration check standard	• At the beginning of every 12 hour shift or more frequent	• 85% TO 115% of the true value
Laboratory Control Spike and Duplicate	• One pair per batch of samples, up to 20 environmental samples, whichever is more frequent.	• See appendix B;Table R under Lab Control Spike Recovery Limits
Matrix Spike and Duplicate	• One pair per batch of samples, up to 20 environmental samples, whichever is more frequent.	• See appendix B;Table R under Matrix Spike Recovery Limits
MDL's	• Annually	• 1-10 times the MDL should be equal to the spike concentration that was used to determine the MDL's
Method Validation	• Annually	• See Table K and L under Initial Demonstration of Capability
Surrogate Standards	• Added to every sample.	• In house limits determined. See appendix B;Table R under Surrogate recovery Limits

Table K

En Chem Initial Demonstration of Capability

Compound/Element	Acceptable Recov. from Authoritative Method	En Chem Initial Demonstration Recovery
1016 Aqueous	70%-130%	93%
1016 Soil	70%-130%	112%
1260 Aqueous	70%-130%	92%
1260 Soil	70%-130%	114%

Table K1
Surrogate Limits

Matrix ⇨ Effective Date: June 2001	Aqueous		Solid	
Surrogate Spike Recovery Parameters ⇩	LCL	UCL	LCL	UCL
Decachlorobiphenyl	22	133	11	142

Table K2
Matrix Spike Recovery Limits

Matrix ⇨ Effective Date: June 2001	Aqueous			Solid		
Surrogate Spike Recovery Parameters ⇩	LCL	UCL	RPD	LCL	UCL	RPD
Arochlor 1016	27	117	21	24	124	24
Arochlor 1260	31	141	18	26	122	24

Table L
ANALYST/TECHNICIAN DATA ASSESSMENT

Analytical Method Acceptance Criteria⇒ Data Assessment Measure ⇓	Method Citation SW If these conditions are not achieved ⇒	Method Citation EPA If these conditions are not achieved ⇒	Method Citation EPA If these conditions are not achieved ⇒
Initial Calibration	• 1	• 1	•
Initial Calibration Verification	• 1	• 1	•
Calibration Blank	• 1	• 1	•
Method Blank	• 2	• 2	•
Internal Standard Area	• 3	• 3	•
Calibration check standard	• 4	• 4	•
Surrogate Recovery	• 5	• 5	•
Accuracy & Precision Laboratory Control Spikes	• 6	• 6	•
Accuracy & Precision Matrix Spikes	• 7	• 7	•

1. Check instrumentation/equipment condition; enter maintenance in instrument log; perform another initial calibration. No data can be reported. Generate on Non-Conformance Memo.

2. In the absence of project specific requirements, sample detects greater than 20 times the method blank contamination level are reported with those not exceeding 20 times re-analyzed or qualified. Generate on Non-Conformance Memo.

3. In the absence of project specific requirements, check to be sure that there are no errors in the calculations. If errors are found, recalculate the data accordingly. Examine chromatograms for interfering peaks and integrated peak areas. Re analyze sample if volume allows. Generate on Non-Conformance Memo.

4. In the absence of project specific requirements, immediately after the flawed CCV analyze another CCV or re-analyzed all samples set after the last acceptable CCV. Generate on Non-Conformance Memo.

5. In the absence of project specific requirements, check to be sure that there are no errors in the calculations. If errors are found, recalculate the data accordingly. Examine chromatograms for interfering peaks and integrated peak areas. If the program allows report the concentrations noting the surrogate recovery is most likely due to co-elution with target compounds. Some samples may require dilution in order to bring one or more target analytes within the calibration range or to overcome significant interferences with some analytes. Re analyze sample if volume allows. If the surrogate recoveries are available from a less-diluted or undiluted aliquot of the sample or sample extract, those recoveries may be used to demonstrate that the surrogates were within the in-house QC limits, and no further action is required. If, upon re-analysis the recovery is again not within limits, report the data as an "estimated concentration." If the recovery is within the limits in the re-analysis, provide the re-analysis data to the data user. Generate on Non-Conformance Memo.

6. In the absence of project specific requirements, the LCS and LCSD are spiked with the same analytes at the same concentrations as the matrix spike. When the results of the matrix spike analysis indicate a potential problem due to the sample matrix itself, the LCS results are used to verify that the laboratory can perform the analysis in a clean matrix. If the LCS and LCSD recoveries do not meet those limits as required in the method or program, the samples shall be re-analyzed. If sample volume does not allow re-analysis the entire prep/analytical batch of samples shall be flagged to reflect the deficiencies. Generate on Non-Conformance Memo.

7. In the absence of project specific requirements, when the results of the matrix spike analysis indicate a potential problem due to the sample matrix itself, the LCS results are used to verify that the laboratory can perform the analysis in a clean matrix. If sample volume allows re-analysis, it shall be performed to verify the matrix effect. When sample volume does not re-analysis, the parent sample and samples from that project site shall be flagged to reflect the deficiencies. Generate a Non- Conformance Memo.

Table M
ANALYST/SUPERVISOR DATA ASSESSMENT

Analytical Method Acceptance Criteria⇒ Data Assessment Measure ⇓	Method Citation SW If these conditions are not achieved ⇒	Method Citation EPA If these conditions are not achieved ⇒	Method Citation EPA If these conditions are not achieved ⇒
Initial Calibration	• 1	• 1	•
Initial Calibration Verification	• 1	• 1	•
Calibration Blank	• 1	• 1	•
Method Blank	• 2	• 2	•
Internal Standard Area	• 3	• 3	•
Calibration Check Standard	• 4	• 4	•
Surrogate Recovery	• 5	• 5	•
Accuracy & Precision Laboratory Control Spikes	• 6	• 6	•
Accuracy & Precision Matrix Spikes	• 7	• 7	•

1. Check instrumentation/equipment condition; enter maintenance in instrument log; perform another initial calibration. No data can be reported. Generate on Non-Conformance Memo.

2. In the absence of project specific requirements, sample detects greater than 20 times the method blank contamination level are reported with those not exceeding 20 times re-analyzed or qualified. Generate on Non-Conformance Memo.

3. In the absence of project specific requirements, check to be sure that there are no errors in the calculations. If errors are found, recalculate the data accordingly. Examine chromatograms for interfering peaks and integrated peak areas. Re analyze sample if volume allows. Generate on Non-Conformance Memo.

4. In the absence of project specific requirements, immediately after the flawed CCV analyze another CCV or re-analyzed all samples set after the last acceptable CCV. Generate on Non-Conformance Memo.

5. In the absence of project specific requirements, check to be sure that there are no errors in the calculations. If errors are found, recalculate the data accordingly. Examine chromatograms for interfering peaks and integrated peak areas. If the program allows report the concentrations noting the surrogate recovery is most likely due to co-elution with target compounds. Some samples may require dilution in order to bring one or more target analytes within the calibration range or to overcome significant interferences with some analytes. Re analyze sample if volume allows. If the surrogate recoveries are available from a less-diluted or undiluted aliquot of the sample or sample extract, those recoveries may be used to demonstrate that the surrogates were within the in-house QC limits, and no further action is required. If, upon re-analysis the recovery is again not within limits, report the data as an "estimated concentration." If the recovery is within the limits in the re-analysis, provide the re-analysis data to the data user. Generate on Non-Conformance Memo.

6. In the absence of project specific requirements, the LCS and LCSD are spiked with the same analytes at the same concentrations as the matrix spike. When the results of the matrix spike analysis indicate a potential problem due to the sample matrix itself, the LCS results are used to verify that the laboratory can perform the analysis in a clean matrix. If the LCS and LCSD recoveries do not meet those limits as required in the method or program, the samples shall be re-analyzed. If sample volume does not allow re-analysis the entire prep/analytical batch of samples shall be flagged to reflect the deficiencies. Generate on Non-Conformance Memo.

7. In the absence of project specific requirements, when the results of the matrix spike analysis indicate a potential problem due to the sample matrix itself, the LCS results are used to verify that the laboratory can perform the analysis in a clean matrix. If sample volume allows re-analysis, it shall be performed to verify the matrix effect. When sample volume does not re-analysis, the parent sample and samples from that project site shall be flagged to reflect the deficiencies. Generate a Non- Conformance Memo.

Table N
REPORT REVIEWER/PROJECT MANAGEMENT DATA ASSESSMENT

Analytical Method Acceptance Criteria⇒ Data Assessment Measure ⇓	**Method Citation SW If these conditions are not achieved ⇒**	**Method Citation EPA If these conditions are not achieved ⇒**	**Method Citation EPA If these conditions are not achieved ⇒**
Holding Time Compliance	• 1	• 1	•
Sample Receipt Conditions and Preservation	• 2	• 2	•
Method Citation	• 3	• 3	•
Reasonableness: Field Duplicate	• 4	• 4	•
Reasonableness: Analytical Comments Qualifications	• 5	• 5	•

1. Flag results with an H and the number of days past hold in parenthesis e.g. H(5).

2. In prose describe the receipt conditions as they relate to the acceptance criteria list in Table H

3. Compare to regulatory program in QAPjP or Chain of Custody.

4. Are duplicate with 40% agreement. If not, internally flag for review.

5. Do related suites of tests agree or are reasonable.

Table O
CORRECTIVE ACTIONS

Analytical Method Acceptance Criteria⇨ Quality Control Measure ⇩	**Method Citation SW If these conditions are not achieved ⇒**	**Corrective Action (Key below)**	**Method Citation EPA If these conditions are not achieved ⇒**	**Corrective Action (Key below)**
Initial Calibration		• 1	•	• 1
Method Blank		• 2	•	• 2
Calibration Check Standard		• 3	•	• 3

1. Check instrumentation/equipment condition; enter maintenance in instrument log; perform another initial calibration

2. Samples detects greater than 20 times the method blank contamination level are reported with those not exceeding 20 times re-analyzed or qualified.

3. Immediately after the flawed CCV analyze another CCV or re-analyzed all samples set after the last acceptable CCV.

Table P
CONTINGENCIES FOR HANDLING OUT-OF-CONTROL OR UNACCEPTABLE DATA

Analytical Method Acceptance Criteria⇒ Quality Control Measure ⇓	Method Citation SW If these conditions are not achieved ⇒	Corrective Action (Key below)	Method Citation EPA If these conditions are not achieved ⇒	Corrective Action (Key below)
Matrix Spike and Duplicate	•	• 1	•	• 1
Internal Standards	•	• 2	•	• 2
Surrogate Standards	•	• 2	•	• 2

1. If Lab Control Spikes pass acceptance criteria, flag parent sample and samples from the entire project with a QC batch associated with analysis indicated matrix effects.

2. When sample volume is available reanalyze the samples. If the second analysis indicates the same internal standard recoveries add analytical comments and effects on data.

STANDARD OPERATING PROCEDURE

TITLE: Mercury Analysis of Aqueous Samples by CVAA

DEPARTMENT: Wet Chemistry

MATRICES: Ground waters, Wastewaters, Drinking Waters, Leach Test Extracts, Seawater, and Brines

DETECTION LIMITS: The method detection limit is 0.088 ug/L and the EQL is 0.20 ug/L.

SCOPE AND APPLICATION: This procedure measures total Mercury (organic and inorganic) in drinking water, surface water, ground waters, leaching extracts, industrial wastewaters, and domestic wastewaters.

REFERENCE: EPA Method 245.1, 600 4-82-055, December 1982
USEPA Test Methods for Evaluating Solid Waste SW-846, 3rd Edition, Method 7470A
USEPA Test Methods for Evaluating Solid Waste SW-846, 3rd Edition, Method 7000, Section 8.7

PROCEDURE SUMMARY:

Cold vapor atomic absorption utilizes the volatile property of elemental mercury at the 253.7 nm wavelength. To release mercury from organic complexes, the sample is digested with oxidizing reagents and acids in a hot water bath. After digestion, the oxidizing reagents are neutralized. Stannous chloride is added to reduce ionic mercury to the ground state. A Flow Injection Analysis System sweeps the volatile elemental mercury out of the sample and into the cell of an atomic absorption spectrophotometer. The absorbance signal is proportional to the amount of mercury in the sample.

REVIEWED BY: Michael J. Helmann — 3/12/02
Michael J. Helmann
Wet Chemistry Supervisor
Date

Julie A. Trivedi — 3/12/02
Julie A. Trivedi
Quality Assurance Officer
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APPROVED BY: Glen A. Coder — 3/12/02
Glen A. Coder
Laboratory Manager
Date

Annual Review

Date:						
Initials:						

DEFINITIONS:

Calibration Blank-A volume of reagent water acidified with the same acid matrix as in the calibration standards. The calibration blank is a zero standard and is used to auto-zero the instrument.

Calibration Standard-A solution prepared from the dilution of stock standard solutions. The calibration solutions are used to calibrate the instrument response with respect to analyte concentration.

Laboratory Control Sample (LCS)- An aliquot of reagent water or other blank matrices to which a known quantity of the method analyte is added in the laboratory. The LCS is analyzed exactly like a sample, and its purpose is to determine whether the methodology is in control and whether the laboratory is capable of making accurate measurements.

Laboratory Control Sample Duplicate (LCSD)-A second aliquot of reagent water or other blank matrices to which a known quantity of the method analyte is added in the laboratory. . The LCSD is analyzed exactly like a sample, and its purpose is to determine whether the methodology is in control and whether the laboratory is capable of making accurate and precise measurements.

Linear Range-The concentration range over which the instrument response to an analyte is linear.

Matrix Spike (MS)-An aliquot of an environmental sample to which a known quantity of the method analyte is added in the laboratory. The MS is analyzed exactly like a sample, and its purpose is to determine whether the sample matrix contributes bias to the analytical results. The background concentration of the analyte in the sample matrix must be determined in a separate aliquot a bias to the analytical results. The background concentration of the analyte in the sample matrix must be determined in a separate aliquot and the measured values in the MSD are corrected for background concentrations.

Method Blank- An aliquot of reagent water or other blank matrices that are treated exactly as a sample including exposure to all glassware, equipment, solvents, reagents, and internal standards that are used with other samples. The method blank is used to determine if the method analyte or other interferences are present in the laboratory environment, reagents, or apparatus.

Method Detection Limit (MDL)-The minimum concentration of an analyte that can be identified, measured, and reported with 99% confidence that the analyte concentration is greater than zero.

Method of Standard Addition (MSA)-The addition of a known amount of analyte to the sample in order to determine the relative response of the detector to the analyte within the sample matrix. The relative response is then used to assess either an operative matrix effect or the sample analyte concentration.

Stock Standard-A concentrated solution containing one or more method analytes prepared in the laboratory using assayed reference materials or purchased from a reputable commercial source.

INTERFERENCES:

Potassium permanganate is added to eliminate possible interference from sulfide. Concentrations as high as 20 mg/L of sulfide as NaS do not interfere with the recovery of added inorganic mercury from D.I. water.

Copper has also been reported to interfere; however, copper concentrations as high as 10 mg/L had no effect on recovery of mercury from spiked samples.

Seawaters, brines and industrial effluents high in chlorides require additional permanganate, as much as 25 mL, because during the oxidation step, chlorides are converted to free chlorine which also absorbs radiation at 253.7 nm. Care must therefore be taken to ensure that free chlorine is absent before the mercury is reduced and swept into the cell. This may be accomplished by using an excess of hydroxylamine hydrochloride reagent, 6 mL.

Certain volatile organic materials that absorb at this wavelength may also interfere. A preliminary run without reagents would determine if this type of interference is present.

SAFETY:

The toxicity or carcinogenicity of each reagent used in this method has not been fully established. Each chemical should be regarded as a potential health hazard and exposure should be as low as reasonably achievable. Laboratory staff should observe all safety procedures as outlined in the Laboratory Health and Safety Manual. Staff should consult Material Safety Data Sheets (MSDS) for information on specific chemicals.

EQUIPMENT AND SUPPLIES:

Balance
BOD bottles: 300 mL
Graduated cylinders: 50 mL, 100 mL
Glass beaker: 1000 mL
Volumetric flasks: 200 mL, 1000 mL, 2000 mL
Volumetric pipets: 20 mL
Eppendorf pipettor and tips
Acid and reagent repipettors
Water bath(s)
Stir bar
Stir plate
Oxford pipettor and tips

REAGENTS AND STANDARDS:

Deionized (D.I.) water
Certified standard solution: 1000 mg/L (1000 ppm)
Second Source Certified standard solution: 1000 mg/L (1000 ppm)
Sulfuric acid (H_2SO_4): concentrated, reagent grade
Nitric acid (HNO_3): concentrated, distilled or mercury-free
Hydrochloric acid (HCL): concentrated, trace grade
Potassium permanganate, low mercury, 5% solution (w/v)

Potassium persulfate, 5% solution (w/v)
Sodium chloride
Hydroxylamine hydrochloride
Stannous chloride

Prepare Potassium permanganate, 5% solution (w/v):
- Dissolve 100 grams potassium permanganate ($KMnO_4$) in 2 liters D.I. water.

Prepare Potassium persulfate, 5% solution (w/v):
- Dissolve 50 grams potassium persulfate ($K_2S_2O_8$) with D.I. water in a 1 liter volumetric flask.

- Warm to dissolve.

Prepare Sodium chloride - hydroxylamine hydrochloride Solution:
- Dissolve 120 grams sodium chloride (NaCl) and 120 grams hydroxylamine hydrochloride ($NH_2OH \cdot HCl$) in 1 liter D.I. water.

Prepare Stannous chloride Solution:
- Add 11 grams stannous chloride ($SnCl_2 \cdot 2H_20$) and 30 mL conc. HCL to 500 mL D.I. water in a 1 liter volumetric flask.

- Dissolve and dilute to volume with D.I. water.

NOTE: These solutions should be prepared fresh monthly.

Prepare Intermediate Calibration Standard, 1 ppm:
- Acid rinse a 100 mL volumetric flask, first with 1:1 HNO_3, then with D.I. water.

- Add 50 - 70 mL D.I. water.

- Add 0.5 mL HNO_3. Wash down with D.I. water.

- Pipet 0.1 mL standard solution (1000 ppm) into the flask using a calibrated Eppendorf pipettor.

- Bring to volume with D.I. water, invert 6 times, and label as 1 ppm Hg intermediate standard.

Prepare Working Calibration Standard, 0.1 ppm:
- Acid rinse a 50 mL volumetric flask.

- Add 0.3 mL HNO_3 to flask. Rinse down with D.I. water and swirl.

- Pipet 5 mL 1 ppm Hg intermediate standard into the flask using a glass or calibrated pipettor.

- Bring to volume with D.I. water, invert 6 times, and label as 0.1 ppm Hg working standard.

Prepare Calibration Standards
- Label one BOD bottle as a blank.

- Label one bottle as a zero standard and one bottle each as 0.2, 1.0, 5.0, 10.0 ppb standards. Using a calibrated pipettor, transfer 0.2, 1.0, 5.0 and 10.0 mL, respectively, of 0.1 ppm working standard into each bottle of the pair. A 2.0 ppb standard (0.20 mL of 1.0 ppm working standard) is necessary for EPT digestions.

- Fill with D.I. water so that the total volume in each bottle equals 100 mL. Use a graduated cylinder.

Prepare Laboratory Control Sample (LCS)

- Transfer 100 mL D.I. water into a BOD bottle using a graduated cylinder.

- Pipet 0.5 mL Hg intermediate standard into bottle using a calibrated Eppendorf pipettor. Label as LCS.

Prepare ICB, CCB and MB (Initial Calibration Blank, Continuing Calibration Blank and Method Blank)

- Transfer 100 mL D.I. water into BOD bottles using a graduated cylinder. Label as ICB, CCB, or MB.

Prepare ICV and CCV (Initial Calibration Verification and Continuing Calibration Verification)

- Acid rinse a 100 mL volumetric flask, first with 1:1 HNO_3, then with D.I. water.

- Add 50 - 70 mL D.I. water.

- Add 0.5 mL HNO_3. Wash down with D.I. water and swirl.

- Pipet 0.1 mL second source standard solution (1000 ppm) into the flask using a calibrated Eppendorf pipettor.

- Bring to volume with D.I. water, invert 6 times, and label as 1 ppm Hg intermediate standard.

- Transfer 100 mL D.I. water into BOD bottles using a graduated cylinder.

- Pipet 0.4 mL 1 ppm Hg intermediate into each BOD bottle using a calibrated Eppendorf pipettor. Label as ICV - 4 ppb or pipette 0.5 mL 1 ppm Hg intermediate standard into each BOD bottle. Label as CCV.

SAMPLE COLLECTION, PRESERVATION, AND STORAGE:

Samples may be collected in glass or plastic bottles. Samples are preserved by adjusting the pH to < 2 with nitric acid (HNO_3). The holding time for preserved samples is 28 days.

QUALITY CONTROL:

Quality Control, Quantified by Comparison to Standard Curve

Correlation Coefficient (r-value)
The correlation coefficient, the measure of linearity of a standard curve, must be 0.995 or greater. If the value is less than 0.995 recalibrate the instrument.

Initial Calibration Verification (ICV)
The ICV is prepared from a source independent from the calibration standards and must be analyzed immediately after calibration. The ICV must meet the rejection criteria of ± 10% of the true value. Recalibrate if the ICV fails. The concentration of the ICV should be near the mid-point of the calibration curve.

Initial Calibration Blank (ICB)
The ICB must be analyzed after the ICV. The absolute value must be ≤EQL. Recalibrate if it fails.

Continuing Calibration Verification (CCV)
The CCV is analyzed after every 10 samples. Rejection criteria is ± 10% of true value. If the CCV fails, the problem must be corrected and the previous 10 samples between the CCV and last CCB must be reanalyzed. Concentration of the CCV should be near the mid-point of the calibration curve.

Continuing Calibration Blank (CCB)
The CCB is analyzed after every CCV. The absolute value must be ≤EQL. If the CCB fails, the problem must be corrected and the previous 10 samples between the last CCB and the CCV must be reanalyzed.

Laboratory Control Sample (LCS)
The LCS is carried through all preparation procedures and analyzed for each matrix type with a frequency of 5%. See current QC charts for control ranges. In cases where the LCS is outside of acceptable ranges all samples prepared in that batch must be re-prepared and re-analyzed.

Method Blank (MB)
A MB is carried through all prep procedures and analyzed with a frequency of 5%. Rejection criteria is <LOD. Other criteria may apply, such as regulatory limit and the analyte concentration in the samples.

Matrix Spike/Matrix Spike Duplicate
One matrix spike and matrix spike duplicate are analyzed for each group of samples that are similar in matrix at a frequency of 5%. Both QC samples must be calculated for accuracy. See current QC charts for control range.

If the both spike recoveries are outside of the specified control limit, the corresponding parent sample is to be post-spiked and the reported result shall be flagged with the "N" qualifier. The control limits for a post-spike are 75-125%. If the post-spike recovery is out-of-control, dilute the corresponding sample and perform a post-spike on the diluted aliquot of sample. Dilute appropriately until an acceptable recovery is obtained.

If the analyte of interest is greater than the linear range, dilute appropriately and post-spike the sample; however, the "N" qualifier is not required. Also, if the analyte of interest is greater than 4x the level of the spike concentration, accuracy calculations are not necessary.

The relative percent difference (RPD) is used to determine whether the precision criteria have been met. Please refer to the current list of control limits. If the RPD is outside of the acceptable control limits, the reported sample result is to be qualified with the "*" flag.

If there is insufficient sample volume to perform a matrix spike and a matrix spike duplicate, an LCS and LCS DUP must be performed.

For EPA METHOD 245.1:

Warm up the lamp at least 30 minutes prior to analysis.

Perform a matrix spike at a minimum of 10% of the samples or one per sample batch, whichever is more frequent.

CALIBRATION AND STANDARDIZATION:

Please refer to EnChem SOP MET-57 for instrument calibration and standardization procedures.

PROCEDURE:

1. Collect samples and bring to room temperature. (Use tubs of hot water if necessary).

2. Heat water bath(s) to 95°C. Water in bath should reach above liquid level in BOD bottles. Note temperature of bath into water bath log book.

3. Prepare reagents as necessary.

4. Write up run on digestion bench sheet and CVAA aqueous bench sheet. Identify the samples for Quality Control..

5. Collect and label acid-washed BOD bottles and acid-washed glassware.

6. Transfer 100 mL, or an aliquot diluted to 100 mL with D.I. water, of each well-mixed sample into a BOD bottle using a graduated cylinder.

7. Spike designated samples by pipetting 0.5 mL 1 ppm Hg intermediate standard into BOD bottles using a calibrated Eppendorf pipettor.

8. All BOD bottles should contain 100 mL volume before adding reagents. Keep stoppers on bottles to avoid contamination problems.

CAUTION: CONTINUE DIGESTION IN HOOD. WEAR SAFETY GLASSES AND GLOVES.

NOTE: All reagents should be added in equal amounts to standards and samples.

9. Add 5 mL concentrated H_2SO_4 using an acid repipettor and mix.

10. Add 2.5 mL concentrated distilled HNO_3 using acid repipettor and mix.

11. Add 15 mL potassium permanganate solution using a calibrated pipettor. Mix, adding additional potassium permanganate crystals if necessary until purple color persists.

12. Add 8 mL potassium persulfate using a calibrated pipettor and mix.

13. Heat BOD bottles in 95°C water bath for 2 hours. (Check to ensure purple color is maintained. Add potassium permanganate crystals if needed. The same amount of crystals must be added to all samples, standards, and QC samples.)

14. Cool bottles to room temperature before analysis.

15. Set up AA instrument for cold vapor analysis. See EN CHEM METHOD MET-57.

16. Add 6 mL sodium chloride-hydroxylamine hydrochloride solution to each BOD bottle using an repipettor. Swirl until no color persists.

CALCULATIONS:

Aqueous Sample Calculation:

Raw Data result (ug/L) X DF = Final Result (ug/L)

DF = Dilution Factor

$$\text{Spike Percent Recovery} = \frac{SSR - SR}{SA}$$

SSR = Spike Sample Result
SR = Sample Result
SA = Spike Added

$$RPD = \frac{MS - MSD}{(MS+MSD)/2} \times 100$$

MS = Method Spike Value
MSD = Method Spike Duplicate Value

POLLUTION PREVENTION:

Pollution prevention encompasses any technique that reduces or eliminates the quantity or toxicity of waste at the point of generation. Laboratory staff should order and prepare only those quantities of reagents that will be used prior to the expiration date. Other appropriate measures to minimize waste generation should be brought to the attention of laboratory management. All laboratory waste shall be handled as directed by the Laboratory Waste Management Plan and Hazardous Waste Contingency Plan.

WASTE MANAGEMENT:

USEPA requires that laboratory waste handling practices conducted be consistent with all applicable rules and regulations. Excess reagents, samples, and method process wastes should be characterized and disposed of in an appropriate manner. Please refer to the EnChem Standard Operating Procedures GEN-5 and GEN-12 for additional information on the disposal of environmental samples and other laboratory wastes.

Standard Operating Procedure

TITLE: Extraction of Water Samples for PCB's.

DEPARTMENT: Semivolatile Organics

REFERENCES: SW-846 3500B Organic Extraction and Preparation
Revision 2, December 1996
SW-846 3510C Separatory Funnel Liquid-Liquid Extraction
Revision 3,December 1996

PROCEDURE SUMMARY:

Waters are collected in a teflon lined cap amber bottle and must be extracted within 7 days of sample collection. All samples must be stored on ice or between 0.0-4.0 C prior to extraction. A known amount of sample is introduced into a separatory funnel with a solvent. Diluent solvent is collected and concentrated to a final volume. The final extract volume is stored in a crimp top auto sampler vial from which an aliquot is injected onto the GC system, depending on the determinitive method to be used.

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M. Suha _____ 4/28/2000
Michael C. Suha — Date
Quality Assurance Officer

APPROVED BY: _____ 4/28/2000
Nils K. Melberg — Date
Laboratory Manager**

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1.0 SCOPE AND APPLICATION

1.1 This method describes a procedure for isolating organic compounds from aqueous samples. The method also describes concentration techniques suitable for preparing the extract.

1.2 This method is applicable to the isolation and concentration of water insoluble and slightly water-soluble organics in preparation for a variety of chromatographic procedures.

2.0 SUMMARY OF METHOD

2.1 A measured volume of sample, usually 1 liter, at a neutral pH, is serially extracted with methylene chloride using a separatory funnel. The extract is dried, concentrated, and, as necessary, exchanged into a solvent compatible with the cleanup or determinative method to be used.

3.0 INTERFERENCES

3.1 Solvents, reagents and glassware can interfere with sample analysis. En Chem purchases high quality solvents and reagents to eliminate this potential of contaminations and require these vendors to stand behind claims made on the solvents purchased. En Chem has adopted and implemented SOPs addressing proper glassware cleaning techniques, tailored to the department using the apparatus.

4.0 APPARATUS AND MATERIALS

4.1 Labline Automated Shaker System, including 2-liter extraction glassware with Teflon stopcock.

4.2 Drying funnel - glass funnel with glass wool at the bottom and filled with baked sodium sulfate.

4.3 Zymark 1.0 ml endpoint tubes.

4.4 Zymark TurboVap II Concentration Workstation.

4.5 Glass beakers-six 250 mL.

4.6 Vials - 12 mL, amber, glass

4.7 pH indicator paper - pH of sample indicated by color coding.

4.8 Disposable pasteur pipettes with pipette bulbs.

4.9 Syringes - 10 uL, 50 uL, 100uL and 1.0 mL

4.10 Graduated cylinder - 1 liter.

4.11 High purity nitrogen gas source.

4.12 5 mL and 10 mL volumetric flasks.

5.0 REAGENTS

5.1 Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available. Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination. Reagents should be stored in glass to prevent the leaching of contaminants from plastic containers.

5.2 Organic-free reagent water - Culligan purified/treated water.

5.3 Sodium sulfate (granular, anhydrous), Na_2SO_4. Purify by heating at 400-C for 4 hours in a shallow tray, or by precleaning the sodium sulfate with methylene chloride. If the sodium sulfate is precleaned with methylene chloride, a method blank must be analyzed, demonstrating that there is no interference from the sodium sulfate. J.T. Baker(ACS).

5.4 Extraction/exchange solvents.

5.4.1 Methylene chloride, CH_2Cl_2 - Pesticide quality or equivalent - Fisher.

5.4.2 Hexane, C_6H_{14} -Pesticide quality or equivalent -Burdick and Jackson

6.0 SAMPLE COLLECTION, PRESERVATION, AND HANDLING

6.1 Waters should be contained in unpreserved liter amber bottles. All samples should be transported on ice and stored at a temperature of 4C. Extraction must take place within 7 days of sampling. Analysis must take place within 40 days of extraction.

7.0 PROCEDURE

7.1 Decant the liquid sample (1.0 L) into the extraction glassware. If less than 1.0 L is present, mark the miniscus of the sample with a marker, and save the amber jar for measurement with a 1.0-L graduated cylinder. If high concentrations are anticipated, a smaller volume may be used and then diluted with organic-free reagent water to 1 liter.

Note: All glassware and other materials that come into contact with the sample, directly or indirectly, are first rinsed with the the extraction solvent.

7.2 Spiking/Surrogate solutions:

7.2.1 Surrogate Standards:

Stock standards from Restek are used to prepare the surrogate spikes. The stock standard contain TCMX and DCB at 200 ug/ml. 125 ul of the stock standed is diluted to 5.0 ml with methylene chloride to make an intermediate standard at a concentration of 5000 ug/L. 100 ul of the intermediate surrogate standard is added to all samples, method blanks, and quality control samples. After extraction the samples are concentrated to a final volume of 10.0 mL such that the true concentration of the surrogates in the samples are 50.0 ug/L. Shelf-life of the solutions is 6 months from the date of preparation. See appendix A for more details.

7.2.2 Spike Standards:

Stock standards from Restek are used at 1000 ug/mL. A different lot number from the calibration standards are used to prepare the spike standards. Intemediate standards are prepared from the stock standards at 5000 ug/L by diluting 50 ul of the stock standard to10 mL. 1.0 mL of the intermediate is spiked into the BS, BSD, MS, MSD; after extraction the sample is concentrated to 10.0 mL such that the true value of the spiked samples will be 500 ug/mL. Shelf-life of the solutions is 6 months from the date of preparation. See appendix B for more details.

The standards used for the LCS and MS are an Arochlor 1016/1260 mixture.

7.3 Add 60 mL of methylene chloride to the separatory funnel.

7.4 Seal the extraction glassware and set the shaking time to 3.0 minutes. Set the speed to 30 r rotatations/minute(full speed).

NOTE: Methylene chloride creates excessive pressure very rapidly. However, venting takes place through a small hole in the side cap of the extraction glassware. For this reason, the automated shaker should always be placed in a hood-ventilation system when using solvent.

7.6 Allow the organic layer to separate from the water phase for a minimum of 2 minutes. If the emulsion interface between layers is more than one-third the size of the solvent layer, the analyst must employ mechanical techniques to complete the phase separation. The optimum technique depends upon the sample and may include stirring, filtration of the emulsion through glass wool, centrifugation, collection of the emulsion in a beaker of sodium sulfate and stirring to break the emulsion, or other physical methods. Collect the solvent extract in 200ml TurboVap tubes. (The tubes should have drying funnels, which contain glass wool and sodium sulfate, positioned on top of the mouth of the tube, for filtration.)

7.7 Repeat the extraction two more times using 60-mL portions of fresh solvent (Sections 7.4 through 7.5). combine the three solvent extracts together in the concentrator tubes. Label the combined extract with it's appropriate sample number.

7.8 Perform the concentration using the Zymark evaporative concentrator technique (Sections 7.9.1 through 7.9.5).

7.9 Zymark TurboVap II Concentration Technique

7.9.1 Rinse the drying funnels with small amounts - 20 mLs of methylene chloride.

7.9.2 Place the concentrator tubes in the TurboVap II water bath, which is set at 35 C. Set the TurboVap in sensor mode. Start each cell that contains a concentrator tube and set the pressure on the TurboVap to 11 psi.

7.9.4 When the extract is concentrated to 0.75 ml, the nitrogen will automatically shut off and an alarm will sound. At this time, add approximately 25.0 ml of hexane for a solvent exchange.

7.9.5 Concentrate the extract to 0.75 ml using the sensor mode. Add about 25.0 ml of hexaneagain and concentrate to 0.75 ml. Remove the tube from the water bath and allow to cool. Rinse only the conical part of the tube, bringing the final volume of the extract to 10.0 ml.

7.9.6 Tranfer the extract to 12.0 ml vials.

7.9.7 The samples are now ready analysis.

8.0 Deviations from the cited references

8.1 Method 3510C recommends the use of Kaderna -Danish glassware and a water bath for the concentration step. Here the use of the Turbo-vap system by Zymark is used to accomplish this portion of the method.

Appendix A

SPIKING STANDARDS FOR PCB

SURROGATE SPIKING SOLUTION for PCB

The mixture contains the following components at 200 ug/mL:

Decachlorobiphenyl
2,4,5,6-tetrachloro-m-xylene

Appendix B

SPIKING STANDARDS FOR PCB

SPIKING SOLUTIONS for PCB

The following aroclors are used for spiking matrix spike and LCS samples and are periodically rotated.

Aroclors are at 1000 ug/mL

Aroclor 1016
Aroclor 1221
Aroclor 1232
Aroclor 1238
Aroclor 1242
Aroclor 1248
Aroclor 1254
Aroclor 1260

STANDARD OPERATING PROCEDURE

TITLE: TCLP - Toxicity Characteristic Leaching Procedure

DEPARTMENT: Leaching

MATRICES: All

DETECTION LIMITS: Not applicable

SCOPE AND APPLICATION: The TCLP is designed to determine the mobility of inorganic and organic contaminants present in liquid, solid, and multi-phase wastes. This specific method produces an extract for inorganic and organic analysis.

REFERENCES: USEPA Test Method for Evaluating Solid Wastes, SW 846, 3rd Edition, Method 1311

PROCEDURE SUMMARY:

For liquid wastes that contain less than 0.5 percent solids, the TCLP extract is the waste filtered through a glass fiber filter.

For wastes containing greater than or equal to 0.5 percent solids, the particle size of the waste is reduced if necessary and any initial liquid phase is separated and stored for later analysis. The solid phase is extracted with an amount of extraction fluid equal to 20 times the weight of the solid phase. One of two different extraction fluids is used, depending upon the alkalinity of the solid phase. After extraction, the liquid extract is separated from the solid phase by filtration through a glass fiber filter. If compatible, any initial liquid phase is combined with the liquid extract and the two are analyzed together. (If multiple phases form upon combination, the liquids are analyzed separately. The results are mathematically combined to yield a volume-weighted average concentration.)

REVIEWED BY: _____ 3/12/02
Michael J. Helmann — Date
Wet Chemistry Supervisor

_____ 3/12/02
Julie A. Trivedi — Date
Quality Assurance Officer

APPROVED BY: _____ 3/12/02
Glen A. Coder — Date
Laboratory Manager

Annual Review

Date:					
Initials:					

INTERFERENCES:

Potential interferences are discussed in the individual preparation and analytical methods.

SAFETY:

The toxicity or carcinogenicity of each reagent used in this method has not been fully established. Each chemical should be regarded as a potential health hazard and exposure should be as low as reasonably achievable. Laboratory staff should observe all safety procedures as outlined in the Laboratory Health and Safety Manual. Staff should consult Materials Safety Data Sheets (MSDS) for information on specific chemicals.

EQUIPMENT AND SUPPLIES:

Agitation equipment: Capable of rotating the extraction vessel in an end-over-end fashion at 30 ± 2 rpm.
Extraction bottles: 2-liter, wide-mouth Nalgene/ 2-liter Teflon or glass bottles for wet chemistry or organic parameters
Filtering assembly:

- Millipore filter apparatus, nitric-acid rinsed.
  NOTE: Wet chemistry glassware should be D.I. water rinsed; organic glassware acetone rinsed.
- Filter flask: 2 liter, nitric-acid rinsed.
- Filters: Borosilicate glass fiber, 0.6 to 0.8 micron pore size
  NOTE: When evaluating the mobility of metals, filters should be acid-washed prior to use by rinsing with 1 N nitric acid followed by two consecutive rinses with D.I. water (a minimum of 4 liters per rinse is recommended. Do not acid-rinse filters for wet chemistry or organic parameters).

pH meter and probe
Balance: Accurate to within ± 0.01 grams (weight measurements are to ± 0.1 grams).
Vacuum pump
Centrifuge
Centrifuge bottles: 1 liter, plastic, nitric-acid rinsed (for metals) or Teflon bottles acetone-rinsed (for organics) or Teflon bottles D.I. rinsed for wet chemistry
Magnetic heating stir plate and stir bars
Beakers: 500 mL, glass with watch glass covers
Volumetric flasks: 1 liter, glass
Graduated cylinders: 100 mL, 2000 mL
Forceps: blunt tip

REAGENTS AND STANDARDS:

(Reagent Shelf Life = 1 Year)
Deionized (D.I.) water
Hydrochloric acid, HCl, 1 N
Nitric acid, HNO_3, 1 N
Sodium hydroxide, NaOH, 10 N
Glacial acetic acid, HOAc
Extraction fluid #1: Add 68.4 mL glacial HOAc to 12 Liters D.I. water. Add 77.1 mL 10 N NaOH. Correct pH of this fluid is 4.93 ± 0.05.
Extraction fluid #2: Dilute 68.4 mL glacial HOAc with D.I. water to a volume of 12 liters. Correct pH of this fluid is 2.88 ± 0.05.

NOTE: These extraction fluids should be monitored frequently for impurities. Check the pH daily, recording pH to nearest 0.01 pH unit, prior to use to ensure that it is accurate. If impurities are found or the pH is not within specifications, the fluid should be discarded and fresh extraction fluid prepared. Extraction fluid solutions are kept for a maximum of one week.

pH buffers: 4, 5, 7,10 pH
Nitric acid (HNO_3): concentrated, distilled. (Used as a preserving agent.)

Prepare sodium hydroxide solution, 10 N

- Weigh 400 grams NaOH into a 1-Liter volumetric flask. Dilute with D.I. water.

CAUTION: This solution will react and the glassware will be hot to the touch.

SAMPLE COLLECTION, PRESERVATION, AND STORAGE:

Samples are collected using an appropriate sampling plan and kept in closed containers prior to extraction. No preservatives are added to samples. (They may be refrigerated unless irreversible physical changes occur. If precipitation occurs, the entire sample should be extracted, including precipitate.) TCLP extracts are stored at 4°C and should be analyzed as soon as possible.

The samples must be extracted within the holding times specified below. The hold time is in days from the date of field sampling to the date of TCLP extraction. Following preparation of the TCLP extract, the holding times specified in the SOP's for sample preparation and analysis shall be applied.

Sample Parameter	*Hold Time*
Volatiles	14
Semi-volatiles	14
Mercury	28
Metals, except Mercury	180

QUALITY CONTROL:

Preparation
A minimum of one blank for every 20 samples should be employed. If both solutions (#1 and #2) are used in a day's work, use extraction fluid #1 for the blank. If only #2 has been used for samples, a leach blank with #2 should be used.

Every 20 times a Teflon bottle is used, a memory blank will be leached. Each time a Teflon bottle is used, it should be recorded in the log book. Each time a Teflon bottle is used as a blank, the number of the bottle should be recorded on the bench sheet.

Analytical
One matrix spike (MS) is performed for each waste type. A minimum of one MS is required for each analytical batch.

For metalic analytes the Method of Standard Additions, MSA, will be employed if: 1) Recovery of the analyte in the MS is less than 50% and the concentration of the analyte in the sample leachate does not exceed the regulatory limit, AND, 2) The concentration of the analyte in the extract is within 20% of the regulatory limit.

A post digestion spike is performed on any sample which contains an analyte within ± 20% of the regulatory limit to test for the presence of spectral and physical interferences. The post spike acceptance limits are 80-120% of the true value. Samples which fail this criteria are analyzed and reported at a dilution.

CALIBRATION AND STANDARDIZATION:

The pH of the extraction fluid is verified prior to use. The pH of Extraction fluid #1 must be 4.93 ± 0.05. The pH of Extraction fluid #2 must be 2.88 ± 0.05.

PROCEDURE:

A. Preliminary Evaluations

Before leaching, perform these evaluations on a representative waste sample of at least 100 grams. (These test samples won't be extracted, but are used for the preliminary evaluation.)

- Percent Solids of Waste.
- Percent Solids < 0.5% (insignificant solids) or ≥ 0.5%.
- Particle Size of Waste Capable of Passing Through 9.5 mm Sieve.
- Determination of Which of Two Extraction Fluids to Use.

Percent Solids of Waste

1. If waste obviously will not yield free liquid during pressure filtration, (i.e., 100% solids), go directly to Evaluation 3, Particle Size.
2. If waste is a liquid or a multi-phase sample, separate the phases by filtration as follows:

NOTE: Some wastes, such as oily wastes or paint wastes, contain material that appears to be liquid, but may not filter. The material within the filtration device is defined as a solid. Do not replace the original filter with a fresh one during this determination.

a. Pre-weigh the glass filter and the container that will receive the filtrate. Assemble the filter and filtering assembly.

b. Weigh out representative sample of waste (100 gram minimum) and record weight to 0.1 grams.

c. To aid filtration, allow slurries to stand to permit the solids to settle. The liquid phase may be filtered before the solid portion of the waste. (Centrifugation may be used to separate wastes that settle slowly.)

d. Quantitatively transfer entire waste sample to filter holder. Spread evenly over surface of filter. Sample should be allowed to reach room temperature if a refrigerated sample would yield less liquid.

NOTE: If some waste material adheres to container and cannot be transferred into filter holder (>1 percent of sample weight), subtract this residue weight from the original sample weight.

e. Gradually apply pressure of 1-10 psi until air moves through filter. If this point is not reached under 10 psi and if no additional liquid has passed through the filter in any 2 minute interval, slowly increase the pressure in 10-psi increments to a maximum of 50 psi.

NOTE: Instantaneous application of high pressure can degrade the glass filter and may cause plugging. When air begins to move through the filter, or when the liquid flow has

ceased at 50 psi (no filtrate within any 2 minute period), stop the filtration.

f. The material in the filter holder is defined as the solid phase of the waste and the filtrate is defined as the liquid phase.

g. Refer to the TCLP logbook example in Appendix 1.

Percent Solids < 0.5% or ≥ 0.5%

1. If waste obviously has a significant amount of solid material, the solid phase must be extracted. Go to Evaluation 3, Particle Size.

2. To determine if waste has < 0.5% solids (no extraction needed):

   a. Remove the solid phase and filter from the filtration apparatus.

   b. Dry the filter and solid phase at 100 ± 20°C until two successive weighings yield the same value within ±1 percent. Record the final weight.

   NOTE: It is recommended that the drying oven be vented to a hood. Take care that the solid will not flash upon heating.

   c. Refer to TCLP logbook example in Appendix 1 .

   d. If the percent dry solids is less than 0.5 percent, the waste (after filtration) is considered the TCLP extract. Proceed to Section B: Extraction Procedure.
   If the percent dry solids is greater than or equal to 0.5 percent, go to Evaluation 3, Particle Size.

Particle Size of Waste

1. Use a fresh representative waste sample for this determination.

2. Evaluate the particle size of the solid portion of the waste. Particle-size reduction is required unless the solid has a surface area per gram of material equal to or greater than 3.1 cm^2. (It would be capable of passing through a 9.5 mm standard sieve.)

3. If necessary, the solid portion of the waste is prepared for extraction by crushing, cutting, or grinding.

NOTE: Surface area requirements are meant for waste materials such as paper or cloth.

Determination of Appropriate Extraction Fluid

1. Use a fresh portion of the solid phase of the waste for this evaluation.

2. Weigh out a small subsample of solid phase, reduce the particle size to approximately 1 mm in diameter or less, if necessary, and transfer 5.0 grams into a 150 mL beaker.

3. Add 96.5 mL D.I. water to the beaker, cover with a watchglass and stir vigorously for 5 minutes using magnetic stirrer.

4. Calibrate the pH meter. (See En Chem SOP, LAB-7.) Measure and record the pH of the sample. If the pH is <5.0, use extraction fluid #1. Go to Section B: Extraction Procedure.

5. If the pH is >5.0, add 3.5 mL 1 N HCl, slurry briefly, cover with a watchglass, heat to 50° C, and hold at 50° C for 10 minutes.

6. Let the solution cool to room temperature.

7. Measure and record the pH. If the pH is <5.0, use extraction fluid #1. If the pH is >5.0, use extraction fluid #2. Proceed to Section B: Extraction Procedure.

8. Discard waste samples used for evaluation purposes.

B. Extraction Procedure

1. If the waste will obviously yield no liquid during filtration, weigh out a representative subsample, 100 gram minimum, record weight to nearest 0.1 gram, and proceed to Step 3.

2. If the waste is a liquid or a multi-phase sample, separate the phases as follows:

   a. Preweigh the container that will receive the filtrate. Assemble the filtering apparatus.

   b. If metals are to be analyzed: Acid wash the filter with 1 N HNO_3, according to the directions under the EQUIPMENT AND SUPPLIES section.

   c. Weigh out a representative subsample of waste, 100 gram minimum, and record the weight to nearest 0.1 gram.

   NOTE: If the sample contains <0.5 percent dry solids, the liquid phase is filtered and considered the extract. Enough sample should be filtered so that all necessary analyses can be performed on the filtrate. Likewise, use enough sample with >0.5 percent solids to generate enough solids for a representative extraction.

   d. To aid filtration, allow slurries to stand to permit the solids to settle. The liquid phase may be filtered before the solid phase. (Centrifugation may be used to separate wastes that settle slowly.)

   e. Quantitatively transfer entire waste sample to filter holder. Spread evenly over surface of filter. Sample should be allowed to reach room temperature if a refrigerated sample would yield less liquid.

   NOTE: If some waste material adheres to container and cannot be transferred into filter holder, (>1 percent of sample weight), subtract this residue weight from the original sample weight.

   f. Gradually apply pressure of 1-10 psi until air moves through filter. If this point is not reached under 10 psi and if no additional liquid has passed through the filter in any 2-minute interval, slowly increase the pressure in 10-psi increments to a maximum of 50 psi.

   NOTE: Instantaneous application of high pressure can degrade the glass filter and may cause plugging. When air begins to move through filter or when the liquid flow ceases at 50 psi, stop the filtration.

g. The material in the filter holder is the solid phase of the waste, and the filtrate is the liquid phase. Weigh the filtrate. The liquid phase may be analyzed or stored at 4°C and added as "free liquids" in step 9, depending upon the percent solids of the sample.

3. If the waste contains <0.5 percent dry solids, go to Step 10. If the waste contains >0.5 percent dry solids and if particle-size reduction is needed, proceed to Step 4. If the waste could pass through a 9.5 mm sieve, quantitatively transfer the solid phase into an extraction bottle along with any filter used to separate the phases. Proceed to Step 5.

4. Prepare the solid portion of the waste by crushing, cutting, or grinding until the particle size has been reduced as necessary. Quantitatively transfer the solids into an extraction bottle along with any filter used to separate the phases.

NOTE: Sieving is not normally required; if needed, use a Teflon-coated sieve to avoid contamination.

5. Determine the amount of extraction fluid to add to the extraction bottle:

$$\text{Weight of Extraction Fluid} = \frac{[20] \times [\%\ \text{solids}] \times [\text{weight of waste filtered}]}{100}$$

For example, use 2000 mL extract fluid for 100 grams dry sample.

6. Slowly add this amount of the appropriate extraction fluid (as determined in preliminary evaluations) to the extraction bottle. Close the bottle tightly (Teflon tape can be used for a better seal) and secure in agitation device.

7. Rotate at 30 ± 2 rpm for 18 ± 2 hours. The rotation speed of the tumbler must be monitored. On a weekly basis, determine the speed of the tumbler and record the speed in the logbook designated for that purpose. The temperature of the room where the extraction takes place shall be maintained at 23 ± 2°C. The room temperature is monitored on a continous basis. The room temperature is acceptable if it is within the range of 21-25°C for at least 14 hours of the 18 hour tumbling period.

NOTE: Pressure may build up within the extraction bottle as agitation continues, (e.g., limed, or calcium carbonate-containing, waste may evolve gases such as carbon dioxide.) To relieve excess pressure, the bottle may be periodically opened and vented into a hood, i.e., after 15 minutes, 30 minutes and 1 hour.

8. Following the 18 ± 2 hours extraction, filter the material in the extraction bottle through a new glass filter as outlined in steps 2e and 2f. The glass fiber filter may be changed to facilitate filtration. (If metals are to be analyzed, all filters should be acid-rinsed with 1 N HNO_3.)

9. Prepare the TCLP extract as follows:

a. If the waste contained no initial liquid phase, the filtered liquid material from Step 8 above is defined as the TCLP extract.

b. For multi-phase samples: If compatible (no multiple phases forming), combine the filtered liquid from Step 8 with the "free liquids" obtained in Step 2g. This combined liquid is defined as the TCLP extract.

c. If the initial liquid phase ("free liquids") is not compatible with the filtered liquid from

Step 8, the liquids may be analyzed separately and the results combined mathematically. See Step 11.

10. The pH of the TCLP extract should be recorded to the nearest 0.01 pH unit. The aliquot used for metals analysis is to be prepared within 4 hours of the completion of filtering. This allows for spiking of an unpreserved subsample of the extract as specified in Method 1311. The remainder of the metals extract must be acidified with HNO_3 to pH <2 by metals prep staff; all other aliquots must be stored at 4°C until analysis.

11. If individual phases are to be analyzed separately, determine the volumes of the individual phases (to ± 0.5 percent), conduct the analyses, and combine the results mathematically:

$$\text{Final Analyte Concentration} = \frac{(V_1)(C_1) + (V_2)(C_2)}{V_1 + V_2}$$

V_1 = The volume of the first phase, in Liters.
C_1 = The concentration of the analyte in the first phase, in mg/L.
V_2 = The volume of the second phase, in Liters.
C_2 = The concentration of the analyte in the second phase, in mg/L.

POLLUTION PREVENTION:

Pollution prevention encompasses any technique that reduces or eliminates the quantity or toxicity of waste at the point of generation. Laboratory staff should order and prepare only those quantities of reagents that will be used prior to the expiration date. Other appropriate measures to minimize waste generation should be brought to the attention of laboratory management. All laboratory waste shall be handled as directed by the Laboratory Waste Management Plan and Hazardous Waste Contingency Plan.

WASTE MANAGEMENT:

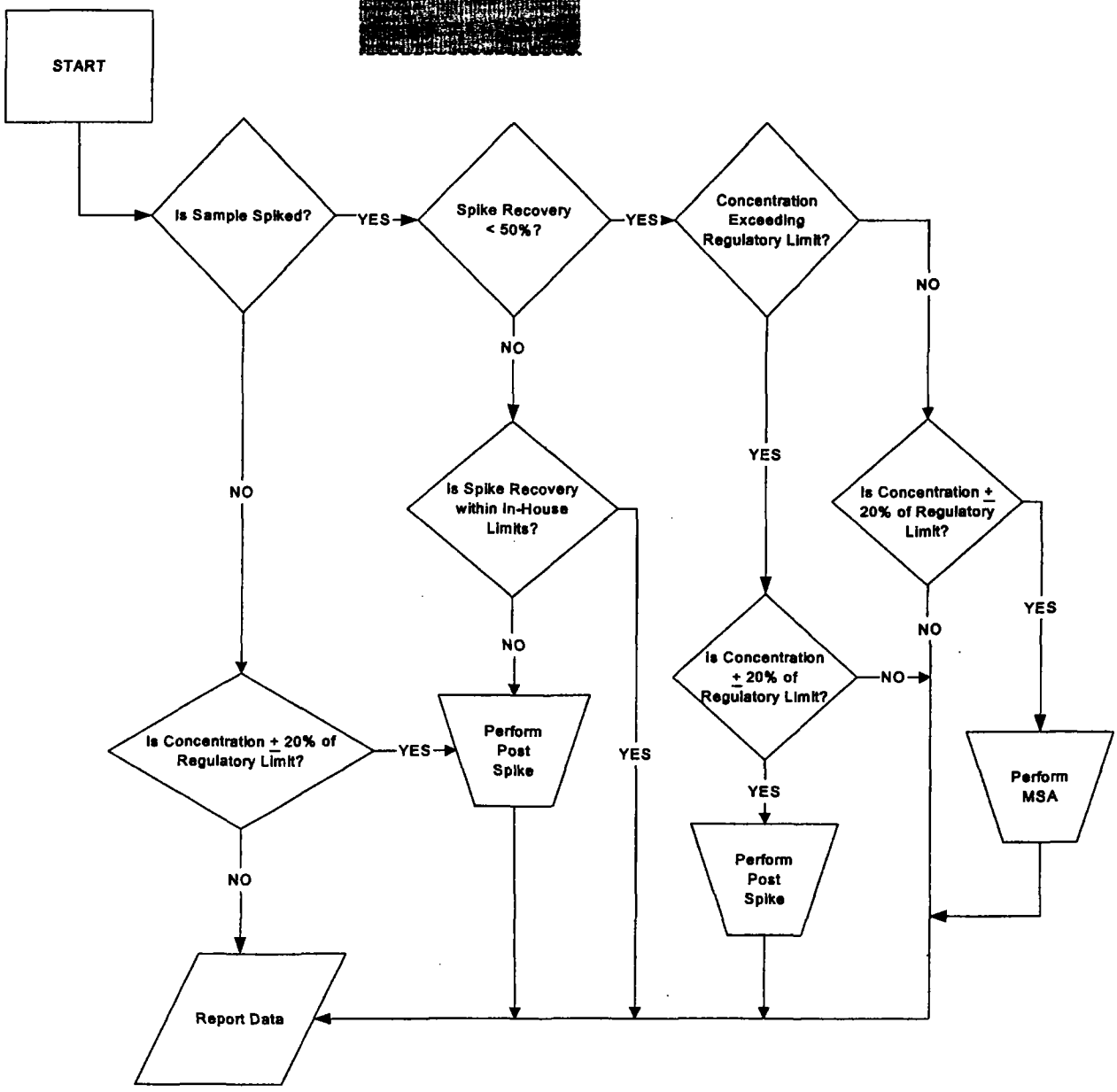
USEPA requires that laboratory waste handling practices conducted be consistent with all applicable rules and regulations. Excess reagents, samples, and method process wastes should be characterized and disposed of in an appropriate manner. Please refer to the EnChem Standard Operating Procedures GEN-5 and GEN-12 for additional information on the disposal of environmental samples and other laboratory wastes.

TABLES: Not Applicable

DIAGRAMS: Not Applicable

FLOWCHARTS:

TCLP Flowchart



APPENDICES:

Appendix 1

EnChem, Inc.

TCLP / SPLP EXTRACTION BENCH SHEET

DATE INITIATED: _____ TIME/TEMPERATURE INITIATED: _____

DATE COMPLETED: _____ TIME/TEMPERATURE COMPLETED: _____

ANALYST INITIALS: _____ REVIEWED BY: _____

REQUIRED ANALYSIS Biota: _____ REVIEWED DATE: _____

Inorganic: _____ SPLP SAMPLE LOCATION: East (1) West (2)

Organic: _____

TCLP Fluid #1 pH: _____ 4.93+/-0.05 Reagents: _____ Fluid #1 Blank ID: _____

TCLP Fluid #2 pH: _____ 2.88+/-0.05 _____ Blank #1 Vessel No.: _____

SPLP Fluid #1 pH: _____ 4.20+/-0.05 _____ Fluid #2 Blank ID: _____

SPLP Fluid #2 pH: _____ 5.00+/-0.05 _____ Blank #2 Vessel No.: _____

I. Physical Characteristics

Sample Number	Sample Description		Multiphase		Particle Size (3)		Particle Size Reduction Technique
	Solid	Liquid	Yes	No	<9.5 mm	>9.5 mm	

(1) For samples collected East of the Mississippi River, use SPLP extraction fluid 1.
(2) For samples collected West of the Mississippi River, use SPLP extraction fluid 2.
(3) If the particle size is > 9.5 mm, prepare sample by crushing, cutting, or grinding.

EnChem, Inc.

TCLP / SPLP EXTRACTION BENCH SHEET

DATE INITIATED: _____ **REVIEWED BY:** _____

DATE COMPLETED: _____ **REVIEWED DATE:** _____

ANALYST INITIALS: _____

II. Determination of Percent Solids

Sample Number	Weight of Filter (g)	Weight of Sample (g)	Weight of Solids (g)	Volume of Filtrate (mL)	Dry Weight of Sample plus Filter (g)	If Multiphase, Are Phases Compatible (1)	Percent Solids (2)	Percent Dry Solids (3)
						Yes / No		
						Yes / No		
						Yes / No		
						Yes / No		
						Yes / No		
						Yes / No		
						Yes / No		
						Yes / No		
						Yes / No		
						Yes / No		
						Yes / No		
						Yes / No		
						Yes / No		
						Yes / No		
						Yes / No		
						Yes / No		
						Yes / No		
						Yes / No		
						Yes / No		
						Yes / No		
						Yes / No		
						Yes / No		

(1) If compatible, combine the filtered liquid resulting from extraction with the initial liquid phase of sample. If the initial liquid phase is not compatible with the filtered liquid resulting from extraction, do not combine. Analyze liquids separately and combine the results mathematically.

(2) Percent Solids = (Weight of Solids / Weight of Sample) * 100 after pressure filtration

(3) Percent Dry Solids = (((Wt. Of Dry Sample + Filter) - Wt. Of Filter) / Weight of Sample) * 100 after pressure filtration and drying.

NOTE: Percent Dry Solids only needs to be determined if Percent Solids is equal to or greater than 0.5% and if a small amount of the filtrate is entrained in the wetting of the filter. If the Percent Solids is less than 0.5%, extraction is not required. The sample is its own extract.

EnChem, Inc.

TCLP / SPLP EXTRACTION BENCH SHEET

DATE INITIATED: _____ REVIEWED BY: _____

DATE COMPLETED: _____ REVIEWED DATE: _____

ANALYST INITIALS: _____

III. pH Determination of the Sample/Extracts and Selection of Extraction Fluid

Sample Number	pH of Sample After 5 Minutes Mixing (1)	pH After Addition of 3.5 mL of 1N HCL and Heating (2)	Extraction Fluid Used		Extraction Vessel No.	Weight of Sample Used for Extraction (g)	Volume of Extraction Fluid Used (mL) (3)	Final pH after Extraction
			1	2				

(1) If the pH is <5.0, use extraction fluid 1

(2) If the pH is >5.0, add 3.5 mL 1N HCl, slurry, cover, heat to 50 C for 10 minutes, cool to room temperature. If pH is <5.0, use extraction fluid 1. If the pH is >5.0 use extraction fluid 2.

(3) Weight of extraction fluid = 20 * percent solids * weight of sample filtered / 100

En Chem, Inc.

SET No: 1

Quality Assurance Document

STANDARD OPERATING PROCEDURE

TITLE: Zero Headspace Extraction for Volatile Compounds

DEPARTMENT: Inorganics - Leach Lab

REFERENCES: EPA Manual SW 846, 3rd Edition (See Application)
ASTM, Vol 11.04 (See Application)

APPLICATION: This method is applicable for samples requiring TCLP (SW 846-1311), ASTM (ASTM D3987-85), and SPLP (SW 846-1312) analysis for volatile compounds.

PROCEDURE SUMMARY:

The Zero Headspace Extraction (ZHE) procedure is designed to simulate the natural leaching process that occurs in the environment in order to determine whether or not a sample is hazardous. The solid and liquid portions of a sample are separated using pressure filtration. D.I. water is added to the solid portion of the sample and tumbled for 18 hours ± 2 hours without headspace. The extract is then collected using pressure filtration. The extract and originally retained liquid portion of the sample are analyzed for volatile organic compounds by GC/MS.

NOTE: This SOP is written primarily for TCLP (SW 846-1311). The only difference for using this procedure for SPLP or ASTM leaching is the buffer solution. SPLP and ASTM uses D.I. water as the leaching buffer.

REVIEWED BY: Jeffrey A. Gordon — Date: 7-22-99
Inorganic Section Supervisor

Gregory J. Graf — Date: 7-23-99
Quality Assurance Officer

APPROVED BY: Glen A. Coder — Date: 7-30-99
Laboratory Manager

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SAMPLE HANDLING AND PRESERVATION:

Samples and extracts should be stored at 4° C with no headspace. Samples for ZHE are not preserved. The extraction must be completed within 14 days of sampling. The analysis (injection date) must be done within 14 days of extraction.

EQUIPMENT AND MATERIALS:

Millipore ZHE vessels
2 teflon cylinder hammers
Tumbler, 30 ± 2 RPM
Balance, capable of weighing to 0.01 g
Beakers
UHP Nitrogen, with a regulator and quick-disconnect fitting
Ziplock bags
500 mL graduated cylinder
40 mL HCL vials
Tygon tubing

REAGENTS:

Deionized (D.I.) water

PROCEDURE:

Cleaning Procedure for ZHE Vessels

See EN CHEM SOP LCH-11

Sample Classification

The TCLP, ASTM, or SPLP waste must be classified as solid, liquid, or multi-phasic. A sample is multi-phasic if there are two distinct layers. The liquid from a multi-phasic sample must be classified as aqueous (ZHE-extract miscible) or non-aqueous (ZHE-extract non-miscible). If the waste arrives at the laboratory for full TCLP, ASTM, or SPLP procedure, the entire classification process will be performed by the inorganic technician in the leach lab. The results will then be supplied to the organic department.

NOTE: If TCLP (SW 846-1311) is requested, refer to : EN CHEM SOP LCH-2.
If SPLP (SW 846-1312) is requested, refer to: EN CHEM SOP LCH-4.
If ASTM (D3987-85) is requested, refer to: EN CHEM SOP LCH-9.

Sample Set-Up

When doing ZHE's three vessels are needed: one for the ZHE blank, one for a transfer vessel, and one for each sample. Every vessel receives a filter except the transfer vessel. All vessels and parts are etched with a number. This makes it easier to identify defective components. A ZHE blank is run every time samples are set up. The vessel in which the blank is run is sequentially rotated, so that all vessels are used for the blank. For every ten samples run through a vessel, a memory blank is run to show that particular vessel is contaminant free.

Blanks should be run on all vessels prior to use and be determined contaminant free. Fill all vessels with DI water. Tumble for 2 hours. Draw off 2 40 mL vials per vessel. Give to organics for analysis.

ZHE Blank Preparation

- When the blank vessel is completely assembled, it needs to be pressurized to eliminate all headspace before transferring the buffer solution. The vessel is connected to nitrogen by using the quick-disconnect Swagelok fitting found at the bottom of the vessel. When the quick-disconnect Swagelok is fitted, a distinct "click" should be heard. The two-way valve on the vessel's top plate is opened, and the vent relief valve on the vessel's bottom plate is closed. Approximately 10-20 psi of pressure is applied to push the piston completely to the top of the vessel, causing the vessel to "jump" slightly. The pressure is released in the vessel by venting the relief valve on the vessel's bottom plate.

- Fill the transfer vessel to its capacity with buffer solution. The vessel's top plate is put on with the two-way valve opened. All the pressure release valves should be closed at this point. The transfer line is then attached tightly to the transfer vessel, and connected to the nitrogen tank. Pressure is applied, being careful not to exceed 50 psi. (Pressure greater than 50 psi may cause the glass fiber filter to break).

- Slowly open the two-way valve, allowing the buffer solution to flow through the transfer line, thus eliminating the air that is in the line. Close the valve when the solution has reached the end of the transfer line, and lightly connect it to the blank vessel. Open both valves (two-way and vent relief) on the blank vessel, and open the two-way valve on the transfer vessel. Tighten the transfer line's connection to the blank vessel once you see the solution begin to leak. This prevents the addition of headspace to the blank vessel.

- When there is no more flow through the transfer line, close the two-way valve on the blank vessel. Release the pressure on the transfer vessel. Pressurize to 40 psi and invert the blank vessel three times. Open the two-way valve slowly to allow any headspace to escape. As soon as liquid begins to elute without bubbles, close the top valve and shut off the nitrogen.

Note: When preparing actual samples, always vent into a hood. At a pressure of 40 psi, all samples and the blank are placed on the tumbler and rotated for 18 hours (± 2 hours) at 30 ± 2 RPM. Record leaching begin and end times in the ZHE logbook. (See Attachment: ZHE Leaching Benchsheet.)

Solid Wastes

- The sample is inspected to see if particle-size reduction is required. The purpose of particle-size reduction is to maximize the total surface area of the sample.

  Note: The combined surface area of many small particles is greater than the surface area of one large particle. Particle-size reduction is necessary if the solid has a total surface area - per gram of material - less than 3.1 square centimeters, or if the sample has particles larger than 1 cm at their narrowest dimension. In other words, the sample should be capable of passing through a 9.5 mm sieve. If necessary, stainless steel trays are chilled along with mallets or similar equipment, and chilled samples (4°C) are broken up with this equipment until the particle size specifications are met.

- If there is no liquid in the sample, the sample is said to contain 100% solids, by definition. The 100% solids sample requires 500 grams of buffer solution. (Since the density of the buffer solution is very near 1 gram/mL, it is assumed that 500 grams of buffer solution is equivalent to 500 mL of buffer solution.)

- The sample is weighed out (after particle size reduction, if necessary) to 25 ± 0.5 grams, and is added to an empty ZHE vessel. The weight of the sample and the vessel number is recorded in the ZHE logbook. The top plate of the vessel is closed to an even seal, and the same procedure used for the blank is followed.

Liquid Wastes

- For liquid samples, weigh the glass fiber filter, and record the weight in the ZHE logbook before the ZHE vessel is assembled. Pour the sample into the vessel and close the top plate tightly. Attach a Luer fitting to the two-way valve on the top plate. Connect a small length of tygon tubing to the luer fitting.

- Pressurize the vessel to 10-20 psi and allow the sample to filter through. Collect the sample in a 40 mL HCL vial. Slowly increase the pressure, by 10 psi increments, up to 50 psi, and hold for 2 minutes. Allow the fluid to collect between pressure increases.

- After filtration: Very carefully remove the filter so as not to tear it! Dry the filter in an oven at 80-120° C for 24 hours. When the weight does not vary by more than one percent over a one hour period, record the last value obtained. Use it to determine the percent dry solids. If the percent dry solids is <0.5%, the fluid eluted into the Tedlar bag is the ZHE extract and no buffer solution is required.

Note: A sample containing particulate matter that immediately clogs the filter during the filtration process, while obviously mostly liquid, is considered to be 100% solids.

Multi-phasic Liquids or Solids Saturated With Liquid

- According to SW846 Method 5030 (7.3.3.2.1): "The sample (for volatile organics) consists of the entire contents of the sample container. Do not discard any supernatant liquids. Mix the contents of the sample container with a narrow metal spatula."

- Place 25 ± 0.5 grams of sample into a ZHE vessel. Pressurize the vessel 10-20 psi. Any liquid that elutes is collected into a tared beaker. Slowly increase the pressure, by 10 psi increments, up to 50 psi. Allow the fluid to collect into the beaker between pressure increases. After two minutes at 50 psi, weigh the beaker and record the weight in the ZHE logbook.

- For multi-phasic wastes, the fluid collected in the beaker is weighed and subtracted from the total sample weight to determine the percent solids.

$$\frac{W_s - W_f}{W_s} \times 100 = \% \text{ Solids}$$

Where:

W_s = Weight of the sample
W_f = Weight of the eluted fluid

(If no liquid elutes out of the vessel, the sample is considered to be 100% solids. The set-up can be used--as it is--for the ZHE extraction.)

- After collecting the fluid portion of the sample, set up the ZHE apparatus - a second time -with a sample size computed according to the following formula:

$$\frac{25}{\% \text{ Solids}} \times 100 = \text{Weight of Sample to be extracted (in grams)}$$

(For example, if the sample weight equals 25.0 g, with 5.0 g of fluid eluted, the % solids equal 80%. The amount of sample to be extracted would be 31.2 g.)

- Collect the fluid via pressure filtration, as described above, into a tared Tedlar bag. Record the weight of the fluid in the ZHE logbook. Store the fluid 4°C until analysis. Add the buffer solution to the vessel after the fluid has been eluted. Calculate the amount of buffer solution needed is according to the following formula:

$$\frac{20 \times \% \text{ Solids} \times \text{Weight of Sample}}{100} = \text{Weight of buffer solution}$$

(For example, if 31.2 g of sample was added to the vessel, with a % solids value of 80%, the weight of buffer solution needed would be 499 g. Again, since the density of the buffer solution is very near 1 gram/mL, it is assumed that 499 grams of buffer solution is equivalent to 499 mL of buffer solution.)

- Add the buffer solution to the ZHE sample vessel via the transfer vessel according to the procedure described in the ZHE blank section. The sample vessel is now ready to be tumbled.

Note: The total number of vessels per tumbler is four. Assemble empty (dummy vessels) to fill any vacant spots in the tumbler. Remove the legs from the vessels, if necessary, before tumbling. If the tumbler makes any bumping or banging noises when turned on, stop the tumbling and re-adjust the vessels.

EXTRACTION, FILTRATION, AND COLLECTION

- After the samples have tumbled for 18 ± 2 hours, stop the tumbler and enter the leaching end time in the ZHE logbook. The legs are re-attached to the ZHE vessels if necessary. Attach Luer fittings to each vessel, and place a tygon tube on each Luer fitting.

- Connect each vessel to the nitrogen tank and apply 20-30 psi of pressure. Open the two-way valve on the top plate of the vessel slowly to release some of the extract through the tubing. When it has reached the end of the tubing, close the valve. Slowly re-open the two-way valve on the vessel allowing the extract to flow into the 40 mL vial. Fill two vials per sample.

- Add pressure slowly when necessary. There should be no gurgling noises, indicating an influx of air, as volatiles may be lost and the sample would then be invalid. (Also, if the filter in the vessel breaks open, the sample becomes invalid. If either of these occur, the entire extraction must be repeated.

- The liquid obtained prior to extraction (from a multi-phasic sample or solid sample saturated with liquid) must either:

  1) If miscible, be combined with the extract and analyzed as one, or

  2) If non-miscible, be analyzed separately from the extract, mathematically combining the results using the following volume-weighted average formula:

$$\frac{V_1 C_1 + V_2 C_2}{V_1 + V_2} = \text{Combined result}$$

Where:
V_1 = The volume of the liquid phase.
V_2 = The volume of the ZHE extract.
C_1 = The concentration of the analyte in the liquid phase.
C_2 = The concentration of the analyte in the ZHE extract.

En Chem, Inc.

Quality Assurance Document

ZHE EXTRACTION LOG TCLP or SPLP	
Project Number:	Date: Initials
ZHE Vessel No.	ZHE Vessel No.
Sample No:	Sample No:
Weight Of Sample: ____ Multiphase ____ MS Spike completed	Weight Of Sample: ____ Multiphase ____ MS Spike completed
ZHE Vessel No.	ZHE Vessel No.
Sample No:	Sample No:
Weight Of Sample: ____ Multiphase ____ MS Spike completed	Weight Of Sample: ____ Multiphase ____ MS Spike completed
ZHE Vessel No.	ZHE Vessel No.
Sample No:	Sample No:
Weight Of Sample: ____ Multiphase ____ MS Spike completed	Weight Of Sample: ____ Multiphase ____ MS Spike completed
ZHE Vessel No.	ZHE Vessel No.
Sample No:	Sample No:
Weight Of Sample: ____ Multiphase ____ MS Spike completed	Weight Of Sample: ____ Multiphase ____ MS Spike completed
ZHE Vessel No.	ZHE Vessel No.
Sample No:	Sample No:
Weight Of Sample: ____ Multiphase ____ MS Spike completed	Weight Of Sample: ____ Multiphase ____ MS Spike completed
ZHE Vessel No.	ZHE Vessel No.
Sample No:	Sample No:
Weight Of Sample: ____ Multiphase ____ MS Spike completed	Weight Of Sample: ____ Multiphase ____ MS Spike completed
ZHE Vessel No.	ZHE Vessel No.
Sample No:	Sample No:
Weight Of Sample: ____ Multiphase ____ MS Spike completed	Weight Of Sample: ____ Multiphase ____ MS Spike completed
ZHE Vessel No.	ZHE Vessel No.
Sample No:	Sample No:
Weight Of Sample: ____ Multiphase ____ MS Spike completed	Weight Of Sample: ____ Multiphase ____ MS Spike completed
ZHE Vessel No.	ZHE Vessel No.
Sample No:	Sample No:
Weight Of Sample: ____ Multiphase ____ MS Spike completed	Weight Of Sample: ____ Multiphase ____ MS Spike completed
ZHE Blank Vessel No:	ZHE Blank ID:
Leach Time Initiation:	Leach Time Completion:

Standard Operating Procedure

TITLE: Volatile Organic Preparation of Solid Matrices by Methods 5035/8260B

DEPARTMENT: Volatile Organics Laboratory

APPLICATION: The method outlined within is used for the detection of volatile organic compounds by lab prep method 5035 and GC/MS 8260B in solid matrices. See the accompanying Target Compound List (TCL) for the analytes to which this method applies.

REFERENCES: 40 CFR, Part 136, Appendix B, Revision 1.11

EPA SW-846, Methods 8260B, December 1996

EPA SW-846, Methods 5035, December 1996

PROCEDURE SUMMARY:

Volatile compounds are purged out of soil matrices using a steady stream of helium. The compounds are trapped on a tenax/silica gel/charcoal trap, which is then heated rapidly, desorbing the compounds onto a GC/MS system. Separation occurs on a capillary column before entering the mass spectrometer, where spectra are generated that can be compared with previously prepared standard spectra. Quantitation is based upon analyte response versus standard response relative to Internal Standards.

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Jeffrey J. Buchner
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M. Suha — Date: 05/11/2000
Michael C. Suha
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APPROVED BY: Nils K. Melberg — Date: 5/15/2000
Nils K. Melberg
Laboratory Manager

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SAMPLE HANDLING AND PRESERVATION:

Soil samples received in Encore samplers must be extruded within 48 hours of collection. If analyzed within the 48 hour time frame, no acid preservation is required. Sodium Bisulfate must be used as a preservative if the samples do not effervescesce and are to be analyzed more than 48 hours from the time of collection or if an Encore sampler is not used in the field.

All samples must be analyzed within 14 days of collection.

INTERFERENCES:

Interferences usually fall under two categories:

1. Masking:
   This occurs when a compound is present at such a high concentration that its peak is broad enough to cover peaks with similar retention times.

2. Matrix Dampening:
   This occurs when the sample, usually a soil, contains substances that impede the purging of volatiles out of the sample. This is indicated by low internal standard areas and/or poor surrogate recovery.

I. APPARATUS AND MATERIALS:

HP 5972 MSD with capillary
column inlet/adapter

Tekmar 3000 Purge and Trap Concentrator

Archon 5100A or Dynatech PTA-30 autosampler

Capillary Gas Chromatography column - 75m x 0.53mm ID DB-624
megabore(J&W Scientific, Inc.) with a 3 µm film thickness; or,
20m X 0.18mm ID DB-624, J&W Scientific, Inc.,with a 1.0 um film thickness.

Syringes: 5 and 50 ml Hamilton gastight with Luer-lock tip; 10, 25, 50,100, 500, 1000 µl gastight microsyringes.

Standard solution storage containers - 1.5 ml, 7 ml, 14 ml amber vials with PTFE-lined screw caps

Disposable magnetic stirring bars - Fischer brand or equivalent.

40-mL glass VOA vials with screw caps

and PTFE lined septum.

Balance - capable of weighing to 0. 1g

REAGENTS:

Gases: Helium, UHP grade

Methanol, Burdick & Jackson Purge-and-Trap Grade or equivalent

Organic Free Water (OFW): Organic Free Water is water with organics present below MDL for most compounds.

Standards:

Primary Standard Sources:
Restek VOC Mixes (Custom and standard stock mixes)
.

Secondary Sources:

Restek VOC Mixes (Custom and standard stock mixes of a different lot #)
Restek Internal Std. Mix.
Restek Surrogate Std. Mix.
Restek 4-Bromofluorobenzene Solution.

Preservative: Sodium Bisulfate Solution, NaHSO4-ACS reagent grade or equivalent at a concentration of 200g/L of OFW.

Ottawa Sand

PROCEDURE:

II. Initial Calibration

Demonstration and documentation of an acceptable initial calibration as defined in SW846-8260B is required before any samples are analyzed, and is required intermittently throughout sample analysis as dictated by results of a continuing calibration check standard.

A. Verification of Instrument Performance

A 50 ng BFB injection must meet spectral criteria as defined SW846-8260B. If the tune spectrum does not meet the criteria after several injections, then the mass spec will need to be retuned with PFTBA calibration gas to update spectra.

B. Preparation of Standards

Stock Reference Standards:

Stock standards may be prepared from neat standards and/or prepared mixes can be purchased. See Appendix B for the Target Compound List. A reference standard prepared from a neat standard is made up at a 10,000 ug/mL concentration as follows:

Place about 9.8 ml of methanol into a 10 ml ground-glass stoppered volumetric flask. Allow the flask to stand unstoppered for a few minutes until all alcohol-wetted surfaces have dried and weigh to the nearest 0.1 mg. Using a 100 µl gastight syringe, add about 20-25 drops of the neat standard to the flask to obtain about 100 mg of standard. Let the drops fall just above the surface of the methanol but do not let the syringe needle touch the sides of the flask neck. Reweigh the flask, dilute to volume, stopper, then mix by inverting the flask several times. Store without headspace in a 7 mL amber vial in a freezer. All data regarding standard preparation must be entered into the standard preparation logbook. A number is assigned to each prepared standard solution and that number is entered on all logs where a solution is used. The vial containing a prepared standard solution must be labeled in accordance with the En Chem SOP, and include the assigned log number. A brief description of the standard must also appear on the label.

Working Standards:

Prepare working standards of both the volatile target compounds at 100 ug/mL in P&T methanol. Prepare an Internal Standard, Surrogates, and an Internal Standard/Surrogate solution (IS/SS) at a concentration of 250 ug/mL in P&T methanol. Record all data regarding preparation of each standard solution in the standard logbook as described above for stock standards. Label the working standard vials as described above for the stock standards. All standards should be stored in a freezer. Prepared standard solutions in methanol are stable for about 4 weeks when stored at -15 to -20°C.

Preparation of Calibration Standards:

An initial calibration consisting of at least five calibration points is analyzed before sample analysis may proceed. The standards are prepared with all target compounds and surrogates at equal concentrations. The calibration levels are prepared as follows:

Calibration Level	Amt. of working standards	InternalStandard/ Surrogate Addition	Volumetric/Syringe
2 ug/L	10 uL	4 uL	500 mL
5 ug/L	2.5 uL	1 uL	50 mL
20 ug/L	10 uL	4 uL	50 mL
50 ug/L	25 uL	10 uL	50 mL
100 ug/L	50 uL	20 uL	50 mL
200 ug/L	100 uL	40 uL	50 mL

The associated volumetric/syringe is inverted three times and the 5 mL aliquot is placed into a non-preserved 40 mL VOA vial containing a magnetic stir bar for analysis. The autosampler adds 5 mL of OFW into the 40 mL VOA vial and 1 uL of Internal Standard mix at 250 ug/mL. The calibration curve and associated standards, blanks, samples and QC for low level soil analysis are purged at 40° C.

Preparation of Calibration Method:

Identify the compounds using reference spectra and retention time data. Assign response factors to each compound using the internal standard technique based on its response in each standard. Refer to the formula sheet for the response factor formula in Appendix A, #1. See Appendix B for the assigned internal standard for each analyte.

Check linearity of response by calculating the percent relative standard deviation (RSD) of the response factors for each compound. The %RSD for each individual Calibration Check Compound (CCC) must be less than or equal to 30.0%. In addition, the minimum acceptable average response factor for System Performance Check Compounds (SPCC) must meet specified Response Factor (RF) criteria. See Appendix C, Table 2 for lists of CCC and SPCC compounds. The validity of the five point calibration is accepted or rejected after making these comparisons. Some common causes of a bad calibration curve are: standard degradation, poor instrument stability or improper purge flow rate.

If the five point calibration is acceptable, a calibration file is constructed that matches the ID file and stores each of the five response factors, the average RF, the %RSD, and concentrations of each target compound and surrogate.

If the five point calibration is not acceptable, the appropriate calibration solution(s) is reanalyzed to obtain acceptable %RSD and RFs over the entire calibration range.

III. Daily Checks and Continuing Calibration Check

A BFB tune, continuing calibration check standard, method blank and LCS/LCSD must be performed at the beginning of each 12 hour period during which sample analyses are performed.

A. Daily BFB Tune

The BFB tune must meet the spectrum criteria as defined in Appendix C, Table 1, before proceeding with the continuing calibration check standard. If the BFB or autotune fails repeatedly, source cleaning may be necessary.

B. Continuing Calibration Check Standard

Prepare a 50 µg/L standard by adding 25 uL of the target compound working standard at 100 ug/mL into a 50 mL syringe of organic free water. Place the 5 mL aliquot into a non-preserved 40 mL VOA vial containing a disposable magnetic stir bar. Place the vial into the autosampler where 5 mLs of OFW and 1 uL of IS/SS (Internal Standard/Surrogate) mix at 250 ug/mL will be added.

Compare the daily response factors with the average response factors from the initial calibration. The relative percent difference for the CCC's must not be greater than 20.0%. The daily response factor of any SPCC compound must be within the criteria as listed in Appendix C, Table 2. The ISTD areas must be -50% to +100% of the initial calibration mid point standard. Check the validity of the ID file's qualitative matches. If the SPCC or CCC conditions are not met, the standard is not valid and should be repeated. Repeated failure to meet acceptance criteria is a sign that the five-point calibration curve is no longer valid. If no extraneous causes of standard failure are suspected, the initial calibration must be repeated. Refer to IIB above.

C. Method Blank

A method blank is prepared with 5 mL of OFW into a 40 mL VOA vial containing a disposable magnetic stir bar. The vial is placed onto the autosampler and 5 mL of OFW and 1 uL of IS/SS mix @ 250 ug/mL is added. The blank is preheated to 40°C and purged. The method blank must be analyzed under the same criteria as the samples.

In addition to the internal standard and surrogate QC requirements, the method blank must also meet the following criteria before sample analysis may begin:

1. No common solvent (Acetone, 2-Butanone, 2-Hexanone, 4-Methyl-2-pentanone and Methylene Chloride) may be present above 5 times the EQL (Estimated Quanitation Limit).

2. Target compounds other than common solvents must not be greater than the r eporting limit.

3. Non-target compound peaks with areas greater than 10% of the area of the nearest internal standard, other than surrogates, must not be present in the chromatogram if tentatively identified compounds (TICS) are requested.

If contaminants are found in the blank, an attempt must be made to identify and minimize/eliminate the source.

D. Lab Control Sample/Lab Control Sample Duplicate

A lab control sample(LCS)/lab control sample duplicate(LCSD) is performed within every 12 hr. analytical clock. Prepare a LCS @ 50 ug/L by spiking 2.5 uL of the second source spike calibration mix at 100 ug/mL through the septa of a non-preserved 40mL VOA vial containing 5g of Ottawa sand, 5mL of OFW and a disposable magnetic stir bar..Repeat this procedure for the LCSD. Insert the vials onto the autosampler where 5 mL of OFW and 1 uL of ISSTD mix @ 250 ug/mL will be added. The LCS/LCSD is preheated to 40°C and purged.

The LCS/LCSD will be used for QC control if Matrix Spike/Matrix Spike Duplicates are not analyzed because of insufficient sample amounts. Due to poor purge efficiencies of some compounds found in the spike mixes, a default percent recovery criteria of 50 to 150 has been established.

IV. Sample Analysis

1. Low Level Soils

A. Sample Prep

Before any En Cores are extruded a test for effervescence will be done using soil from the supplied total solids container. Place 5 mLs of 200g/L Sodium Bisulfate solution into a disposable beaker. Scoop out of the total solids container approximately 5g of the sample. Check for effervescence. Drop into the sample a pH strip. Record the pH in the preservation log book. If the pH is>2 an extra gram of solid Sodium Bisulfate must be added to this sample at the time of preservation. If effervescence occurs skip to section A2. If no effervescence occurs both 5g En Cores are extruded as follows:

A1-Place 5 mLs of 200g/L Sodium Bisulfate solution into a 40 mL VOA vial containing a disposable magnetic stir bar and, if necessary, the extra 1g of Sodium Bisulfate*. Put the vial on an analytical balance capable of weighing to 0.01g and zero the weight. Extrude the sample from the Encore(s) into the vial and re-cap the vial(s). Re-weigh the vial(s) in order to obtain the sample weight. Record the weight of the sample in a logbook and also record the weight of the sample on the vial. Dispose of the Encore(s) properly. A2-If effervescence is observed:

Place 5 mLs of OFW into a 40 mL VOA vial containing a disposable magnetic stir bar. Put the vial on an analytical balance capable of weighing to 0.01g and zero the weight. Extrude the sample from the Encore(s) into the vial and re-cap the vial(s). Re-weigh the vial(s) in order to obtain the sample weight. Record the weight of the sample in a logbook and also record the weight of the sample on the vial. Dispose of the Encore(s) properly. .

Samples received pre-weighed in the field must be in 40 mL VOA vials and contain a magnetic stir bar, acid preservative and a field tare weight. The analyst will compare the field tare weight to the weight of the sample before analysis. The weight of the sample should be recorded.

* It is important to note that all samples must be extruded from the Encore sampler within 48 hrs. of collection. If the samples are to also be analyzed within the 48 hr. criteria, no acid preservation is required. If analysis is to occur after 48 hrs. but within 14 days, the samples must be preserved with Sodium Bisulfate. The ratio of Sodium Bisulfate to sample weight is 0.2g of preservative to 1g of sample. [1]

B. Analysis

Before any sample analysis may begin, all instrument criteria, as specified by SW-846 8260B, must be met. Place the already weighed 40 mL VOA vial containing the sample, stir bar and 5 mL of OFW or Sodium Bisulfate solution onto the autosampler where another 5 mLs of OFW and 1 uL of IS/SS @ 250 ug/mL will be added. The sample is preheated to 40°C and purged. The stir bar is moving continuously during the purge cycle. This also helps in compound recovery by breaking down any clumps that may remain in the sample.

Identify and quantitate the target compounds using relative retention times (RRT) and average response factors from the 5-point calibration curve. For establishing correspondence of the RRT, the sample component RRT must compare within +/- 0.06 RRT units of the RRT of the continuing calibration standard component. Compare computer-matched compounds with reference spectra to accept or reject each identification. All ions present in the reference spectrum that are at least 10% of the base peak must be present in the sample background-subtracted spectrum. Also, the relative intensities of these ions must agree within +/-30% between the standard and sample spectra. While this is a good guideline, acceptance or rejection will depend upon the judgment of the analyst.

It will be necessary to adjust the sample weight for quantitation purposes. Any analyte hits outside of the calibration range, 200 ug/kg, will be extracted into Methanol and analyzed under methods 5030B and 8260B water criteria**.

** If samples are screened and pre-weighed in the field or other analytical facilities, sample aliquots of 1g may be analyzed following the low level guidelines if received in 40 mL VOA vials with magnetic stir bars and preserved in Sodium Bisulfate.

2. Medium Level Soils

A. Sample Prep

A 25g Encore sampler is extruded into 20 mLs of Purge and Trap Methanol in a 60 mL soil jar. The tare weight of the jar and the approximate weight of 20 mLs of MEOH (15.8g) is

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[1] Hold Presevation time may be adjusted as new data becomes available.

added and recorded. The jar containing the 25g plug then placed on the balance and the tare weight recorded. The final sample weight is found by subtracting the weight of the jar/MEOH from the weight of the jar/MEOH/sample. Methanol is then added to bring the final ratio of soil to MEOH to 1:1. The amount of surrogate @ 2500 ug/mL to be added is equal in uLto the final weight/volume of the sample/MEOH.

A 5g Encore may also be used in place of a 25g Encore sampler. It will follow the same procedure as mentioned above except the sample will be extruded into a 40 ml VOA vial containing 5 mLs of P&T MEOH. The most important aspect is to maintain a 1:1 ratio of soil to methanol.

Samples received pre-weighed in the field should also have a 5g or 25g sample aliquot already preserved in MEOH. The same procedure for medium level preparation, as listed above, is followed.

QC samples are prepped per 20 samples. A MEOH blank is made by adding 10ml of P & T methanol into 2oz jar containing 10g Ottawa Sand. In order to obtain a final concentration of 50 ug/L for surrogates, 10 uL of surrogate mix @ 2500 ug/mL is spiked into the VOA vial. The same process is used for Lab Control Sample/Lab Control Sample Duplicate except 50 uL of MS mix #1 and 50uL of MS mix#2 of a second source spike mix @ 500 ug/mL is also added. The LCS/LCSD is used when MS/MSD samples are not available due to insufficient sample amounts.

B. Analysis

Samples that have been medium level extracted must be run under 5030B criteria. A 1 mL aliquot of the methanol extract is added to 49mLs of OFW in a 50mL syringe or volumetric. The syringe/volumetric is inverted three times and the sample poured into a 40 mL non-preserved VOA vial. Once the vial is placed onto the autosampler, 1 uL of ISTD mix @ 250 ug/mL is added to a 5 mL aliquot of sample and purged.

Refer to section IV.B for the identification and quantitation of analytes detected within the analysis.

Quality Control:

The surrogate recoveries and internal standard areas must be checked to determine that they are within the established control limits. If the requirements are not met, the sample is reanalyzed. The internal standard areas for the method blank and samples analyzed must be within -50% to +100% of the calibration check standard's internal standard areas for compounds of concern. If an internal standard is outside of control limits but no compounds of interest are being quantified, no re-analysis is necessary. If the second analysis is within control limits, the data from that analysis is reported. If the failure is repeated, data from the first analysis is reported and the anomaly is discussed in a sample narrative.

A sample matrix spike (MS) and matrix spike duplicate (MSD) must be performed for every 20 samples per instrument of the same matrix. A LCS/LCSD or BS/BSD will be performed if insufficient sample is received for MS/MSD purposes. A matrix type for an MS/MSD is defined either as a heated low level soil or an unheated medium level soil extract. The spiking solution must contain all constituents of concern and be from a secondary source other than the reference standards used in the calibration. Because Method 5035 does not allow for the preserved soil vial to be opened after preservation, the analyst will use a 10ul syringe and inject 2.5ul of 100 ppm matrix spiking solution through the septa of the 40ml VOA vial prior to analysis. The percent recoveries for each spiked compound must be checked to determine that they are within the established control limits.

E. Annual Quality Control

1. Method Detection Limits (MDLs):

MDLs are determined as specified in 40 CFR, Part 136, Appendix B or SW846 Chapter One and must be performed once a year per instrument for all target compounds of interest. At least seven replicates will be analyzed per set. Each analysis will be prepared at a concentration of 1.0 µg/L for all target compounds and spiked with 50 µg/L of the IS/SS.

2. Surrogate Recovery Limits:

One set each of surrogate recovery limits for heated soils and unheated medium level soil extracts must be established for all instruments performing GC/MS analysis. These limits are to be updated annually using data from all GC/MS instruments.

3. Matrix Spike and Duplicate (MS/MSD) Recovery Limits:

One set each of MS/MSD recovery limits for heated soils and unheated medium level soil extracts must be established for all instruments performing GC/MS analysis. These limits are to be updated annually using data from all GC/MS instruments.

APPENDIX A

FORMULA SHEET

#1 RESPONSE FACTOR:

$$RF = \text{RESPONSE FACTOR} = \frac{(Ax)}{(Ais)} \times \frac{(Cis)}{(Cx)}$$

where:

Ax = Area of analyte's characteristic ion of quantitation

Ais = Area of corresponding internal standard's ion of quantitation

Cis = Concentration of corresponding internal standard

Cx = Concentration of analyte to be measured

APPENDIX B

TARGET COMPOUND LIST, INTERNAL STANDARDS, SURROGATES, AND ESTIMATED QUANTITATION LIMITS (EQL's) FOR LOW LEVEL AND MEDIUM LEVEL SOIL MATRICES

	LOW LEVEL EQL*	MEDIUM LEVEL EQL
INTERNAL STANDARD: PENTAFLUOROBENZENE		
Dichlorodifluoromethane	5	25
Chloromethane	5	25
Bromomethane	5	25
Vinyl chloride	5	25
Chloroethane	5	25
Trichlorofluoromethane	5	25
Dichlorofluoromethane	5	25
Allyl Chloride	5	125
Acrolein	5	125
Acetone	5	125
Diethyl Ether	5	25
1,1-Dichloroethene	5	25
Iodomethane	5	125
Acrylonitrile	5	125
Methylene chloride	5	25
Carbon disulfide	5	25
Trans-1,2-Dichloroethene	5	25
Methyl Tert Butyl Ether	5	25
1,1-Dichloroethane	5	25
Vinyl Acetate	5	125
2-Butanone	5	125
Diisopropyl Ether	5	25
Cis-1,2-Dichloroethene	5	25
Bromochloromethane	5	25
Chloroform	5	25
2,2-Dichloropropane	5	25
Tetrahydrofuran	5	125
1,2-Dichloroethane	5	25
1,1,1-Trichloroethane	5	25
1,1-Dichloropropene	5	25
Carbon tetrachloride	5	25
Benzene	5	25
125		
Dibromofluoromethane (SURR)		

* Lower quantitation limits may be obtained depending on project requirements
Units: ug/kg

APPENDIX B - Continued

TARGET COMPOUND LIST, INTERNAL STANDARDS, SURROGATES, AND ESTIMATED QUANTITATION LIMITS (EQL's) FOR LOW LEVEL AND MEDIUM LEVEL SOIL MATRICES

	LOW LEVEL EQL	MEDIUM LEVEL EQL
INTERNAL STANDARD: 1,4-DIFLUOROBENZENE		
Diethoxymethane	5	125
Trichloroethene	5	25
1,2-Dichloropropane	5	25
Dibromomethane	5	25
Bromodichloromethane	5	25
2-chloroethyl vinyl ether	5	150
cis-1,3-Dichloropropene	5	25
4-Methyl-2-pentanone	5	125
25		
INTERNAL STANDARD: CHLOROBENZENE-d5		
Toluene	5	25
trans-1,3-Dichloropropene	5	25
1,1,2-Trichloroethane	5	25
Tetrachloroethene	5	25
1,3-Dichloropropane	5	25
Dibromochloromethane	5	25
2-Hexanone	5	125
1,2-Dibromoethane	5	25
Toluene-d8 (SURR)		
Chlorobenzene	5	25
1,1,1,2-Tetrachloroethane	5	25
Ethylbenzene	5	25
m-, p-, Xylene	10	25
o-Xylene	5	25
Styrene	5	25
Bromoform	5	25
Isopropylbenzene	5	25
Trans-1,4-Dichloro-2-Butene	5	150
Cis-1,4-Dichloro-2-Butene	5	150
4-Bromofluorobenzene (SURR)		

APPENDIX B - Continued

TARGET COMPOUND LIST, INTERNAL STANDARDS, SURROGATES, AND ESTIMATED QUANTITATION LIMITS (EQL's) FOR LOW LEVEL AND MEDIUM LEVEL SOIL MATRICES

	LOW LEVEL EQL*	MEDIUM LEVEL EQL
INTERNAL STANDARD: 1,4-Dichlorobenzene		
Bromobenzene	5	25
1,1,2,2-Tetrachloroethane	5	25
1,2,3-Trichloropropane	5	25
n-Propylbenzene	5	25
2-Chlorotoluene	5	25
4-Chlorotoluene	5	25
1,3,5-Trimethylbenzene	5	25
Tert-Butylbenzene	5	25
1,2,4-Trimethylbenzene	5	25
Sec-Butlybenzene	5	25
1,3-Dichlorobenzene	5	25
1,4-Dichlorobenzene	5	25
P-Isopropyltoluene	5	25
1,2-Dichlorobenzene	5	25
N-Butylbenzene	5	25
1,2-Dibromo-3-chloropropane	5	25
1,2,4-Trichlorobenzene	5	25
Naphthalene	5	25
Hexachlorobutadiene	5	25
1,2,3-Trichlorobenzene	5	25
Hexachloroethane	5	25
2-Methylnaphthalene	5	25

APPENDIX C

TABLE 1

GC/MS PERFORMANCE STANDARD

Bromofluorobenzene (BFB)

Ion Abundance m/z	Criteria
50	15-40% of mass 95
75	30-60% of mass 95
95	Base peak, 100% relative abundance
96	5-9% of mass 95
173	Less than 2% of mass 174
174	Greater than 50% of mass 95
175	5-9% of mass 174
176	95-101% of mass 174
177	5-9% of mass 176

TABLE 2

Calibration Check Compounds (CCC)

1,1-Dichloroethene
Chloroform
1,2-Dichloropropane
Toluene
Ethylbenzene
Vinyl Chloride

* The % RSD for the initial calibration must be less than 30%.
* The % RSD for the continuing calibration must be less than 20%.

System Performance Check Compounds (SPCC)

	Minimum RRF
Chloromethane	0.10
1,1-Dichloroethane	0.10
Bromoform	0.10
1,1,2,2-Tetrachloroethane	0.30
Chlorobenzene	0.30

Standard Operating Procedure

TITLE: Volatile Organic Preparation of Aqueous Matrices and High Concentration Soils by Method 5030B

DEPARTMENT: Volatile Organic Laboratory

APPLICATION: The method outlined within is used for the detection of volatile organic compounds by lab prep method 5030 in solid matrices.

REFERENCES: EPA SW-846 5030B
Revision, December 1996

PROCEDURE SUMMARY:

Volatile compounds are purged out of aqueous samples using a steady stream of helium. The compounds are trapped on a tenax/silica gel/charcoal trap, which is then heated rapidly, desorbing the spectrometer, where spectra are generated that can be compared with previously prepared standard spectra. Quantitation is based upon analyte response verses standard response relative to Internal Standards.

REVIEWED BY: _____ 5/11/2000
Jeffrey J. Bussiner — Date
Volatile Organics Group Leader

_____ 05/11/2000
Michael C. Suha — Date
Quality Assurance Officer

APPROVED BY: _____ 5/15/2000
Nils K. Melberg — Date
Laboratory Manager

F:\SOP\3\VOA\G3-VOA-3.DOC

1.1 This method describes a purge-and-trap procedure for the analysis of volatile organic compounds (VOCs) in aqueous samples and water miscible liquid samples. It also describes the analysis of high concentration soil and waste sample extracts prepared in Method 5030.

1.2 Method 5030 can be used for most volatile organic compounds that have boiling points below 200°C and are insoluble or slightly soluble in water. Bolatile water-soluble compounds can be included in this analytical technique; however, quantitation limits (by GC or GC/MS) are approximately ten times higher because of poor purging efficiency. The method is also limited to compounds that elute as sharp peaks from a GC column packed with graphitized carbon lightly coated with a carbowax or a coated capillary column. Such compounds include low molecular weight halogenated hydrocarbons, aromatics, ketones, nitriles, acetates, acrylates, ethers, and sulfides.

1.3 Method 5030 in conjunction with Modified Method 8015 (GC/FID), may be used for the analysis of aliphatic hydrocarbon fraction in the light ends of total petroleum hydrocarbons, e.g., gasoline. For the aromatic fraction (BTEX), use Method 5030 and Method 8021B (GC/PID). A total determinative analysis of gasoline fractions may be obtained using Methods 8021B (GC/PID) in series with Modified Method 8015 where allowable by local regulations.

1.4 Water samples can be analyzed directly for volatile organic compounds by purge-and-trap extraction and gas chromatography. Higher concentrations of these analytes in water can be determined by direct injection of the sample into the chromatographic system or by dilution of the sample prior to the purge-and-trap process.

1.5 High concentrations soil extracts in methanol can also be prepared by this method.

1.6 This method is restricted to use by or under the supervision of trained analysts. Each analyst must demonstrate the ability to generate acceptable results with this method.

2.0 SUMMARY OF METHOD

2.1 Samples: An inert gas is bubbled through a portion of the aqueous sample at ambient temperature, and the volatile components are efficiently transferred from the aqueous phase to the vapor phase. The vapor is swept through a sorbent column is heated and backflushed with inert gas to desorb the components onto a gas chromatographic column.

3.0 INTERFERENCES

3.1 Impurities in the purge gas, and from organic compounds out-gassing from the plumbing ahead of the trap, account for the majority of contamination problems. The analytical system must be demonstrated to be free contamination under the conditions of the analysis by running laboratory reagent blanks. The use of non-polytetrafluoroethylene (non-PTFE) lastic coating, non-PTFE thread sealants, or flow controllers with rubber components in the purging device must be avoided, since such materials out-gas organic compounds which will be concentrated in the trap during the purge operation. These compounds will result in interference's or false positives in the determinative step.

3.2 Samples can be contaminated by diffusion of volatile organics (particularly methylene chloride and fluorocarbons) through the septum seal of the sample vial during shipment and storage. A trip bland prepared from organic-free reagent water and carried through sampling and handling protocols serves as a check on such contamination.

3.3 Contamination by carryover can occur whenever high-concentration and low-concentration samples are analyzed sequentially. whenever an unusually concentrated sample is analyzed, it should be followed by an analysis of organic-free reagent water to check for cross-contamination. The trap and other parts of the system are subject to contamination. Therefore, frequent brake-out and purging of the entire system may be required.

3.4 The laboratory where volatile analysis is performed should be completely free of solvents. Special precautions must be taken to determine methylene chloride. Random background levels can result. Since methylene chloride will permeate through PTFE tubing, all GC carrier gas lines and purge gas plumbing should be constructed of stainless steel or copper tubing. Laboratory workers' clothing previously exposed to methylene chloride fumes during common semivolatile extraction procedures can contribute to sample contamination. The presence of other organic solvents in the laboratory where volatile organics are analyzed will also lead to random background levels and the same precautions are taken. To accomplished this En Chem has two separate facilities to divide the semivolatiles group from the volatile preparation and analysis.

4.0 APPARATUS AND MATERIALS

4.1 Microsyringes - 10-uL, 25-uL, 250-uL, and 1,000-uL.

4.2 Two 50-ml glass syringes with Luer-Lok tip (other sizes are acceptable depending on sample volume used).

4.3 Volumetric flasks; Class A- 10-ml and 100-ml, with ground-glass stoppers.

4.4 Vials - 40-ml, for Purge and Trap autosampler.

4.5 Purge-and-trap device - Tekmar 3000 and Dynatech Archon or PTA30 W/S.

(The purge-and-trap device consists of three separate pieces of equipment: the sample purger, the trap, and the desorber. Several complete devices are commercially available.)

4.5.1 Tekmar, 5 ml fritless sparge tube.

4.5.2 Supelco, VOCARB 3000, conditioned at 270 C for 30 minutes.

These combinations require rapid heating to desorption temperatures of 245°C to 270°C (follow manufacturer's instructions). At these increased temperatures, catalytic and thermal decomposition of analytes has been reported.

<u>NOTE</u>: Even newly constructed traps may have become contaminated prior to their first used from airborne vapors. These highly adsorptive materials must be kept tightly sealed in an area of minimum organic vapor contamination.

4.6 Capillary GC Columns - DB-624 20m x 0.18 mm i.d. x 1.0 um film thickness.

4.6.1 Capillary column directed into ion source.

4.7 Capillary injection port inlet equipped with Electronic Pressure Control (EPC), set at 0.5 ml/min, 34 mls/min split flow at the total flow outlet.

5.0 REAGENTS

5.1 Organic Free Water (OFW): Organic Free Water is water with organics present below MDL for most compounds.

5.2 Standards

Primary Standard Sources:

Restek VOC Mixes (Custom and Standard Stock Mixes)

Secondary Sources:

Restek VOC Mixes (Custom and standard stock mixes of a different lot #)
Restek Internal Std. Mix.
Restek Surrogate Std. Mix.
Restek 4-Bromofluorobenzene Solution

Note: If GC - FID/PID analysis is performed the Standards used are those appropriate for that particular technique.

5.3 Gases: Helium, UHP grade

6.0 SAMPLE COLLECTION, PRESERVATION, AND HANDLING

6.1 Samples should be stored in capped bottles, with minimum headspace, at 4°C or less in an area free of solvent fumes. The size of any bubble caused by degassing upon cooling the sample should not exceed 5 - 6 mm. When a bubble is present, also observe the cap and septum to ensure that a proper seal was made at time of sampling. If the sample was improperly sealed, the sample should be discarded.

6.2 All samples should be analyzed within 14 days of collection that are preserved; 7 days for unpreserved water samples. Samples not analyzed within this period must be noted and data are considered minimum values.

7.0 PROCEDURE

7.1 The purge-and-trap technique for aqueous samples is found in Sec. 7.2 and guidance for analysis of solvent extracts from the High Concentration Method in Method 5035 is found in Sec. 7.3.

7.2 This section provides guidance on the analysis of aqueous samples and samples that are water miscible, by purge-and-trap analysis.

7.2..1 Initial calibration and verification

This is a preparation method but would not be complete without a reference on instrument calibration. Two techniques are used for analysis of samples. They are GC and GC/MS.

7.2.1.1 GC initial calibration and verification information can be found in the Standard Operating Procedure for analysis used in conjunction with this preparation.

7.2.1.2 GC/MS initial calibration and verification information can be found in the Standard Operating Procedure for analysis used in conjunction with this preparation.

7.2.2 Sample screening

7.2.3.1 All samples and standard solutions must be allowed to warm to ambient temperature before analysis.

7.2.3.2 Adjust the purge gas flow rate (nitrogen or helium) to 40 ml/min on the urge-and-trap device. Optimize the flow rate to provide the best response for chloromethane and bromoform, if these compounds are analytes. Excessive flow rate reduces response, whereas insufficient flow reduces bromoform response.

7.2.3.3 Place the sample on the autosampler. If reruns are necessary because of a calibration curve exceedance follow the steps below.

7.2.3.4 The following procedure is appropriate for diluting purgeable samples. All steps must be performed without delays until the diluted sample is in a 40 ml VOA Vial.

7.2.3.4.1 Dilution's are made in gastight 50ml syringes.

7.2.3.4.2 Calculate the approximate volume of organic-free reagent water added to the syringe and add slightly less than this quantity of organic-free reagent water to the syringe barrel.

7.2.3.4.3 Inject the proper aliquot of sample to create the desired dilution into the 50 ml syringe. Dilute the sample to the mark with organic-free reagent water. Invert, and shake

three times. Repeat the above procedure for additional dilution's.

7.2.3.4.4 Fill a 40-ml voa vial with the diluted sample from the 50 ml syringe prepared in Section 7.2.3.4.3.

7.2.4.7 Place the voa vial on the autosampler. All dilution's should keep the response of a major constituent (previously saturated peaks) in the upper half of the linear range of the curve.

7.2.4.8 The autosampler will add the 1.0ul of the internal standard and surrogate to the sample and transfer 5mls over to the 5ml sparge tube on the Tekmar 3000 concentrator.

7.3 Preparation of solvent extracts form High Concentration Samples prepared by Method 5035.

7.3.1 If a screening procedure was followed, use the estimated concentration to determine the appropriate volume. Otherwise, estimate the concentration range of the sample from the low-concentration analysis to determine the appropriate volume.

7.3.2 Table 1 can be used to determine the volume of solvent extract to add to the 49ml of organic-free reagent water for analysis. If the sample was submitted as a high-concentration sample, start with 1.0ml. All dilution's must keep the response of a major constituents (previously saturated peaks) in the upper half of the linear range of the curve.

7.3.3 Remove the plunger from a 50ml Luer-lok type syringe and fill until overflowing with organic-free reagent water. Replace the plunger and compress the water to vent trapped air. Adjust the volume to 49 ml. Pull the plunger back to 50ml to allow volume for the addition of the sample extract and of standards. Also add the volume of solvent extract determined in Section 7.3.1 or 7.3.2 and a volume of the same solvent used in Method 5035 to to a 1000uL (excluding methanol in standards).

7.3.4 Fill the 40ml VOA vial with the sample and place on autosampler rack. The autosampler will add 1ul of the internal standard and transfer the sample and standards to the 5ml sparge tube.

7.3.5 Proceed with the analysis as outlined in the specific determinative method. Analyze all reagent blanks on the same instrument as that used for the samples. The standards and blanks should also contain 1000ul of methanol matrix match.

8.0 QUALITY CONTROL

8.1 Before processing any samples, the analyst should demonstrate through the analysis of an organic-free reagent water method blank that all glass ware and reagents are interference free. Each time a set of samples is extracted, or there is a change in reagents, a method

blank should be processed as a safeguard against chronic laboratory contamination. The blank samples should be carried through all stages of the sample preparation and measurement.

8.2 Standard quality assurance practices should be used with this method. Field duplicates should be collected to validate the precision of the sampling technique. Each analysis batch of 20 or less samples must contain: a reagent blank; either a matrix spike/matrix spike duplicate or a blank spike and duplicate sample analysis; and a laboratory control sample, unless the analytical method provides the guidance.

8.3 Surrogate standards should be added to all samples when specified in the appropriate analytical method.

TABLE 1

QUANTITY OF METHANOL EXTRACT REQUIRED FOR ANALYSIS OF HIGH-CONCENTRATED SOIL/SEDIMENTS

Approximate Concentration Range	Volume of Methanol Extract[a]
500-10,000ugk/g	1000ul
1,000-20,000ugk/g	500ul
5,000-100,000ug/kg	100ul
25,000-500,000ug/kg	20ul

Calculate appropriate dilution factor for concentrations exceeding this table.

a. The volume of methanol added to 50ml of water being purged should be kept constant. Therefore, add to the 50ml syringe whatever volume methanol is necessary to maintain a volume of 1000ml added to the syringe.

ATTACHMENTS B-3

Laboratory QC Checks and Acceptance Criteria

GCMS BNA

QUALITY CONTROL

Preparation Method ⇨ Quality Control Measure ⇩	SW846 8270C
Initial Calibration	Minimum of five levels; lowest level near but above MDL.
Method Blank	One per batch of samples, up to 20 environmental samples, whichever is more frequent.
DFTPP Tune Check	One at the beginning of a 12 hour time clock or more frequent.
Calibration Verification Standard (CVS)	One at the beginning of a 12 hour time clock or more frequent.
Independent Calibration Verification	Independent source run immediately after the calibration curve.
Laboratory Control Spike and Duplicate	One pair per batch of samples, up to 20 environmental samples, whichever is more frequent.
Matrix Spike and Duplicate	One pair per batch of samples, up to 20 environmental samples, whichever is more frequent.
Internal Standards	Added to every sample.
Method Validation	Annually
MDL	Annually
Surrogate Standards	Added to every sample.

GCMS BNA

CONTINGENCIES FOR HANDLING OUT-OF-CONTROL OR UNACCEPTABLE DATA

Analytical Method Acceptance Criteria⇨ Quality Control Measure ⇩	Method Citation SW If these conditions are not achieved ⇒	Corrective Action (Key below)
Matrix Spike and Duplicate	•	• 1
Internal Standards	•	• 2
Surrogate Standards	•	• 2

1. If Lab Control Spikes pass acceptance criteria, flag parent sample to indicate matrix effects.

2. When sample volume is available reanalyze the samples. If the second analysis indicates the same internal standard recoveries add analytical comments and effects on data.

PCB

QUALITY CONTROL

Preparation Method ⇨ Quality Control Measure ⇩	**SW846 8082**
Initial Calibration	Minimum of five levels; lowest level near but above MDL.
Method Blank	One per batch of samples, up to 20 samples, whichever is more frequent.
Calibration check Standard (CCS)	One at the beginning of a 12 hour time clock or more frequent.
Independent Calibration Verification	Independent source run immediately after the calibration curve.
Laboratory Control Spike and Duplicate	One pair per batch of samples, up to 20 environmental samples, whichever is more frequent.
Matrix Spike and Duplicate	One pair per batch of samples, up to 20 environmental samples, whichever is more frequent.
Method Validation	Annually
MDL	Annually
Surrogate Standards	Added to every sample.

PCB
CORRECTIVE ACTIONS

Analytical Method Acceptance Criteria⇨ Quality Control Measure ⇩	Method Citation SW If these conditions are not achieved ⇒	Corrective Action (Key below)
Initial Calibration		• 1
Method Blank		• 2
Calibration Check Standard		• 3

1. Check instrumentation/equipment condition; enter maintenance in instrument log; perform another initial calibration

2. Samples detects greater than 20 times the method blank contamination level are reported with those not exceeding 20 times re-analyzed or qualified.

3. Immediately after the flawed CCV analyze another CCV or re-analyzed all samples set after the last acceptable CCV.

GCMS Volatiles

QUALITY CONTROL

Preparation Method ⇨ Quality Control Measure ⇩	SW846 5030B	SW846 5035
Initial Calibration	Minimum of five levels; lowest level near but above MDL	Minimum of five levels; lowest level near but above MDL
Method Blank	One per batch of samples, up to 20 environmental samples, whichever is more frequent.	One per batch of samples, up to 20 environmental samples, whichever is more frequent.
Continuing Calibration Verification	Mid-level calibration standards run at the beginning of each 12 hour time clock.	Mid-level calibration standards run at the beginning of each 12 hour time clock.
Independent Calibration Verification	Mid-level calibration standard from an independent source to verify initial calibration curve.	Mid-level calibration standard from an independent source to verify initial calibration curve.
Laboratory Control Spike and Duplicate	One pair per batch of samples, up to 20 environmental samples, whichever is more frequent.	One pair per batch of samples, up to 20 environmental samples, whichever is more frequent.
Matrix Spike and Duplicate	One pair per batch of samples, up to 20 environmental samples, whichever is more frequent.	One pair per batch of samples, up to 20 environmental samples, whichever is more frequent.
Internal Standards	Added to every sample.	Added to every sample.
Surrogate Standards	Added to every sample.	Added to every sample.

GCMS Volatiles
CORRECTIVE ACTIONS

Analytical Method Acceptance Criteria⇨ Quality Control Measure ⇩	Method Citation SW If these conditions are not achieved ⇒	Corrective Action (Key below)
Initial Calibration		• 1
Method Blank		• 2
Matrix Spike and Duplicate		• 3
Continuing Calibration Verification		• 4

1. Check instrumentation/equipment condition; enter maintenance in instrument log; perform another initial calibration

2. Samples detects greater than 20 times the method blank contamination level are reported with those not exceeding 20 times re-analyzed or qualified.

3. If Lab Control Spikes pass acceptance criteria, flag parent sample to indicate matrix effects.

4. Immediately after the flawed CCV analyze another CCV or re-analyzed all samples set after the last acceptable CCV.

APPENDIX C

Site Health and Safety Plan

APPENDIX C: SITE HEALTH AND SAFETY PLAN
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ATTACHMENTS

Attachment C-1: Hazardous Property Information
Attachment C-2: Levels of Personal Protective Equipment (PPE)
Attachment C-3: Personnel Training Requirements
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APPENDIX C: SITE HEALTH AND SAFETY PLAN

This Site Health and Safety Plan is specifically prepared for:

Project Location: Eagle Zinc Company Site, Hillsboro, Illinois
Environ Case Number: 21-7100E

This Site Health and Safety Plan has been developed to protect the health of personnel involved in site activities and the surrounding community and is consistent with the following regulations and guidance:

- 20 CFR 1910.120 (i) (2) – Occupational Health and Safety Administration: Hazardous Waste Operations and Emergency Response, Interim Rule, December 19, 1986.
- USEPA Order 1440.2 – Health and Safety Requirements for Employees Engaged in Field Activities.
- USEPA Order 1440.3 – Respiratory Protection.
- USEPA Occupational Health and Safety Manual.
- USEPA Interim Standard Operating Procedures (September, 1982).

All personnel participating in the field must be trained in the general and specific hazards unique to the job and, if applicable, meet recommended medical examination requirements (see Attachments C-1, C-2, C-3 and C-4 for hazardous property information, levels of personal protective equipment, personnel training requirements and medical surveillance program, respectively). All site personnel and visitors shall follow the guidelines, rules, and procedures contained in this safety plan. The project manager or site health and safety officer may impose any other procedures or prohibitions believed to be necessary for safe operations.

This plan is prepared to inform all field personnel, including ENVIRON personnel and subcontractors, of the potential hazards on the site. However, ENVIRON and its

subcontractors must assume direct responsibility for their own employees' health and safety.

This plan supersedes all other health and safety plans written for this site.

A. Introduction

1. **Site Location:** Eagle Zinc Company Site
   Hillsboro, Illinois

2. **Plan Prepared:** F. Ross Jones
   March 2002

3. **Plan Approved:** Roy O. Ball, Ph.D., P.E.
   March 2002

4. **Plan Revised:**
   (if applicable)

5. **Revision Approved:**
   (if applicable)

6. **Summary of Possible Hazards on This Job**

Chemical Hazards may result from potential exposure to volatile organic compounds (VOCs), or contaminated soil, sediment, residues, surface water or ground water. The concerned routes of potential chemical exposure include: inhalation of dust generated during drilling activities; dermal contact when handling environmental samples, drill cuttings, or other equipment; and ingestion usually occurring through accidental means, inadequate decontamination, or poor personal hygiene practices. Refer to Section D for identified chemical contaminants.

Physical Hazards may result from potential slipping, tripping, and falling hazards. Physical hazards may be avoided by exercising caution in performing fieldwork. In addition, PPE can reduce field personnel's dexterity, narrow the field of vision, and diminish communication and hearing capabilities.

Temperature Hazards may result from seasonal variances. Certain field activities may occur during summer or winter months. Thus, field personnel must be familiar with the symptoms of cold and heat stress. Refer to Section I on Hazard Mitigation for details.

Traffic Hazards may result from working near heavy equipment (i.e., drill rigs). Field personnel should avoid walking under suspended loads, and in front of or in back of moving equipment. All personnel not directly handling the operating equipment should maintain a safe distance.

Acoustical Hazards may result from close proximity to heavy equipment or other machinery producing excessive noise levels. Sound pressure levels exceeding an 8-hour time weighted average (TWA) of 85 dBA and/or causing elevated voice levels to converse, require the use of hearing protection. Hearing protection (i.e., ear plugs or insulated ear muffs) must be worn when in close proximity to operations involving heavy equipment or machinery producing excessive noise levels. Long-term exposure to excessive sound pressure levels may cause temporary or permanent hearing loss, abnormal hormone secretions, and tensing of muscles.

Fire Hazards may result from the use of diesel (flash point 110 – 190°F) or other fuel (gasoline). To avoid fire hazards, personnel will separate flammable materials and ignition sources by a minimum of 50 feet. Personnel should also use non-sparking, explosion-proof equipment while working with flammable materials.

7. Required Personal Protective Items and Equipment for this Project

- Steel-Toe Boots – worn on site grounds at all times;
- Waterproof/Chemical Resistant Disposable or Reusable Overboots- worn during surface water/sediment sampling or any other activities for which work must be conducted in standing water or muddy conditions.
- Hardhat, Safety Glasses, and Earplugs – worn for drilling activities;
- Gloves – worn during any activity with potential dermal contact with chemicals (when handling all environmental samples); and,

- Respirator – On Site available as needed.

B. Persons Responsible And Involved

1. **Project Manager:** F. Ross Jones, P.G.

   Health and Safety Responsibilities: Overall responsibility for project compliance with health and safety plan.

2. **Site Supervisor:** Chris Greco

   Health and Safety Responsibilities: Implementation of the site-specific health and safety plan for all field-related activities.

3. **Site Health and Safety Officer:** Chris Greco

   Health and Safety Responsibilities: Same as site supervisor.

4. **Others:** ENVIRON Field Personnel

   Health and Safety Responsibilities: Responsible for providing health and safety consultation for the project.

5. **Subcontractors:** Driller, Surveyor, and Excavation Contractor

   Health and Safety Responsibilities: Responsible for the safe operation of equipment while on Site, and for the health and safety of their own employees both on Site and off Site. This includes implementing a site and task specific health and safety plan.

C. Facility Background And Planned Activities

1. Facility Background and Description

The Site is located in the Township of Hillsboro, Illinois. Hillsboro is located in central Montgomery County, Illinois, approximately 50 miles northeast of St. Louis, Missouri and 35 miles south of Springfield, Illinois. The Site is approximately 132 acres in size and is defined as the parcels of land currently owned by Eagle Zinc Company.

The Site is located in a mixed commercial/industrial/residential area located in the northeastern part of Hillsboro. The Site extends from Smith Road south to an unnamed tributary to the Middle Fork of Shoal Creek. Industrial Drive extends north and south along much of the eastern property boundary. North of the Site is Smith Road, a small facility called Hayes Abrasives, a golf course, and farm fields. Industrial Drive, an asphalt company, a railroad corridor, and the former Hillsboro Glass Company facility (now a steel warehouse) are located east of the Site. Some small commercial/industrial facilities (University of Illinois Extension office, Fuller Brothers Construction/Ready Mix, Illinois Wood Preservers, Hillsboro Rental, Vogel Plumbing) are located south of the Site. Some undeveloped land and a residential area containing single- and multi-family dwellings are located west of the Site. The nearest residential properties are located approximately 200 feet west of the southern and central part of the Site.

It is estimated that between 10 and 15 percent of the Site is covered by buildings. Approximately 23 buildings currently exist at the Site, some of which are currently unused or are used only for storage purposes. The types of buildings currently used for facility operations include the office/laboratory building, manufacturing/processing buildings, equipment/raw material/finished product storage buildings, baghouses, and maintenance facilities. Other Site features include railroad spurs, raw material and residual material stockpiles, two storm water detention ponds, a small pond in the southeast corner of the property, and several paved and unpaved roadways.

2. Site History

Zinc processing operations began at the Site in 1912, at which time the facility operated as a zinc smelter under the name Lanyon Zinc Company. The smelting products reportedly included zinc and sulfuric acid. The Site was purchased by Eagle-Picher Industries in 1919. Eagle-Picher conducted zinc smelting and manufacture of sulfuric acid until approximately 1935. Subsequent to 1919 and most likely in the early 1920s, the manufacture of zinc oxide and leaded zinc oxide commenced at the Site. The leaded zinc oxide was manufactured by combining basic lead sulfate (obtained from off-Site sources) with zinc oxide. Additional details on the leaded zinc oxide operation are currently unavailable; however, these activities ceased around 1958. Eagle-Picher continued to manufacture zinc oxide at the Site until November 1980, at which time the Site was purchased by The Sherwin-Williams Company. According to Sherwin-Williams personnel, The Sherwin-Williams Company conducted manufacturing operations for a period of less than one year. In 1984, the facility was sold by The Sherwin-Williams Company to Eagle Zinc Company, a division of T.L. Diamond & Company. Eagle Zinc has continued manufacturing zinc oxide using the American process employed by Sherwin-Williams and Eagle-Picher.

Zinc oxide has been manufactured at the Site using both direct and indirect processes. The indirect process reportedly involved the processing of zinc metal in a muffle furnace. The direct process, which continues to be used at the Site (the American process) involves the processing of zinc ores and stockpiled furnace residues in a rotary kiln furnace. While it is likely that Eagle-Picher, Sherwin-Williams and Eagle Zinc all used the direct process, only Eagle-Picher and Sherwin-Williams used the indirect process (muffle furnace). Residual materials historically generated by the manufacturing operations have included, among other things, rotary kiln residue, muffle dross, metallic zinc particles, and refractory bricks from the facility's furnaces. Zinc oxide is used in many applications, including the paint and ceramics industries, agricultural products, rubber products and cosmetics.

As discussed above, other products historically manufactured at the Site include leaded zinc oxide, metallic zinc, and sulfuric acid. Sulfuric acid was reportedly manufactured at the Site by roasting zinc sulfide to remove the sulfur, with the southwest surface water pond were used to provide non-contact cooling water. In addition, the facility has produced a fine-grained product that is rich in carbon by screening stockpiled rotary residues using a rotary screen and other methods.

The pyrometallurgical process currently used by Eagle Zinc for zinc oxide manufacturing, and which had also been used by Eagle-Picher and Sherwin-Williams, is known as the American process. As currently implemented, this process involves mixing zinc-bearing feedstocks with sized anthracite coal at the mix room. The coal is delivered to the Site by railcar; the zinc ore is delivered to the Site by railcar and truck. The furnace mix is fed into a natural gas-fired rotary furnace, 8-foot diameter by 50 foot long, at the Block 2 Furnace Building. The natural gas provides the heat source and the anthracite coal provides a reducing atmosphere to reduce the zinc feedstocks to zinc vapor. The zinc vapor is drawn from the rotary furnace into a refractory brick combustion chamber and combusted to zinc oxide by the addition of ambient air. The zinc oxide, suspended in the vapor stream (products of combustion and air), is drawn into a steel flue and a series of steel cooling loops to cool the zinc oxide and vapor stream before it is collected in a baghouse. The residue left in the rotary furnace is expelled from the rotary furnace into the discharge chamber, quenched in water and hauled to a pad for storage. The zinc oxide collected in the baghouse is conveyed to the refinery and stored in bins before refining. Based on the physical and chemical properties of the zinc oxide, bins of zinc oxide may be blended while being refined. The refining process involves conveying the zinc oxide through a natural gas fired rotary dryer in which the temperature of the zinc oxide is varied to achieve the desired product characteristics. The historic operation of the American process would have been similar.

Eagle Zinc currently produces two products: zinc oxide by the American process (described above), and a carbon-rich by-product by a screening process.

The facility screens stockpiled rotary residues using a double rotary screen with ¼-inch and 2-inch screen sizes to produce the fine-grained product that is rich in carbon. This operation is conducted on a concrete pad located immediately west of the zebra building. Large and medium oversize materials created by this process are currently stockpiled to the west of the concrete pad. Eagle Zinc is exploring options for beneficial reuse of this material and is consulting with IEPA on these efforts.

3. Hazardous Incidence History

Based on information provided by Site personnel, as well as pre-RI/FS data collected at the Site, no incidents involving specific releases or spills of hazardous chemicals have occurred at the Site.

4. Purpose of Activity/Objective of Work

The overall purpose of the RI is to investigate the Site's physical characteristics, identify sources of contamination, and determine the nature and extent of contamination at the Site. The results of the field sampling activities will provide input to the baseline risk assessment and the evaluation of potential response action alternatives in the Feasibility Study. The baseline risk assessment, to be completed as part of the RI, will quantify the actual or potential risk to human health and the environment posed by contaminants potentially present in soil, sediment, surface water and ground water on Site, as well as sediment and surface water in drainageways located off Site. The evaluation of removal alternatives for the Site will include an assessment of risk. The results of the RI will be sufficient to provide a basis for the development and evaluation of potential remedial response alternatives for the site during the subsequent FS. The RI for the Eagle Zinc Site will be conducted in accordance with the *Guidance for Conducting Remedial Investigations and Feasibility Studies Under CERCLA* (USEPA, 1988b), as applicable.

The Site activities will be conducted in two separate phases: Phase 1-Contaminant Characterization, and Phase 2-Contaminant Migration Assessment.

Phase 1 will include the investigation and sampling of soil, sediment and plant residues. Phase 2 will include the investigation and sampling of surface water and ground water. The following items present an overview of the Site investigation activities to be carried out during the RI.

- Soil Investigation: Approximately 130 shallow soil borings will be completed in on-Site areas to characterize and delineate the extent of organic and inorganic contaminant concentrations at the site. The majority of soil borings will be completed in the areas previously identified as Areas 1 through 4, with a minimum of 25 borings in each of these areas. Soil borings will also be completed in the manufacturing plant area and in the historically undeveloped northern and western portions of the site property. A minimum of 10 soil borings will be completed in each of these three areas.

  Soil samples will be screened on-Site for metals using a portable X-ray fluorescence (XRF) analyzer. The XRF results will be used to identify samples that will be retained for metals analyses, as well as to identify locations and depths of additional soil borings.

  Approximately 20% of the soil samples will be retained for laboratory analysis. The soil samples will be analyzed for the full suite of Target Analyte List (TAL) metals.

  The soil samples will also be screened for organic vapors at the same time as the XRF screening mentioned above. Approximately 10% of the total soil samples will be retained for a more comprehensive suite of analytes, including Target Compound List (TCL) organic compounds and polychlorinated biphenyls (PCBs).

- Sediment Investigation: A sediment investigation will be conducted in on-Site and off-Site portions of the storm water and surface water drainage

systems that originate on-site or enter the site from adjacent properties. The principal objective of the sediment investigation will be to characterize the nature and extent of inorganic impacts on sediments in the drainage systems. The samples will be collected as transect composites at sediment accumulation points or at representative locations in the drainage ditches/streams on-Site and off-Site and screened for organic vapors using a PID.

A minimum of 16 sediment samples will be collected for laboratory analyses and all sediment samples will be analyzed for TAL metals. Approximately 25% of sediment samples will also be analyzed for a more comprehensive suite of analytes including TCL organic compounds and PCBs. The initial phase of sampling shall include collection of a sediment sample at or near the confluence of the eastern drainage ditch with Lake Hillsboro.

- Residue Investigation: Samples will be collected from each discrete residue piles or groups of similar piles. The samples will be collected as composites from trenches excavated to the base of the piles. The samples will be tested for metals using the Toxicity Characteristic Leaching Procedure (TCLP) and the Synthetic Precipitation Leaching Procedure (SPLP).

- Surface Water Investigation: An evaluation will be made of potential on-Site sources of storm and surface water impacts and potential locations of surface and storm water discharges. Results of the Phase 1 sediment sampling will be used to determine final Phase 2 surface water sampling locations, which will include some locations where Phase 1 sediment samples were collected. Samples near storm water Outfalls 001 and 002 will also be included in this sampling program.

- Ground Water Investigation: A series of temporary monitoring wells/piezometers will be installed in the southwestern portion of the property,

in the manufacturing plant area, and in the northern and western portions of the Site. The temporary wells will be used to 1) provide information concerning the hydraulic relationship between the shallow groundwater and the southwest pond; 2) better characterize the pattern of shallow groundwater flow on-site and off-site; and 3) assist in the identification of locations for additional permanent monitoring wells. Approximately 20 temporary wells will be installed.

Groundwater screening samples will be collected the temporary wells to assist in the identification of locations for installation of additional permanent monitoring wells.

Based on the results from the temporary wells and ground water flow patterns, approximately 10 permanent monitoring wells at or near selected temporary well locations.

Following the completion and development of the permanent monitoring wells, all existing and newly installed monitoring wells will be sampled for TAL metals. The metals analyses will be conducted using filtered and unfiltered samples to determine total and dissolved metals concentrations. In addition, a minimum of four samples will be analyzed for TCL organics and PCBs. A complete round of water level measurements will be made to allow for the construction of a site-wide groundwater elevation contour map.

5. Site Status

The Eagle Zinc Company facility is an active manufacturing concern with full-scale operations requiring 3 full shifts, 7 days per week.

6. Surroundings

See site definition in Section C.1.

7. Site Map

See Figures II-1 and II-2 in the RI/FS Work Plan.

8. Climate

The following information on the climate of Hillsboro, Illinois was obtained using on-line sources of historical weather data. The climate of Montgomery County is considered continental and temperate. The summer months are hot and humid with an average temperature of 75° Fahrenheit (F) and an average daily high temperature is 87° F. The winter months are moderately cool with an average temperature of 31° F and an average daily high temperature of 40° F. Rainfall is well distributed throughout the year, with the highest average rainfall in May. Total annual precipitation for the area is approximately 41 inches. Approximately 57 percent, or 23 inches, of the total annual precipitation occurs as rain from April through September and coincides with the growing season. The average total snowfall accumulation is approximately 18 inches.

The following information is for Springfield, Illinois, which is located approximately 35 miles north of Hillsboro. The average relative humidity is 83 percent in the morning and 63 percent in the afternoon. With the exception of January, the prevailing average wind direction throughout the year is from the south. In January, the average wind direction is from the west-northwest. The average wind speeds are greatest in January, March, April, and November, at 13 miles per hour (mph). The lowest average wind speeds are in July and August, at 8 mph.

D. Identified Chemical Contaminants

1. Identified Chemical Contaminants Known to be Present

As a result of historical and ongoing investigations of the Site, the list of potential contaminants of concern (PCOCs) includes the following metals:

- Antimony

- Arsenic
- Beryllium
- Cadmium
- Chromium
- Copper
- Lead
- Selenium
- Thallium
- Zinc

2. Chemical Contaminants Characterization

As discussed in the PSE Report, significant site characterization activities have been completed, including:

- An October 1993 CERCLA expanded site inspection conducted by IEPA, which included collection of on-site residue samples and on-site/off-site soil and sediment samples.
- A May 1998 investigation conducted by GBI, which included collection of on-site soil and residue samples.
- A July 1998 investigation conducted by GBI, which included sampling of storm water outfalls 001 and 002; and
- A December 1998 investigation conducted by GBI, which included installation and sampling of nine monitoring wells.

E. General Work Practices

- No one will be permitted to engage in hazardous work operations alone.
- Smoking, eating, or drinking will not be permitted within the work zones.
- Personnel should keep track of weather conditions and wind direction to the extent they could affect potential exposure.

- Personnel should be alert to any abnormal behavior on the part of other workers that might indicate distress, disorientation, or other ill effects.
- Personnel should never ignore symptoms that could indicate potential exposure to chemical contaminants. These should be immediately reported to their supervisor or the Site Health and Safety Officer.
- Visible indicators of potential immediate danger to life and health (IDLH) conditions include:

  1) Large containers and tanks that must be entered.
  2) Enclosed spaces such as buildings or trenches that must be entered.
  3) Potentially explosive or flammable situations (indicated by bulging drums, effervescence, gas generation, or instrument readings).
  4) Extremely hazardous materials (not expected at this site) such as cyanide, phosgene, or sources of significant radiation.
  5) Visible vapor clouds.
  6) Areas where biological indicators such as dead animals or vegetation are located.

F. Site Control/Work Zones

1. Locations of Exclusion Zone, Contamination Reduction Zone, and Decontamination Area and Support Zone

The exclusion zone will be that area immediately surrounding the work being performed. It will be the responsibility of the Site Health and Safety Officer to prevent unauthorized personnel from entering the exclusion zone. When necessary, such as in high traffic areas, the exclusion zone will be delineated with barricade tape, cones, and/or barricades. It is not anticipated that a contamination reduction zone, decontamination area, or support zone will be required.

Decontamination of PPE and sampling equipment will take place within the exclusion zone immediately surrounding the work being performed. Decontamination of larger drilling equipment (e.g., augers) will be performed within a designated decontamination area established on the Eagle Zinc property.

2. Site Control/Security Measures

The site is not secured by fencing or other access controls. All field equipment will either be brought to the site each day, or stored overnight in at a secure indoor location provided by plant personnel.

3. Safety Plan Locations

A copy of the health and safety plan will be kept at each field location whenever work is being performed.

G. Site Resources

Toilet facilities: Existing Eagle Zinc facilities or portable toilet.

Drinking water supply: Field personnel will supply their own bottled drinking water

Telephone: Mobile phones will be carried by field personnel. On-site phones are also available in the Eagle Zinc office Building.

Other: Emergency contact locations and phone numbers are listed in Section O.

H. Hazard Analysis
Post at Job Site

Task Identifuer	Task	Mechanical (A)	Physical (B)	Electrical (C)	Chemical (D)	Temp (E)	Acoustical (F)	Ergonomic (G)	Fire (H)	O2 Deficiency (I)	Radiation (J)	Biohazard (K)	Traffic (L)
SB	Soil Borings/Soil Sampling	X	X	X	X	X	X	X				X	X
RP	Residue Pile Excavations	X	X		X	X	X	X				X	
MW	Monitoring Well Installation/Sampling	X	X	X	X	X	X	X				X	X
SD	Sediment Sampling							X					
SW	Surface Water Sampling		X		X	X		X				X	

(A) Mechanical hazards include all hazards associated with handling field equipment and machinery (e.g. hand and foot injuries and burns).
(B) Physical hazards include slip, trip, and fall (from an elevated surface or into a trench).
(C) Electrical hazards include electrical shocks from field equipment and underground utilities.
(D) Chemical hazards include inhalation and dermal exposure to various chemicals.
(E) Temperature hazards include heat stress, sunburn, heat exhaustion, hypothermia, frostbite, and cold stress.
(F) Acoustical hazards include exposure to elevated levels of noise.
(G) Ergonomic hazards include back strain caused by improper lifting, etc.
(H) Fire hazards include explosion hazards.
(I) Oxygen deficiency hazards include enclosed spaces and poor circulation.
(J) Radiation hazards include radiation greater than 2mR/hr.
(K) Biohazards include exposure to vegetation, insects, animals, and blood-borne pathogens.
(L) Traffic hazards include moving vehicles.

I. Hazard Mitigation

1. Mechanical Hazards

Activities: SB, RP, MW

Mitigation:

- Do not stand near excavator buckets or drilling rigs.
- Verify that all equipment is in good condition.
- Do not stand or walk under elevated loads or ladders.
- Do not stand near unguarded excavations and trenches.
- Do not enter excavations or trenches over 5 feet deep that are not properly guarded, shored, or sloped.
- Consult HSC if other mechanical hazards exist.

2. Physical Hazards

Activities: All

Mitigation:

- Contain all purge water in drums to avoid creating a wet surface.
- If the work area is wet, wear boots with good treads and alert the personnel of wet surface.
- If the area is wet or muddy, sprinkle saw dust on the surface to prevent slip.
- Consult HSC if other physical hazards exist.

3. Electrical Hazards

Activities: SB, MW

Mitigation:

- Locate and mark buried utilities before drilling.
- Utilities located by public and/or private locating services prior to work.
- Maintain following clearances from overhead power lines:

– 750-50,000 Volts	10 Feet Clearance
– 50,000-75,000 Volts	11 Feet Clearance
– 75,000-125,000 Volts	13 Feet Clearance
– 125,000-175,000 Volts	15 Feet Clearance
– 175,000-379,000 Volts	21 Feet Clearance
– 379,000-550,000 Volts	27 Feet Clearance
– 550,000-1,000,000 Volts	42 Feet Clearance

- Contact utility company for minimum clearance from high power lines.
- If unavoidably close to buried or overhead power lines, have power turned off, with circuit breaker locked and tagged.
- Properly ground all electrical equipment if possible.
- Avoid standing in water when operating electrical equipment.
- If equipment must be connected by splicing wires, make sure all connections are properly taped.
- Be familiar with specific operating instructions for each piece of equipment.
- Insure equipment used in wet areas has GFCI protection or is double-insulated.

4. Chemical Hazards

Activities: All

Mitigation:

- Use personal protective equipment indicated in Section V.K.
- Conduct direct reading air monitoring to evaluate respiratory hazards (list instrument, action level, monitoring location, and action to be taken in Section J).

- All work conducted in the area of residue piles will be conducted up-wind of the piles; no work shall be conducted in areas where prolonged or sustained emissions of dust are observed unless appropriate engineering controls are implemented.
- Consult HSC for personal air monitoring.

5. Temperature Hazards

Activities: All

Mitigation:

Heat Stress – If employees will wear impermeable suits during this project, take frequent breaks in shaded area when the ambient temperature exceeds 75°F. Unzip or remove coveralls during breaks. Have cool water or electrolyte replenishment solution available. Drink small amounts frequently to avoid dehydration. Count the pulse rate for 30 seconds as early as possible in the rest period. If the pulse rate exceeds 110 beats per minute at the beginning of the rest period, shorten the work cycle by one-third. Signs and symptoms of heat stress include:

1. Heat rash, which may result from exposure to heat or humid air.
2. Heat cramps are caused by heavy sweating with inadequate electrolyte replacement. Signs and symptoms include muscle spasms and pain in the hands, feet and abdomen. Persons experiencing these symptoms should rest in a cooler area, drink cool liquids (not cold) and gently massage cramped muscles.
3. Heat exhaustion occurs from 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. Persons experiencing these symptoms should lie down in a cooler area, drink cool liquids with

electrolytes, remove any protective clothing, and cool body with wet compresses at forehead, back, neck, and/or armpits.

4. Heat stroke is the more 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/or death occur. 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.

Sunburn Hazards – Skin exposure to ultraviolet rays can result in sunburn. Wear light-colored long-sleeved shirt, hat, and sunscreen to protect against sunburn.

Hypothermia and Frostbite – Hypothermia occurs when the internal body (core) temperature falls below 95 °F, at which time the body tries to generate more heat through shivering. Further heat loss would result in the following symptoms: speech difficulty, loss of manual dexterity, lethargy, slow reactions, mental confusion, and muscle rigidity.

When the ambient temperature is below freezing, frostbite or trench foot (immersion foot) may accompany or complicate the symptoms of hypothermia. Frostbite is the freezing of living tissues with a resultant breakdown of cell structure. Symptoms include superficial redness of the skin, dull pain, blisters, and skin discoloration (white, yellow-white or blue-white) when tissue becomes frozen.

During the winter season, the following protective measures will be taken by the Site personnel:

1. All outside operations will cease when the wind chill becomes –25 °F or colder, or at the discretion of the SHSO.
2. Wear layers of loose, dry clothing and stay dry.
3. Avoid overheating and remove clothing layers when starting to perspire.
4. Perform periodic exercise to help keep the extremities warm.

5. Do not touch frozen metal with bare skin, particularly the tongue.
6. Avoid skin contact with evaporative liquids (such as gasoline and alcohol) below 39 °F.
7. Keep all exposed skin covered and keep head covered at all times. Humans lose 75% of their body heat from the head.
8. Wear boots that are waterproof, with liners. Remove liners at night to let them dry.
9. Use foot powder inside boots to absorb any moisture in boots.
10. Use safety sunglasses with green or amber lenses on sunny days to prevent snow blindness.

If hypothermia or frostbite occurs, the victim will be treated only by the personnel who have been trained in treating such injuries.

6. Acoustical Hazards

Activities: SB, RP, MW

Mitigation:

- Wear earplugs if elevated noise is encountered (e.g., difficult to carry out conversations when standing next to each other).

7. Ergonomic Hazards

Activities: All

Mitigation:

- While lifting heavy equipment, keep the back straight and legs bent. If the equipment cannot be lifted in this manner, it is too heavy to lift alone. Call other personnel, or use a mechanical device for lifting.

8. Fire and Explosion Hazards

Activities: None

Mitigation:

- Conduct direct reading air monitoring to evaluate explosion hazards (list instrument, action level, monitoring location, and action to be taken in Section 10).
- Use care when handling compressed air/gas cylinders and lines.

9. O_2 Deficiency

Activities: None

Mitigation:

- Oxygen deficiency is generally concerned with confined spaces. Confined spaces include trenches, pits, sumps, elevator shafts, tunnels, or any other area where circulation of fresh air is restricted or ability to readily escape from the area is restricted. Consult HSC prior to entering confined space.

  - Obtain permit for confined space entry.
  - Monitor O_2 and organic vapors before entering. If following values are exceeded, do not enter:

    1. O_2 less than 19.5% or greater than 25%.
    2. Total hydrocarbons greater than 5 ppm above background, if all air contaminants have not been identified.
    3. Concentrations of specific contaminants exceeding action level in Section 9 if all contaminants are identified.

- Monitor O_2 and organic vapors continuously while inside confined space. If values cited in Section 9 are exceeded, evacuate immediately. Record instrument readings.
- At least one person must be on standby outside the confined space that is capable of pulling workers out of confined space in an emergency.
- Use portable fans or blowers to introduce fresh air to confined spaces whenever use of respirator is required.
- Work involving the use of flame, arc, spark, or other source of ignition is prohibited within a confined space.

10. Radiation Hazards

Activities: None

Mitigation:

11. Biohazard

Activities: All

Mitigation:

- Poison oak, poison ivy.
- Poisonous reptiles.
- Insects that are potential disease carriers, harmful, or poisonous. Examples include ticks, mosquitoes, and poison spiders.

12. Traffic Hazards

Activities: SB, MW

Mitigation:

- Traffic will be controlled when work is performed areas of the plant that are subject to vehicular traffic. The work area will be coned-off, as appropriate.

J. Air Monitoring

1. Gases and Vapors

For air monitoring of gases and vapors, the action levels and the actions to be taken are summarized in the following table. Background readings will be taken every day and recorded in the field log. Personnel exposures shall be monitored continuously during intrusive activities, and readings shall be recorded approximately every 15 minutes, or as appropriate.

Instrument & Agents Monitored	***Calibration* Standard**	***Action Levels****	***Actions***
Organic Vapor Meter to Monitor VOCs	**Isobutylene (100 ppm)**	**1 ppm above background sustained for 1 minute**	**If OVM reading is 1 ppm above background or greater for 1 minute in the breathing zone, stop work and don a respirator or employ other mitigation measures such as increased ventilation to the work area.**

2. Explosion Hazard

Not Expected.

3. Oxygen Deficiency

Not Expected.

K. Required Personal Protective And Related Safety Equipment

LEVEL D

Comments: Level D PPE provides minimal skin protection and no respiratory protection.

Activity

List #

Head

SB, TP, MW Hardhat

Eye

All Safety Glasses

Hand

ALL Neoprene

Nitrile

PVC

Body

NONE Tyvek Suit – Avoid direct dermal contact with the soils on site

Hooded Tyvek/Saranex Suit

Lung

NONE SCBA (open circuit, pressure demand):

Full Face Respirator

Half Face Respirator

Ear

SB, TP, MW Earplug, type = foam

Foot

ALL Steel-toed boots. Disposable or reversible over boots – activities within shallow water bodies or muddy conditions

L. Decontamination And Spill Containment

1. Sampling and Construction Equipment Decontamination

Dedicated sampling equipment and monitoring well construction materials which have been pre-cleaned or have been prepackaged will not require decontamination prior to use. Sample containers will be pre-cleaned as specified in the QAPP. All non-dedicated sampling equipment will require decontamination prior to its use. Equipment will be decontaminated by either steam cleaning with a high-pressure washer, or washing in a low phosphate detergent wash such as Alconox™, followed by a rinse with municipal water and a final distilled water rinse.

2. Personnel Decontamination

All site personnel should minimize contact with contaminants. All disposable PPE will be disposed of in on-site trash receptacles. Non-disposal PPE must be decontaminated, particularly the safety boots. Any PPE that cannot be decontaminated should be disposed of along with waste generated from field operations. Personnel shall wash and remove PPE prior to leaving the site. At a minimum, gross removal of contaminants from the PPE, removal of the PPE, and washing of hands and face shall be required upon leaving the site.

3. Investigation-Derived Material Disposal

Five types of investigative-derived waste (IDW) will be generated during the course of this investigation. These IDWs are: (1) soils (hollow-stem auger and direct-push drill cuttings and excess soil samples). (2) monitoring well development water, (3) decontamination water, (4) monitoring well purge water, and (5) personal protective equipment (e.g., gloves).

Soil cores and soil samples generated from direct-push drilling will be returned to the borehole from which they were extracted. As organic contaminants are not known to be a concern for soils in either the unsaturated or saturated zone, soil cuttings generated by hollow-stem auger drilling for well installation will be spread on the ground surface at or near the well location. If field evidence of contamination is observed based on PID readings or visual observations, the drill cuttings will be containerized and labeled according to their source. The contents of the containers will then be sampled for determination of hazardous waste characteristics. If determined to be non-hazardous, the waste will be disposed as a special industrial waste. If the waste is found to be hazardous, the waste will be disposed of at a permitted off-site treatment, storage and disposal (TSD) facility.

Wastewater from equipment decontamination, well development, and well purging will be containerized. The analytical results for the ground water samples will be used to determine the disposition of these wastewaters. Disposal options include placement of the water in the Site's engineered storm water retention basin or off-site disposal as a nonhazardous waste.

Waste personal protective equipment generated during the site activities does not have the potential to accumulate significant amounts of hazardous waste due to the nature of the materials. Personal protective equipment such as gloves will be cleaned, if necessary, and allowed to dry before being disposed with the plant's industrial solid waste.

4. Spill Containment

- All drums and containers used during cleanup shall meet appropriate DOT, OSHA, and EPA regulations for the waste that they will contain.

- Drums and containers shall be inspected and their integrity assured prior to being moved.

- Operations on site will be organized so as to minimize the amount of drum or container movement.

- Fire extinguishing equipment meeting OSHA 29 CFR Part 1910 Subpart I shall be on hand and ready for use to control fires, as necessary.

M. Hazard Communication

In order to comply with OSHA 29 CFR 1910.120, Hazard Communication, ENVIRON has established a written Hazard Communication Program, which is maintained at each ENVIRON office. The following procedures are applicable to this site. All employees will be briefed on this program, and will have a written copy for review.

1. Container Labeling

All containers received on site will be inspected to ensure the following: (1) All containers are clearly labeled as to the contents; (2) the appropriate hazard warnings are noted; and (3) the name and address of the manufacturer are listed.

All drums contain IDW's staged for characterization and off-site disposal will have a label affixed with the following information: (1) the identity of the waste generator, (2) the boring, well, or excavation identification and sample depth, (3) the waste matrix (e.g. soil, water, product). and (4) the date of waste generation.

2. Employee Training and Information

Prior to starting work, each ENVIRON employee and subcontractor will attend a site health and safety orientation and will receive information and training on the following, as applicable t the daily on-site activities:

(1) an overview of the requirements contained in the Hazard Communication Standard, 29 CFR 1910.120;

(2) chemicals present in their workplace operations;

(3) location and availability of a written hazard program;

(4) physical and health effects of the hazardous chemicals;

(5) methods and observation techniques used to determine the presence or release of hazardous chemicals;

(6) how to lessen or prevent exposure to these hazardous chemicals through usage of control/work practices and personal protective equipment;

(7) emergency procedures to follow if they are exposed to these chemicals;

(8) how to read labels and review MSDSs to obtain appropriate hazard information; and

(9) locations of MSDS files and hazardous chemical lists.

N. Documentation

All ENVIRON personnel medical and training records are retained at the home office for each staff member. Site records will consist of any necessary training and medical surveillance verification documentation for personnel.

1. Safety Plan Distribution Record

a. ENVIRON Employees

All project staff must sign indicating they have read and understand the Site Health and Safety Plan. A copy of the Site Health and Safety Plan will be made available for their review and readily available at the job site.

b. Contractors, Subcontractors

A copy of this safety plan shall be provided to contractors and subcontractors who may be affected by activities covered under the scope of this Site Health and Safety Plan for their information only, although the contractors and subcontractors remain responsible for the safety of their own employees. All contractors and subcontractors must comply with applicable OSHA, EPA, and local government rules and regulations.

2. Health and Safety Meeting

All personnel participating in the project must receive initial health and safety orientation. Thereafter, periodic tailgate safety meetings will be required as deemed necessary by the site health and safety officer.

3. Visitor

It is ENVIRON's policy that visitors must furnish their own personal protective equipment. All visitors are required to sign the visitor log and comply with health and safety plan requirements. If the visitor represents a regulatory agency concerned with site health and safety issues, the site health and safety officer shall also immediately notify the HSC.

O. Contingency/Emergency Information

1. Required Emergency Equipment Location

Safety shower/eyewash:	In first aid kit
First aid kit:	In ENVIRON field vehicle.
Fire extinguisher:	In ENVIRON field vehicle.

2. Emergency Telephone Numbers

Ambulance:	911
Police:	911
Fire department:	911
Hospital:	(217) 532-6111

Poison Control Center:	(800) 233-3360
CHEMTREC:	(800) 424-9300
Project Manager (office)	(847) 444-9200
HSC	Cellular Phone: *To Be Inserted*
Eagle Zinc (main line)	(217) 532-3971
ENVIRON Field Office	*To Be Inserted*

3. Standard Procedures for Reporting Emergencies

When calling for assistance in an emergency situation, the following information should be provided:

1. Name of person making call
2. Telephone number at location of person making call
3. Name of person(s) exposed or injured
4. Nature of emergency
5. Actions already taken

Recipient of call should hang up first--not the caller.

4. Route to the Hospital:

1. Exit the plant heading south on Industrial Park Drive.
2. Turn right (west) on School Street.
3. Turn left (south) on Anna Street.
4. Turn right (west) on Tremont Street.

See Hospital Location Map in Attachment C-5.

Hillsboro Area Hospital
1200 E Tremont St
Hillsboro, IL 62049-1900
(217) 532-6111

ATTACHMENT C-1

Hazardous Property Information

ATTACHMENT C-1
HAZARDOUS PROPERTY INFORMATION

Material	Water Solubility[a]	Specific Gravity	Vapor Density	Flash Point °F	Vapor Pressure	LEL UEL	LD_{50} mg/kg	PEL-TWA[g]	IDLH Level	Odor Threshold or Warning Concentration (ppm)	Hazard[j] Property	Dermal[k] Toxicity	Acute[l] Exposure Symptoms
Metals													
Antimony	[b]	6.69	N/A	None	N/A	[f]	7,000	0.5 mg/m3	None specified	None specified	C		FGIKLNQ
Arsenic	[b]	2.21[x]	N/A	None	N/A	[f]	763	0.010 mg/m^3	None specified	None specified	CEG	CJG	ACDGJMOQR
Beryllium	[b]	1.85	N/A	None	N/A	[f]	0.496	2 µg/m^3	None specified	None specified	C		IJMNR
Cadmium	[b]	8.642	N/A	None	N/A	[f]	225	0.005 mg/m^3	None specified	None specified	C		ABGHIKLMNQR
Chromium	[b]	7.20	N/A	None	N/A	[f]	- -	1 mg/m^3[h]	None specified	None specified	C		FMNQ
Copper	[b]	8.94	N/A	None	N/A	[f]	- -	1 mg/m^3	None specified	None specified	C		FGIJMOR
Lead	[b]	11.3437	N/A	None	N/A	[f]	- -	50 mg/m^3	None specified	None specified	C		ACDFGKOQR
Selenium	[b]	4.28	N/A	None	N/A	[f]	6,700	0.2 mg/m^3	None specified	None specified	C		IJKN

ATTACHMENT C-1
HAZARDOUS PROPERTY INFORMATION

Material	Water Solubility[a]	Specific Gravity	Vapor Density	Flash Point °F	Vapor Pressure	LEL UEL	LD_{50} mg/kg	PEL-TWA[g]	IDLH Level	Odor Threshold or Warning Concentration (ppm)	Hazard[j] Property	Dermal[k] Toxicity	Acute[l] Exposure Symptoms
Silver	[b]	10.5	N/A	None	N/A	[f]	- -	0.01 mg/m^3	None specified	None specified	C		IN
Titanium	[b]	4.26	N/A	None	N/A	[f]	- -	10 mg/m^3	None specified	None specified	C		M
Zinc	[b]	7.14	N/A	None	N/A	[f]	- -	10 mg/m^3	None specified	None specified	C		DF

SITE-SPECIFIC SUBSTANCES
(Add hazardous property information on any substances that are of concern at the site but are not listed above.)

EXPLANATIONS AND FOOTNOTES

ATTACHMENT C-1
HAZARDOUS PROPERTY INFORMATION

Material	Water Solubility[a]	Specific Gravity	Vapor Density	Flash Point °F	Vapor Pressure	LEL UEL	LD_{50} mg/kg	PEL-TWA[g]	IDLH Level	Odor Threshold or Warning Concentration (ppm)	Hazard[j] Property	Dermal[k] Toxicity	Acute[l] Exposure Symptoms

Water solubility is expressed in different terms in different references. Many references use the term "insoluble" for materials that will not readily mix with water, such as gasoline. However, most of these materials are water soluble at the part per million or part per billion level. Gasoline, for example, is insoluble in the gross sense, and will be found as a discrete layer on top of the ground water. But certain gasoline constituents, such as benzene, toluene, and xylene, will also be found in solution in the ground water at the part per million or part per billion level.

[a] Water solubility expressed as 0.2 g means 0.2 grams per 100 grams water at 20 °C.

[b] Solubility of metals depends on the compound in which they are present.

[c] Several chlorinated hydrocarbons exhibit no flash point in a conventional sense, but will burn in the presence of high energy ignition source or will form explosive mixtures at temperatures above 200 °F.

[d] Practically non-flammable under standard conditions.

[e] Expressed as mm Hg under standard conditions.

[f] Explosive concentrations of airborne dust can occur in confined areas.

[g] OSHA Time weighted Average (TWA) Permissible Exposure Limits (PELs) except where noted in h and i.

[h] TLV-TWA adopted by the American Conference of Governmental Industrial Hygienists (ACGIH), which is lower than the OSHA PEL.

[i] REL-TWA recommended by the National Institute for Occupational Safety and Health (NIOSH). A TLV or PEL has not been adopted by ACGIH or OSHA.

ATTACHMENT C-1
HAZARDOUS PROPERTY INFORMATION

Material	Water Solubility[a]	Specific Gravity	Vapor Density	Flash Point °F	Vapor Pressure	LEL UEL	LD_{50} mg/kg	PEL-TWA[g]	IDLH Level	Odor Threshold or Warning Concentration (ppm)	Hazard[j] Property	Dermal[k] Toxicity	Acute[l] Exposure Symptoms

[j]
A - corrosive
B - flammable
C - toxic
D - volatile
E - reactive
F - radioactive
G - carcinogen
H - infections

[k] Dermal Toxicity data is summarized in the following three categories;
Skin Penetration
- A - negligible penetration (solid-polar)
+ B - slight penetration (solid-nonpolar)
++ C - moderate penetration (liquid/solid-nonpolar)
+++ D - high penetration (gas/liquid-nonpolar)
Systemic Potency
E - slight hazard - LD_{50} = 500-15,000 mg/kg
lethal dose for 70 kg man = 1 pint-1 quart
F - moderate hazard - LD_{50} = 50-500 mg/kg
lethal dose for 70 kg man = 1 ounce-1 pint
G - extreme hazard - LD_{50} = 10-50 mg/kg
lethal dose for 70 kg man = drops to 20 ml
Local Potency
H - slight - reddening of skin
I - moderate - irritation/inflammation of skin
J - extreme - tissue destruction/necrosis

ATTACHMENT C-1
HAZARDOUS PROPERTY INFORMATION

Material	Water Solubility[a]	Specific Gravity	Vapor Density	Flash Point °F	Vapor Pressure	LEL UEL	LD_{50} mg/kg	PEL-TWA[g]	IDLH Level	Odor Threshold or Warning Concentration (ppm)	Hazard[j] Property	Dermal[k] Toxicity	Acute[l] Exposure Symptoms

[l] Acute Exposure Symptoms
- A - abdominal pain
- B - central nervous system depression
- C - comatose
- D - convulsions
- E - confusion
- F - dizziness
- G - diarrhea
- H - drowsiness
- I - eye irritation
- J - fever
- K - headache
- L - nausea
- M - respiratory system irritation
- N - skin irritation
- O - tremors
- P - unconsciousness
- Q - vomiting
- R - weakness

[x] Relative density with respect to Air at ambient temperatures.

ATTACHMENT C-2

Levels of Personal Protective Equipment (PPE)

ATTACHMENT C-2

Levels of Personal Protective Equipment (PPE)

1. Selection of PPE

The selected PPE should be able to resist degradation, penetration, and permeation by the contaminants present at the Site. In selecting the appropriate protective material, the following should be considered: chemical resistance; tear and puncture resistance; flexibility; thermal stress; cleanability and durability.

PPE will be selected, used and maintained in accordance with 29 CFR 1910.132, *General requirements*. Eye and face PPE requirements will be in accordance with 29 CFR 1910.133, *Eye and face protection*, and ANSI Z87.1-1979. Respiratory protection will be selected in accordance with 29 CFR 1910.134, *Respiratory protection*. Selection of foot protection will conform with ANSI Z41.1-1983, and 29 CFR 1910.136, *Occupational foot protection*. PPE for the head will be in accordance with 29 CFR 1910.135, *Occupational head protection*, and ANSI Z89.1-1986.

Levels of PPE

The four levels of PPE are Levels A, B, C, and D, with Level A providing the highest available level of respiratory, skin, and eye protection. A summary of the basic PPE ensemble for Levels A, B, C, and D is provided below. PPE selection for operations at the Site will be tailored to address specific task conditions.

Level A

Level A PPE provides the maximum degree of respiratory, skin, and eye protection. A Level A PPE ensemble should include:

- Full-facepiece self-contained breathing apparatus (SCBA) or full-facepiece supplied air respirator with escape SCBA;

- Fully encapsulating, chemical-resistant suit, safety boots and inner gloves; and

- Hardhat (if overhead or bump hazards exist).

Level B

Level B PPE provides the maximum level of respiratory protection. Since chemical-resistant clothing is not considered gas, vapor, or particulate tight, Level B PPE does not provide the maximum skin protection. However, a good quality, hooded, chemical-resistant one-piece garment with taped wrists and ankles provides a reasonable degree of protection against splashes of liquids and lower concentrations of chemicals in ambient air. It is the minimum level recommended for confined space entries and initial Site entries until the hazards have been further identified. Level B PPE should be used when any one of the following criteria is met:

- The type and atmospheric concentration of substances have been identified and require a high level of respiratory protection but less skin protection -- this includes atmospheres with IDLH concentrations of specific substances that do not represent a severe skin hazard or atmospheres that do not meet the criteria for use of air-purifying respirators;
- Atmosphere contains less than 19.5% oxygen; or
- Presence of incompletely identified vapors or gases is indicated by air monitoring instruments but vapors and gases are not suspected of containing high levels of chemicals harmful to skin or capable of being absorbed through the intact skin.

Level C

Level C PPE provides the same level of skin protection as Level B PPE, but a lower level of respiratory protection. Air-purifying respirators can be used only if the substance has adequate warning properties; the individual passes a qualitative fit-test for the mask; an appropriate cartridge/canister is used and its service limit concentration is not exceeded; and Site operations are not likely to generate unknown compounds or excessive

concentrations of already identified substances. Level C PPE can be used when all of the following conditions are met:

- Oxygen concentrations are not less than 19.5%;
- Atmospheric contaminants, liquid splashes, or other direct contact will not adversely affect any exposed skin;
- Types of air contaminants have been identified, concentrations measured, and a cartridge or canister is available that can remove the contaminant;
- Atmospheric contaminant concentrations do not exceed IDLH levels; and
- Job functions do not require self-contained breathing apparatus (SCBAs).

Level D

Level D PPE provides minimal skin protection and no respiratory protection. Level D PPE can be used when the following conditions are met:

- Atmosphere contains no known hazard;
- Oxygen concentrations are not less than 19.5%; and
- Work functions preclude splashes, immersion, or the potential for unexpected inhalation of or contact with hazardous levels of any chemicals.

2. Respirator Fit Test

Prior to the initiation of any fit testing, personnel must be certified as medically able to wear a respirator. The respirator fit test is conducted to ensure proper facepiece-to-face seal. A secure fit is important with positive-pressure equipment, and is essential to the safe functioning of negative-pressure equipment, such as most air-purifying respirators. Personnel will receive instruction on proper wear and maintenance of the respirator.

Qualitative fit tests will be conducted annually in accordance with the ANSI *Practices for Respiratory Protection*, Z88.2-1989. In addition, a negative and positive fit check will be performed each time an employee dons the air-purifying respirator (APR). Documentation of annual respirator fit tests will be kept in the Support Zone.

Negative and Positive Fit Check

The negative and positive pressure fit check will be performed each time an employee dons the APR. The negative pressure fit check involves closing off the inlet openings to the APR cartridges by covering with the palms of the hands. If an inward leakage of air is detected, the APR should be checked for material defects and refitted or replaced with another APR.

The positive pressure fit check is performed by placing the palm of hand over the exhalation valve and gently exhaling for 10 seconds to create positive pressure inside the facepiece. If an outward air leak is detected, the APR should be readjusted. If after readjustment leakage still occurs, another APR should be used.

3. PPE Inspection Checklist and Maintenance

PPE inspections will be conducted upon receipt of PPE from the factory or distributor; when it is issued to workers; after use or training; and prior to maintenance. Periodic inspections of stored equipment will be conducted routinely, whenever a question arises concerning the appropriateness of the selected equipment, or when problems with similar equipment arise. At a minimum, PPE inspection should include the following:

A. Clothing

Before use:

- Determine that the clothing material is correct for the specified task.
- Visually inspect for:
  - Imperfect seams
  - On-uniform coatings
  - Tears
  - Malfunctioning Closures
- Hold up to light and check for pinholes

- Flex product:
  - Observe for cracks
  - Observe for other signs of shelf deterioration

- If the product has been used previously, inspect inside and out for signs of chemical breakthrough or deterioration, such as:
  - Discoloration
  - Swelling
  - Stiffness

During the work task, periodically inspect for:

- Evidence of chemical attack such as discoloration, swelling, stiffening, and softening. Keep in mind that chemical permeation can occur without any visible effects.
- Closure failure
- Tears
- Punctures
- Seam discontinuities

B. Gloves

Before use, pressurize glove to check for pinholes. Either blow into glove, then roll gauntlet towards fingers or inflate glove and hold under water. In either case, no air should escape.

C. Respirators

SCBA/supplied air/air-purifying:

- Inspect SCBA/supplied air/air-purifying respirators before and after each use, at least monthly when in storage and during cleaning. Air-purifying respirators should be inspected before each use to be sure they have been adequately cleaned.
- Check all connections for tightness, inspect air lines prior to each use for cracks, kinks, cuts, frays, and weak areas.
- Check for proper setting and operation of regulators and valves (according to manufacturer's recommendations) and check operation of alarms.
- Check material conditions for:
  - Signs of pliability
  - Signs of deterioration
  - Signs of distortion
- Check faceshields and lenses for:
  - Cracks
  - Crazing
  - Fogginess
- Examine cartridges or canisters to ensure that:
  - They are the proper type for the intended use,
  - The expiration date has not passed, and

They have not been opened or used previously.

ATTACHMENT C-3

Personnel Training Requirements

ATTACHMENT C-3

Personnel Training Requirements

All personnel performing on-site operations with the potential for exposure to hazardous substances or health hazards will meet the personnel training requirements in accordance with 29 CFR 1910.120(e). The training policies and procedures will ensure that personnel can recognize hazards, understand emergency response procedures, and have the knowledge necessary to enable them to perform their assigned jobs in a manner that ensures employee and public safety. Documentation of appropriate health and safety training, as described below, and medical surveillance participation, as described in the HASP, will be required to gain access to on-site areas other than the Support Zone. Documentation of all training, including initial 24-hour or 40-hour health and safety training, 8 hours of annual refresher training, 8 hours of supervisor training, supervised field experience, first aid training, CPR certification, and confined space entry or Level B training, if applicable, will be kept on-site.

1. Initial Training

A. Basic Health and Safety Training

A minimum of 24 hours of initial health and safety training off-site is required to obtain on-site access to areas other than the Support Zone. All personnel engaged in or supervising activities in the EZ or CRZ will have a minimum of 40 hours of initial health and safety training off-site, meeting the requirements of 29 CFR 1910.120(e)(3).

B. Supervised Field Experience

All personnel with 24 hours of initial health and safety training are also required to have a minimum of 1 day of field experience under the direct supervision of an experienced supervisor. Personnel with 40 hours of initial health

and safety training are required to have a minimum of 3 days of field experience under the direct supervision of an experienced supervisor.

C. Supervisor Training

All on-site managers and supervisors directly responsible for, or who supervise personnel engaged in invasive site activities will have received the initial 40-hour health and safety training and at least 8 additional hours of specialized off-site training consistent with 29 CFR 1910.120(e)(4). This specialized training will include topics such as, but not limited to, regulatory compliance, management of on-site health and safety hazards and recognition of special personnel training needs.

D. Health and Safety Officer Training

Health and safety officers will be trained to a level required by their job function and responsibility. This will include training in implementation of HASPs and compliance with applicable health and safety requirements.

E. First Aid and CPR Training

A minimum of two individuals certified by the American Red Cross (or equivalent) to render first aid and CPR will be available during each shift. The Site Health and Safety Officer (SHSO) will have first aid and CPR training. A list of additional on-site personnel qualified to perform first aid and CPR will be posted throughout the Site.

2. Refresher Training

All personnel who have received 24 hours or 40 hours of initial health and safety training will receive 8 hours of refresher training annually, as specified in 29 CFR 1910.120(e)(8). Topics to be covered in this training program will include those specified in the initial 40-hour health and safety training and/or those specified in the supervisory training course, as well as a critique of incidents that could serve as training examples.

Project-specific refresher training will be provided when the project scope is changed and/or when the hazards change.

A. Site Safety Briefings

Site safety briefings will be conducted prior to the start of each work day or work shift to discuss health and safety issues, changes in work procedures, exposure incidents and other relevant information. The SHSO and Site Coordinator will conduct these meetings. Prior to each change in operations, the meetings will address PPE use and maintenance, physical safety hazards from machinery, protection from chemical hazards, decontamination procedures, protection from heat/cold stress and specific safety requirements associated with the new operations. During the meetings, the SHSO or SC will identify on-site personnel qualified to perform first aid and CPR. All changes in the HASP will be reviewed during the morning safety briefing. A record of the meeting will be written daily and signed by all participants. These records will be stored in the field office.

B. Visitor's Briefing

Visitors will not be permitted to enter areas other than the Support Zone unless documentation of training, as described above, is presented to the SHSO. All visitors will be trained by the SHSO in hazard recognition, personnel hygiene and Site safety rules, use of PPE, and emergency response procedures. Visitors requesting on-site access to areas other than the Support Zone will be required to review and sign off on the HASP (see Section 12 of the HASP) to ensure understanding and compliance with the provisions in the

ATTACHMENTS C-4

Medical Surveillance

ATTACHMENT C-4

The goals of the medical surveillance program are to monitor the health of potentially exposed personnel through the use of medical examinations and diagnostic laboratory testing, to provide medical care for occupational injury or illness, to keep accurate records for future reference and to ensure the selection of personnel physically able to safely perform the work assigned. The medical surveillance program supports and monitors the effectiveness of the primary health and safety goal of controlling worker exposure to hazardous substances. OSHA regulations relating to medical surveillance during hazardous waste operations are detailed in 29 CFR 1910.120(f). Medical examinations will be performed by or under the supervision of a licensed physician, preferably one knowledgeable in occupational medicine.

Documentation of current participation in a medical surveillance program and fitness for duty, including ability to wear respiratory protective equipment, will be necessary for all personnel who work on-site in areas other than the Support Zone. However, all specific medical information and examination results obtained in the course of administration of the medical surveillance program will be maintained by the examining physician as confidential.

1. Baseline Medical Examinations

The baseline medical examination serves two major purposes: (1) it determines the individual's fitness for duty, including the ability to work while wearing a respirator; and (2) it provides baseline data for comparison with future medical data. The baseline medical examination will include, at a minimum, the following:

- Complete occupational and medical history;
- Physical examination;
- Blood count and chemistry profile;
- Urinalysis with microscopic review;
- Chest x-ray;

- Pulmonary function tests;
- Resting electrocardiogram (EKG); and
- Cardiac stress test (at physician's discretion).

Certification of fitness for duty and ability to wear personal protective equipment must be provided to gain access to on-site areas other than the Support Zone. However, all specific medical information obtained in the course of administration of the medical surveillance program will be maintained as confidential.

2. Periodic Medical Examinations

Each individual enrolled in the medical surveillance program will be subject to periodic medical surveillance examinations. In general, personnel involved in field activities with a frequency of greater than 30 days per year will receive medical examinations at least annually. Periodic medical examinations should include the parameters included in the baseline examination, with the exception of the chest x-ray and EKG, which are repeated after the baseline examination at the physician's discretion and with agreement of the individual.

3. Special Medical Examinations

Special medical examinations or consultations will be arranged for personnel exposed in an emergency situation to hazardous substances at concentrations above the OSHA-PELs without adequate protection. This will be done as soon as possible after the overexposure has been determined by the SHSO, in consultation with the Health and Safety Manager.

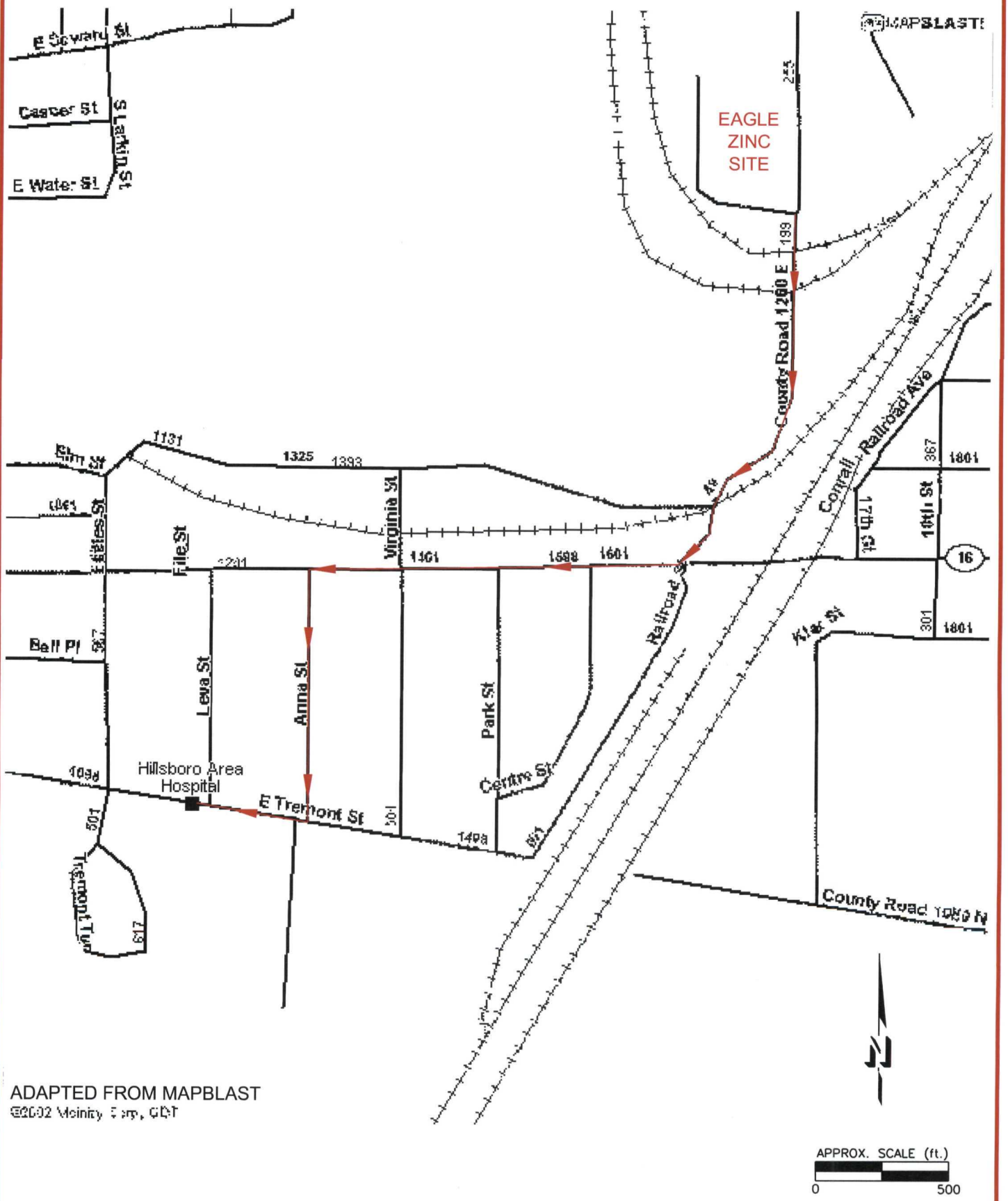
Special medical examinations shall also be arranged upon notification by the individual that he/she has developed signs or symptoms indicating a possible overexposure to hazardous substances, or if the examining physician determines that more frequent medical examinations are necessary. Non-scheduled medical examinations may also be directed at the discretion of the Health and Safety Technical Committee and the Site Health and Safety Officer.

4. Special Circumstances

Any individual who is on a medication that may interfere with the ability to perform his/her job function, or who may require special medical attention, must notify the SHSO of these circumstances prior to commencing work at the Site.

ATTACHMENTS C-5

Hospital Location Map



ENVIRON 740 Waukegan Road, Suite 401, Deerfield, IL 60015	Hospital Location Map Eagle Zinc Hillsboro, Illinois	Attachment C-5

Drafter: APR Date: 6/25/02 Contract Number: 21-7400E Approved: Revised:

CPFs/Eagle Zinc-...oro/RIFS Work Plan/Appendix C/Attachments/Attchmt C-5 (Hospital Location Map)

APPENDIX D

Baseline Risk Assessment Plan

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APPENDIX D: BASELINE RISK ASSESSMENT PLAN

A. Baseline Human Health Risk Assessment

Based on the relevant historical data and data generated during the planned RI, a baseline human health risk assessment (HHRA) will be conducted for the Site. The HHRA will quantify risks posed by the Site and will analyze public health impacts of the remedial alternatives. The methodology presented in this plan conforms to the *Risk Assessment Guidance for Superfund, Human Health Evaluation Manual* (1991) and any current USEPA guidance.

The purpose of the HHRA will be to characterize the potential current and future human health risks associated with existing environmental conditions in on-Site areas and off-Site areas investigated for potential impacts. These estimated risks will be based on the current and most likely future land uses. The preliminary list of potential exposure scenarios includes:

- Current/Future On-Site Employees;
- Future On-Site Construction Workers;
- Future On-Site Trespasser;
- Future On-Site Residents (Hypothetical);[1] and
- Current/Future Off-Site Residents.

The HHRA will be composed of the following key components:

- Hazard Identification,
- Exposure Assessment,
- Dose-Response Assessment,
- Risk Characterization, and
- Uncertainty Assessment.

[1] Residential development of the site is not permitted under current zoning ordinances.

Ultimately, the HHRA results will be used in a risk management framework. In making decisions concerning what actions, if any should be taken at the Site (e.g., collection of additional sampling data, implementation of remedial actions), the results of the HHRA will be used in concert with other information about the Site. The HHRA process will identify whether a selected scenario/exposure point combination presents unacceptable risks and identifies the Contaminants of Concern (COCs) that contribute the most to these estimated risks.

1. Conceptual Site Model

The conceptual site model describes the exposure conditions that will be evaluated in the HHRA. These exposure conditions are characterized by (1) the potentially exposed populations; and (2) the potential exposure pathways, which consist of a contaminant source, an environmental medium transporting constituents from the contaminant source to a receptor, and a route of entry for constituents into the receptor. Based on the evaluation of data collected at the site prior to the RI/FS, a preliminary conceptual site model was developed.

The conceptual site model is based on the current and reasonably expected future use of the Site. The current on-site land use is industrial, and there is no use of ground water at the Site. Potable water is provided to the Site by the local public water purveyor. In the HHRA, information regarding future land use plans and factors affecting ground water use will be evaluated to determine the likelihood that the current land and ground water uses on-Site will remain the same in the future.

The current off-Site land use is a mixture of residential, commercial and industrial and there is no known use of ground water at adjacent and nearby downgradient properties. Potable water is reportedly provided to residents and businesses in the area of the site by the local public water purveyor, the Township of Hillsboro.

a) **Current and Future On-Site Exposures**

i. **Adult Workers**

Under this scenario, on-Site workers may be exposed to constituents in surface soil via ingestion, inhalation, and dermal contact. They may also inhale vapors and particulates emitted from unsaturated soil to the ambient air.

ii. **Adult Construction Workers**

Under this scenario, on-site construction workers may be exposed for a short time period to constituents in both surface and subsurface soil via ingestion, inhalation and dermal contact during hypothetical future construction activities at the site. It will also be assumed that adult construction workers may inhale vapors and particulates emitted from the soil to the ambient air and that those workers could be exposed to ground water through ingestion, inhalation and dermal contact.

b) **Current and Future Off-Site Exposures**

i. **Off-Site Residents**

Under this scenario, off-Site residents may be exposed to constituents in ground water, as well surface water and sediments in the storm water drainageways and associated tributary streams through incidental ingestion, inhalation and dermal contact. The need to evaluate off-site soils as a pathway will be predicated on the results of on-site soil and residual waste pile sampling and visual inspections conducted during the RI.

2. **Risk Characterization**

Human health risks will be estimated based on the conceptual site model outlined above and based on USEPA risk assessment policy and guidance. Key

elements of the approach are provided below.

a) Equations and Models

The HHRA will include evaluation of available analytical data to estimate chemical concentrations at locations of hypothetical human exposure. The standard equations needed to estimate exposure concentrations and risks will be taken from USEPA guidance (USEPA 1989, 1991b, 1996).

b) Exposure Factors

The potential frequency and duration of exposure for the receptors defined in the conceptual site model will be based on conservative estimates associated with typical activities for the receptors. Methods for exposure assessment and relevant standard default exposure factors necessary to develop RME estimates will be taken from USEPA guidance (e.g., USEPA 1991, 1991a, 1992b, 1996, 1997c, 2000a) with adjustments, as appropriate, to account for site-specific conditions and activities. In all cases, the most current USEPA guidance will be followed in developing exposure factors. USEPA Region 9 (USEPA 1999) provides the most up-to-date guidance regarding dermal exposure factors (skin surface area, adherence factors, and absorption values). The magnitude of exposure will be based on the Reasonable Maximum Exposure (RME) calculated in accordance with current USEPA guidance (USEPA 1992a, USEPA 1997a).

c) Toxicity Values

USEPA-derived toxicity values from USEPA's Integrated Risk Information System (IRIS) will be used. For constituents lacking values in IRIS, other USEPA toxicity values will be used according to the following hierarchy: (1) Health Effects Assessment Summary Tables (USEPA 1997b); (2) National Center for Environmental Assessment (NCEA); (3) USEPA Region 3 Risk-Based Concentration (RBC) table (USEPA 2000b); and (4)

USEPA Region 9 Preliminary Remediation Goals (PRGs) table (USEPA 1999).

Carcinogenic risks will be estimated using USEPA-derived cancer slope factors (CSFs) for constituents having a USEPA weight-of-evidence classification of Group A, B, or C. The CSFs are 95% upper confidence bounds on the risk per unit dose (USEPA 1997b).

The potential for non-cancer effects will be estimated using USEPA-derived reference doses (RfDs). The RfD represents a conservative estimate of the daily exposure to the human population, including sensitive subpopulations that are likely to be without an appreciable risk of deleterious effects during a specified exposure period. USEPA's derivation of RfDs incorporates several uncertainty factors (or safety factors) which, in combination, generally range from 100- to 1,000-fold (USEPA 1997b). Chronic exposures will be evaluated using chronic toxicity values and sub-chronic exposures will be evaluated using sub-chronic toxicity values.

Oral CSFs and oral RfDs will be used for evaluating oral (ingestion) exposures, and inhalation CSFs and inhalation RfDs will be used for evaluating inhalation exposures. When a USEPA-derived inhalation toxicity value is expressed as an air unit risk or inhalation reference concentration (RfC), these values will be converted to inhalation CSFs and inhalation RfDs based on 70 kg body weight and 20 m^3/day inhalation rate.

d) Risk Characterization and Uncertainty Analysis

ENVIRON will calculate risks by integrating exposure scenarios and assumptions with toxicity data. Cancer risk estimates and Hazard Quotients (HQs) for individual chemicals will be summed within exposure pathways and the cumulative risk estimates and Hazard Indices (HIs) summed across pathways for each receptor, as appropriate. However, consistent with EPA guidance, high-end cancer and noncancer risk estimated for more than one pathway will not be combined unless a hypothetical receptor is likely to consistently face the high-end exposure via more than one pathway

simultaneously. Summing across pathways also may result in a significant overestimate of potential risks. For example, if chemical concentrations are assumed to be at the 95th upper confidence level (UCL) of the mean (USEPA 1997a), then adding chemical exposures results in a highly unlikely overall risk estimate. Any Hazard Index estimate that exceeds unity (i.e., above 1) will be evaluated further in the risk assessment, with chemicals contributing to that exceedance segregated by effect and by mechanism of action to derive separate hazard indices for each group. The results of the risk characterization will also be evaluated in the context of the uncertainty inherent in the assumptions used in the risk calculations.

B. Ecological Risk Screening Evaluation

An ecological risk screening (ERS) evaluation will be conducted following appropriate USEPA guidance (e.g., USEPA 1997d, 1998, 2000c). The ERS will involve screening of ecological risks and will rely on contaminant data obtained during remedial investigation activities. Based on USEPA guidance, the ERS will include the following steps.

1. Screening Level Problem Formulation and Ecological Effects Evaluation

The screening level problem formulation will consider aspects such as:

- ecological setting;
- chemicals or classes of chemicals;
- contaminant fate and transport processes;
- mechanisms of ecotoxicity of the contaminants for the probable categories of receptors;
- potentially complete exposure pathways; and
- preliminary endpoints.

This step will include a field survey by a qualified environmental ecologist, as well as the development of maps, figures and color photographs of the Site and

surrounding area, as appropriate, to identify environmentally significant areas at and near the site as well as potential migration pathways to those environmentally sensitive areas. The overall purpose of this step will be to identify the ecological setting at the Site and relevant off-Site areas, and to provide a preliminary evaluation of ecological exposure pathways and endpoints.

2. Screening Level Preliminary Exposure Estimate and Risk Calculation

In this step, chemicals of potential ecological concern (COPECs) will be identified through the comparison of the maximum concentrations of chemicals in, or with the potential to migrate to, the identified environmentally significant areas of the site to appropriate benchmark values for soil, sediment and surface water (USEPA 2000c). The maximum detected concentrations will be divided by the benchmark values to determine a Screening Hazard Quotient (SHQ). Contaminants with SHQs above 1.0 and those without screening values will be identified as COPECs. The COPECs will then be carried through to the next step, as described below.

3. Problem Formulation

At this stage, one or more COPECs determined as part of the risk calculations may be eliminated from further consideration based on the refinement of certain assumptions, such as background/reference location comparisons and the nature of the contaminants. If COPECs remain after refinement, this step will also involve designation of assessment endpoints (i.e., the ecological population community and specific characteristics that are to be protected), and further development of a conceptual model.

In order to identify assessment endpoints, ecological resources present at the site and the immediately surrounding area (e.g., habitat types, species, and functional attributes) will be characterized based upon a review of available data from the literature, existing site-specific reports, input from USEPA personnel, and the aforementioned ecological field survey. This characterization will include a listing of known occurrences of state and federal threatened/endangered species,

determined by consultation with state and federal natural resource agencies. The conceptual model will describe predicted relationships between the assessment endpoints and the stressors (e.g., chemicals) to which they may be exposed, develop risk hypotheses based on the predicted relationships, and identify measurement endpoints.

This information will be used to determine what further evaluations, if any, will be necessary to complete the ecological risk assessment. If required, any further evaluations would be consistent with appropriate USEPA guidance.

C. References

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USEPA Region 4 (2000c) *Region 4 Ecological Risk Assessment Bulletins – Supplement to RAGS.*